Document Title

Summary of the fate and behaviour in the environment for Aclonifen

Data Requirement(s)

Regulation (EC) No 1107/2009 & Regulation (EU) No 283/2013

Document MCA

Section 7: Fate and behaviour in the environment

According to the Guidance Document SANCO/10181/2013 for applicants on preparing dossiers for the approval of a chemical active substance

Date

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Version history

Date [yyyy-mm-dd]	Data points containing amendments or additions[1] and brief description	Document identifier and version number
2020-01-13	Original version	M-676648-01-1
2020-03-16	Section CA 7.2.4 deleted. Summary for Document M-676096-01-1 moved to Section CA 7.2.1. References to Document M-676285-01-1 replaced with references to Document M-676285-02-1. Updated version includes Bayer DART number for each reference in document.	M-676648-02-1
2020-07-08	Section CA 7.1.1.2 updated with the OECD summary of an anaerobic study Document M-687746-01-1 that was not available at the time of the submission.	M-676648-03-1

[1] It is suggested that applicants adopt a similar approach to showing revisions and version history as outlined in SANCO/10180/2013 Chapter 4, 'How to revise an Assessment Report'.

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CA 7 FATE AND BEHAVIOUR IN THE ENVIRONMENT

Aclonifen was included in Annex I to Council Directive 91/414/EEC in 2008 (Directive 2008/116/EC, Entry into Force on 01 August 2009). This present dossier in support of approval renewal includes all the data submitted at the time of the Annex I inclusion, in summaries updated and re-evaluated as necessary to take account of current validity criteria and data requirements.

CA 7.1 Fate and behaviour in soil

The fate and behaviour of aclonifen in soil has been investigated in a comprehensive series of laboratory studies and, when required, supported with data from field experiments. A number of studies were submitted for the first inclusion of aclonifen into Annex I of Council Directive 91/414/EEC and reviewed under uniform principles (DAR, Germany, 2006). In addition a number of new studies are provided for the current EU review. All valid environment fate studies are considered in the M-CA 7 dossier.

In the previous EU review soil DT_{50} values for aclonifen from two aerobic laboratory studies were criticized (Addendum to DAR, Germany, 2008). The original laboratory studies were conducted with [U-^{14}C-aniline] labelled aclonifen or non-labelled aclonifen. Three new aerobic soil metabolism studies (KCA 7.1.1.1/04 ([redacted], 2016, M-558848-01-1), KCA 7.1.1.1/05 ([redacted], 2019, M-674036-01-1) and KCA 7.1.1.1/06 ([redacted] P. & [redacted] D., 2019, M-674477-01-1) have been conducted to supplement the original soil studies. One of the original soil studies (KCA 7.1.1.1/01 ([redacted], 1994, M-174177-02-1) is still considered valid and acceptable but the other three studies conducted between 1982 to 1988 are proposed as supplementary data only as they do not meet current requirements. To fully define the fate of the molecule, one of the new studies KCA 7.1.1.1/06 ([redacted] P. & [redacted] D., 2019, M-674477-01-1) was conducted with [phenoxy-UL-^{14}C]-aclonifen.

The original anaerobic laboratory study was not considered acceptable in the previous EU review and Study KCA 7.1.1.2/02 ([redacted], H.P. & [redacted] 2011, M-404038-01-1) conducted with [U-^{14}C-aniline] labelled aclonifen was provided as new data not yet reviewed under uniform principles. To fully define the fate of the molecule, a new anaerobic study KCA 7.1.1.2/04 (M-687746-01-1) was conducted with [phenoxy-UL-^{14}C]-aclonifen. The soil photolysis of aclonifen has been investigated in Study KCA 7.1.1.3/01 ([redacted] A. & [redacted] R., 1994, M-174175-01-1) which was evaluated during the previous EU review and is still considered acceptable. Cleavage of aclonifen or its minor soil metabolite M-01 could lead to the formation of phenol (or hydroquinone from cleavage of M-01). A review of the published information on phenol and hydroquinone under anaerobic and photolytic conditions is provided in KCA 7.1.1.2/03 and KCA 7.1.1.3/02 ([redacted], 2019; M-676285-02-1).

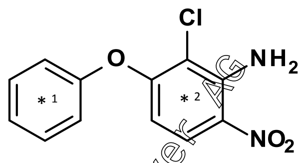
Terrestrial field dissipation studies with aclonifen, formulated as BANDUR®, a suspension concentrate containing 600 g/L aclonifen, were conducted at six trial sites in Europe (KCA 7.1.2.2.1/01, KCA 7.1.2.2.1/02 and KCA 7.1.2.2.1/03). These studies were evaluated during the previous EU review and are still considered as reliable to assess the rate of degradation of aclonifen under field conditions.

The half-lives determined for aclonifen indicate some persistence leading to residual residue levels remaining one year after application under Northern European climates. Consequently, accumulation studies (KCA 7.1.2.2.2/01 and KCA 7.1.2.2.2/02) were conducted to determine aclonifen levels in soil following annual applications over a three year period. No accumulation was observed throughout the studies.

Kinetic modelling assessments of laboratory aerobic soil and field studies according to FOCUS Degradation Kinetics (2006, 2014) are provided (KCA 7.1.2.1.1/07, [redacted] & [redacted], M-674934-01-1 and KCA 7.1.2.2.1/06, [redacted] & [redacted], M-675285-01-1).

The adsorption and desorption of aclonifen has been investigated in two studies, KCA 7.1.3.1.1/01 ([redacted], P., 1991, M-174332-01-1) which was evaluated during the previous EU review and is still considered valid and KCA 7.1.3.1.1/02, a new study for Annex 1 renewal submission ([redacted], E. & [redacted], 2019, M-562667-02-1).

The studies have investigated the fate and behaviour in soil following application of ^{14}C-aclonifen, uniformly labelled in either the phenoxy or aniline rings.



* Denotes position of [^{14}C]-radiolabel

1 = [Phenoxy-UL-^{14}C]-aclonifen   2 = [Aniline-UL-^{14}C]-aclonifen

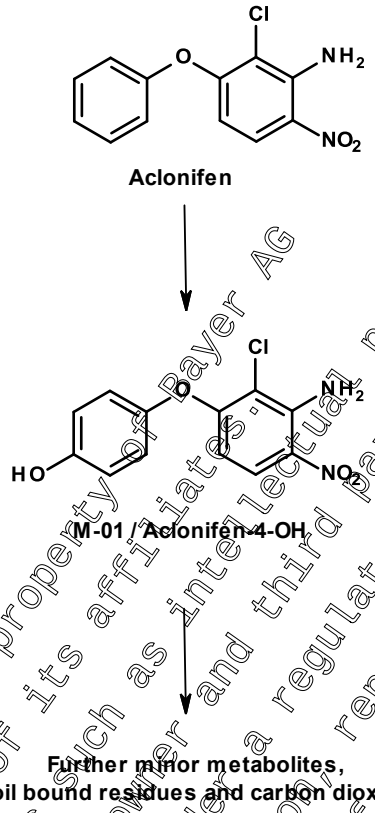
Microbial breakdown of aclonifen in soil leads to the formation of non-extractable soil bound residues, which accounted for a maximum of 20 to 58% of the applied [aniline-UL-^{14}C]-aclonifen and 42 to 71% of the applied [phenoxy-UL-^{14}C]-aclonifen, with very few intermediate products observed. Carbon dioxide was formed at maxima of between 1 to 12% AR in soil treated with the aniline label and 14 to 29% AR in soil treated the phenoxy label.

Supplemental studies have also been conducted to investigate the metabolism of aclonifen in soil under anaerobic and sterile conditions and to determine if photolysis contributed to the degradation of aclonifen on soil surfaces.

Under sterile conditions aclonifen was relatively stable confirming that its metabolism is largely microbially mediated. Non-extractable soil bound residues and material bound to aqueous soluble soil colloids were observed under sterile conditions at relatively constant levels throughout the incubation period, but at lower levels than observed in microbially viable soils, indicative of metabolites of aclonifen also binding to the soil matrix with time in microbially active soils. Aclonifen was more rapidly metabolised under flooded anaerobic conditions. Anaerobic metabolism of aclonifen led to the formation of non extractable soil residues indicating the metabolic pathway was similar to that observed under aerobic conditions. Under anaerobic conditions numerous minor unidentified metabolites were formed from the point when the redox potential in soil and water layer became reductive. The presence of light accelerated the rate of degradation on soil, with no unique metabolites formed exceeding 0.2% of applied radioactivity.

During the course of these studies, no metabolites have been observed at amounts > 5% of applied. The hydroxylated metabolite M-01 was detected at a maximum of 1.5%. Soil extracts were ultra-centrifuged to confirm polar material detected by TLC (but not by HPLC) was radioactive material bound to aqueous soluble soil colloids. This material was observed immediately after application at a maximum of 12 % AR in aerobic soil studies. Polar material retained on the origin of normal phase TLC plates (but not detected by HPLC) was also detected at a maximum of 4.4 % AR after normal extraction and 2.2 % AR after reflux) in a soil photolysis study. In later aerobic soil studies in the same soils, which were centrifuged to separate extracts from soil, only aclonifen was detected in soil extracts.

Figure 7-1 Metabolic pathway for aclonifen in soil



Aclonifen has been found to metabolise at a moderate rate in laboratory soil studies. DT_{50} values at 20ºC ranged from 35.3 to 252.3 days with a geometric mean of 79.1 days. The results have been normalised to standard temperature and soil moisture according to FOCUS recommendations prior to using in FOCUS groundwater and surface water exposure assessments.

Table 7.1-1: Summary of laboratory DT_{50} values for aclonifen

Compound	Laboratory Normalised DT_{50} (20 °C and pF2)		
	DT_{50} range (days)	Number of datasets (n)	Geometric mean DT_{50} (days) for exposure assessment
Aclonifen	35.3 – 252.3	12	79.1

Field experiments were conducted to investigate the behaviour of aclonifen under more realistic conditions. Aclonifen was found to have moderate rates of degradation under field conditions with DT_{50} values similar to those observed under laboratory conditions. To provide a conservative risk assessment, the worst-case field DT_{50} value of 196.8 days was used to calculate the predicted environmental concentration in soil, including $PEC_{soil\ accumulation}$. The half-lives determined for aclonifen indicate some persistence leading to residual residue levels remaining one year after application under Northern European climates. Consequently accumulation studies were conducted to determine aclonifen levels in soil following annual applications over a three year period. No accumulation was observed throughout the studies.

Table 7.1- 2: Summary of field DT_{50} values for aclonifen

Compound	Field dissipation $DisT_{50}$ (not normalised)		
	$DisT_{50}$ range (days)	Number of datasets (n)	Worst-case $DisT_{50}$ (days) for exposure assessment
Aclonifen	31.8 – 196.8	6	196.8

The adsorption/desorption characteristics of aclonifen and its minor soil metabolite M-01 were determined in standard batch equilibrium experiments. Standard adsorption / desorption studies indicated that aclonifen is immobile in soil.

Table 7.1- 3: Summary of soil adsorption coefficients for aclonifen and its metabolite M-01

Compound	K_{foc} (L/Kg) Geometric mean	Freundlich exponent (1/n) Arithmetic mean
Aclonifen	5727	0.88
M-01	4821	0.85

CA 7.1.1 Route of degradation in soil

Data Point:	KCA 7.1.1/01
Report Author:	[redacted]
Report Year:	2004
Report Title:	Aclonifen Metabolic fate in the environment: Cleavage of the diphenyl ether bond
Report No:	C034458
Document No:	M-234959-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Not applicable
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

KCA 7.1.1/01 is a position paper (M-234959-01-1, [redacted], 2004) prepared for the first inclusion of aclonifen into Annex I of Council Directive 91/414/EEC. It was evaluated, accepted and included in the Aclonifen Draft Assessment Report, Volume 3, B8 Environmental fate and behaviour data, November 2006. The document is a review of the environmental fate studies that have been conducted on aclonifen, focusing on the position of the radiolabel in the test material and the fate and behaviour in the environment of potential metabolites arising from cleavage of the diphenyl ether linkage. For procedural reasons it has to be included in the current dossier however it is now superseded as a soil study with [phenoxy-UL-^{14}C]-aclonifen has been conducted. Consequently, the document is now classified as invalid.

CA 7.1.1.1 Aerobic degradation

The route of aerobic degradation of aclonifen in soil has been investigated in four reliable studies in a total of nine different soils (12 soil datasets in total).

Report reference	Author, Year	Aniline Label	Phenoxy Label	Comment
KCA 7.1.1.1/01 M-174177-02-1	[redacted], 1994	✓	×	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.1.1.1/02 M-165109-01-1	[redacted], H., 1983	✓	×	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered as supporting data.
KCA 7.1.1.1/03 M-291254-01-1	[redacted], 2007	-	-	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered as supporting data.
KCA 7.1.1.1/04 M-558848-01-1	[redacted], 2016	✓	×	New data not yet reviewed under UP.
KCA 7.1.1.1/05 M-674036-01-1	[redacted] and [redacted], 2019	✓	×	New data not yet reviewed under UP.
KCA 7.1.1.1/06 M-674477-01-1	[redacted] P. & [redacted] D., 2019	×	✓	New data not yet reviewed under UP.

Study KCA 7.1.1.1/01 was evaluated during the previous EU review and is still considered as reliable to assess the aerobic degradation of aclonifen. Study KCA 7.1.1.1/02 was evaluated during the previous EU review and was considered acceptable to assess the aerobic degradation of aclonifen but not the anaerobic degradation. For reasons elaborated in the study summary is now considered only as supporting data to assess the aerobic degradation of aclonifen and not reliable for anaerobic degradation. Report KCA 7.1.1.1/03 is a statement on the extraction efficiency in the original aclonifen soil studies and is included as supporting data.

Studies KCA 7.1.1.1/04, KCA 7.1.1.1/05 and KCA 7.1.1.1/06 are provided as new data not yet reviewed under uniform principles.

Data Point:	KCA 7.1.1.1/01
Report Author:	[redacted]; [redacted]
Report Year:	1995
Report Title:	[14C]-Aclonifen: Soil degradation under various experimental conditions in accordance with the danish pesticide registration requirements
Report No:	R008566
Document No:	M-174177-02-1
Guideline(s) followed in study:	BBA: IV, 4-1
Deviations from current test guideline:	Current Guideline: OECD 307 (2002) Soil pH not reported
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of aclonifen applied at a rate of 3.16 mg/kg, equivalent to 2.4 kg/ha, was investigated in three soils for up to 118 days. The soils used were classified (USDA classification) as a clay loam ([redacted] Farm soil), a silt loam ([redacted] Field) and a loamy sand ([redacted]). The soils were incubated in the dark, at a moisture content equivalent to 60% of field capacity under aerobic conditions at 20 ºC. The radiochemical purity and specific activity of [U-^{14}C-aniline]-labelled aclonifen were ≥ 99 % and 3.46 MBq/mg, respectively. In addition, [redacted] soil was also incubated separately at a reduced moisture content (30% of field capacity), at a reduced application rate 0.33 mg/kg equivalent to 0.25 kg/ha and under sterile conditions.

Samples were taken for extraction and analysis immediately after treatment (day 0) and after 7, 24, 32, 45, 63, 94 and 118 days of incubation. Sterile samples were taken analysis at 0, 24, 45, 94 and 118 days. Soil samples were either exhaustively extracted at ambient temperature with acetonitrile / water (4 / 1, v/v) or soxhlet extracted with acetonitrile/acetone (4:1; v/v). Concentrated soil extracts were analysed by reverse phase high performance liquid chromatography (HPLC) and normal phase thin layer chromatography (TLC).

Material balance was 98.0 ± 8.9% (range = 91.5 to 106.9% of applied radioactivity, % AR). Extractable [^{14}C]-residues decreased from a maximum of 104.4% AR at day 0 to a minimum of 35.2% AR at day 118. Non extractable [^{14}C]-residues generally increased throughout the study, to reach approximately 57.6% AR by 118 days. At study termination, evolved $^{14}CO_2$ reached a maximum of 5.2% AR. Significant levels of organic volatiles were not observed.

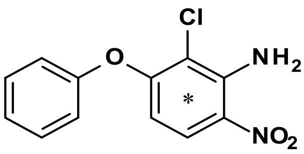
Parent compound decreased from a maximum of 88% AR at day 0 to a minimum of 30% AR at the end of the study. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit non-normalised DT_{50} values of 93.3, 54.6 and 82.2 days and DT_{90} values of >1000, 396.3 and 273.0 days for [redacted] Farm, [redacted] Field and [redacted] soils.

The only major degradation product detected was Unknown 1 (called M1 in the report), which represented radioactivity detected on the origin of thin layer chromatography plates and was detected in all soils at maxima of 9.9, 11.9 and 8.0% in [redacted] Farm, [redacted] Field and [redacted] soils, respectively. It was concluded that Unknown 1 was material bound to aqueous soluble soil colloids, not removed from the extracts prior to analysis. Unknown 1 was detected immediately after application at a maximum of 11.9%. At later timepoints Unknown 1 did not exceed 10%. Representative extracts were ultra-centrifuged and the amounts of solid radioactive material detected in the pellet were comparable to the amounts of radioactive origin material (Unknown 1) detected in both normal and reverse phase TLC. Three remaining metabolites M-01 (called RPA 407074 in the report), Unknowns 2 and 3 (called M2 and M3 in the report) formed in soil were minor and did not exceed 5% AR at two consecutive timepoints (maximum 1.5% AR) throughout the incubation period.

I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: [U-^{14}C-aniline]-aclonifen



* Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC) 2-chloro-6-nitro-3-phenoxy-aniline

CA registry number:	74070-46-5
Lot or batch number:	GXR302A
Specific activity:	3.46 MBq/mg
Radiochemical purity:	>99%
Stability of test compound:	Shown to be stable under the conditions of the test
Application vehicle:	Acetonitrile

2. Soil

Three agricultural soils collected from three sites in the United Kingdom in February 1994, [redacted] Farm (clay loam), [redacted] Field, (silt loam) and [redacted] (loamy sand). The soils were collected from the field in February 1994 sieved to 2 mm prior to use. Soils were collected from specific locations with no application of chemicals of the same class within at least 5 years. Specific details are shown below.

Table 7.1.1.1- 1: Physico-chemical characteristics of the soil used in aerobic soil study

Characteristic / Code	Units	[redacted] Farm	[redacted] Field	[redacted]
Soil code		94/8/2	94/9/2	94/10/2
Origin	Country	Essex, UK	Essex, UK	Suffolk, UK
Location		[redacted] Farm, [redacted]	[redacted] Farm, [redacted]	[redacted], [redacted][6]
Particle Size Analysis				
Total Sand (0.063 - 2.0 mm)	%	54.9	24.0	82.9
Silt (0.002 - 0.063 mm)	%	36.6	51.5	11.7
Clay (<0.002 mm)	%	8.5	24.5	5.4
Textural Class	DIN USDA	Silty sand Clay loam	Silty loam Silt loam	Silty sand Loamy sand
pH [2]		6.5 to 6.9	7.0	6.8
Organic Carbon	%	1.1	1.9	1.5
Cation Exchange Capacity	meq/100g	5.7	17.1	4.5
Moisture Content at Field Capacity (FC)	%	39.4	62.4	47.7
Soil Moisture During Incubation 60% FC	%	23.6	37.4	28.6
30% FC		n.a.	n.a.	14.31
Soil Microbial Biomass Initial	mg microbial C / 100 g soil	23.0	34.8	29.8
Final		12.1	28.4	17.6[3]
				23.9[4]
				18.5[5]

[1] Calculated from data in the report.
[2] Soil pH values were not reported but were obtained from historical records of soils collected from the same site for the first submission of aclonifen.
[3] Final soil microbial biomass of [redacted] soil treated at 2.4 kg/ha and incubated at 60% FC.
[4] Final soil microbial biomass of [redacted] soil treated at 0.25 kg/ha and incubated at 60% FC.
[5] Final soil microbial biomass of [redacted] soil treated at 2.4 kg/ha and incubated at 30% FC.
[1] Same location as [redacted] Farm on [redacted] Road, [redacted].

B. STUDY DESIGN AND METHODS

1. In-life dates:

23 March 1994 – 11 November 1994

2. Experimental design

Parameter		Description
Duration of test		118 Days
Soil condition		Soil sieved to 2 mm.
Application rate	Target	2.7 kg a.i./ha (assuming 1.5 g/cm^3 bulk density and depth of 5 cm)
	Actual	2.4 kg a.i./ha (assuming 1.5 g/cm^3 bulk density and depth of 5 cm)
Concentration in test system	Nominal	3.50 mg/kg
	Measured	3.16 mg/kg
Number of replications		A single flask
Test apparatus		1000 mL glass flasks containing 100 g dry weight equivalent of soil
Test material application	Identity of solvent	Acetonitrile
	Volume of application solution	1000 µL per 100 g soil dry weight
	Application method	Dropwise to soil surface by syringe and soil then mixed thoroughly
Traps for CO_2 and organic volatiles		A 1M potassium hydroxide trap followed by an ethylene glycol trap
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	20 ± 2 °C
	Moisture content	60% of field capacity. Moisture content checked and adjusted every 7 or 14 days during the experiment.
	Lighting	Dark
	Additional experiments	[redacted] soil was also incubated at 1) a reduced moisture content (30% of field capacity), 2) at a reduced application rate 0.33 mg/kg equivalent to 0.25 kg/ha and 3) under sterile conditions. Samples of soil intended for the sterile soil degradation experiment were autoclaved at 122 °C for 25 minutes and incubated in the same manner as non-sterile samples with incoming air passing through a filter to maintain the sterility of the samples. The sterility of the soil and trapping solutions was confirmed at each sampling point.

Sampling

Parameter		Description
Sampling intervals	Aerobic, non-sterile	0, 7, 24, 32, 45, 63, 94 and 118 DAT
	Aerobic, sterile	0, 24, 45, 94 and 118 DAT
	Untreated soils for biomass	Day 0 and at end of incubation
Soil sampling procedures		Complete treated samples were removed at each sampling time and extracted as detailed below.
Collection of CO_2 and volatile organics		Monitored weekly for the first thirty days, every second week thereafter to the end of the study. Volume of solutions measured and aliquots taken for LSC.

Analytical procedures

On day 0 and day 7 soil samples were extracted by soxhlet extraction with acetonitrile / water (4 / 1, v/v). At timepoints after this, soil samples were exhaustively extracted at ambient temperature with acetonitrile / water (4 / 1, v/v) with up to six extraction steps conducted. The individual extracts were quantified by LSC, pooled and then concentrated by rotary evaporation at 60°C. Aliquots of the concentrates were analysed by reversed-phase HPLC and/or TLC analyses.

Following extraction, soil samples were air-dried and homogenised, and the remaining unextracted radioactivity quantified by combustion.

With the exception of the zero time samples, trap solutions were removed for analysis at each sampling time. The volume of each trapping solution was measured and the radioactivity present was determined by LSC.

Degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.1.1/07. A brief summary of the approach for trigger endpoints is provided below.

The non-normalised data was best fit by the First Order Multiple Compartment (FOMC) model in [redacted] Farm and [redacted] Field soils, with $\chi 2$ errors of 8% and the Simple First Order (SFO) model in [redacted] soil with a $\chi 2$ error of 6%.

II. RESULTS AND DISCUSSION

The total recoveries and distribution of radioactivity are shown in detail in Table 7.1.1.1- 2 to Table 7.1.1.1- 7. The recoveries, extractable and non-extractable residue and trap contents at the beginning and end of the study are summarised below.

Mass Balance

Total radioactivity	Sum of activity in soil extracts, soil residue on combustion and that trapped as $^{14}CO_2$ and volatile organics in the 1M potassium hydroxide and ethylene glycol traps.
Recovery at 0 DAT	90.4 to 106.9% AR
Overall recovery (all samples)	Range 91.5 to 106.9%, mean 98.0% AR

Extractable residues aerobic non-sterile soil	Extractable residues declined with time in all soils.	
	Total extractable residues at 0 DAT	85.8 to 104.4% AR
	Total extractable residues at end of study (118 DAT)	35.2 to 85.0% AR
Bound residues aerobic non-sterile soil	Bound residues generally increased throughout the incubation period in all soils.	
	Bound residues at end of study (118 DAT)	7.2 to 57.6% AR
Extractable residues aerobic sterile soil	Extractable residues very slowly declined with time	
	Total extractable residues at 0 DAT	95.6% AR
	Total extractable residues at end of study (118 DAT)	91.6% AR
Bound residues	Bound residues very slowly increased throughout the incubation period	

aerobic sterile soil	Bound residues at end of study (118 DAT)	4.4% AR

Volatilisation

$^{14}CO_2$ aerobic non-sterile soil	Carbon dioxide evolution slowly increased throughout the incubation.	
	$^{14}CO_2$ evolved at end of study (118 DAT)	5.2% AR
Other volatiles	No other volatiles were observed (≤ 0.1% AR)	

$^{14}CO_2$ aerobic sterile soil	Carbon dioxide evolution slowly increased throughout the incubation.	
	$^{14}CO_2$ evolved at end of study (118 DAT)	0.2% AR
Other volatiles	No other volatiles were observed (< 0.1% AR)	

Soil treated at 2.4 kg/ha and incubated at 60% field capacity

The distribution of radioactivity was similar in all three soils, although towards the end of the study, extractability was greater in [redacted] Farm soil where the amount of extractable radioactivity had declined to 50% after 118 days, compared with *ca.* 36% in [redacted] Field soil and [redacted] soil (treated at 2.4 kg/ha and 60% field capacity).

[redacted] soil treated at reduced application rate

The majority of the applied radioactivity from [redacted] soil treated at an application rate of 0.25 kg/ha was extractable throughout the incubation period, and levels of extractable radioactivity only gradually decreased with time (with *ca.* 75% extractable at the end of the incubation period). The levels of unextractable radioactivity were lower than in the comparable experiment conducted at 2.4 kg/ha, reaching a maximum of 21 % after 118 days incubation in [redacted] soil.

[redacted] soil treated at reduced soil moisture content

Levels of extractable radioactivity from [redacted] soil treated at an application rate of 2.4 kg/ha and incubated at 30% field capacity declined from 104% at day 0 to 85% at day 118. Organic volatiles and carbon dioxide were detected at a maximum of 0.1% and 0.7%, respectively and non-extractable residues were ≤ 8.5% of the applied radioactivity.

[redacted] soil incubated under sterile conditions

Sterile samples were used in this study to determine the effect that microbial activity had on metabolism. No organic volatiles and virtually no CO_2 were detected in volatile traps. A range of 91.6% to 99.8% of the applied radioactivity was extractable throughout the incubation period. Non-extractable residues were ≤ 4% of the applied radioactivity.

Table 7.1.1.1- 2: Distribution of radioactivity in [redacted] Farm soil incubated under aerobic conditions
Application Rate: 2.4 kg/ha Moisture Content: 60% FC

	% of applied radioactivity							
DAT	**0**	**7**	**24**	**32**	**45**	**63**	**94**	**118**
Carbon Dioxide	na	<0.1	0.3	0.4	0.4	0.6	1.2	1.6
Organic Volatiles	na	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Total Extracted	86.6	92.6	77.0	57.7	54.9	48.2	65.0	50.3
Unextracted Soil Residues	11.3	12.8	20.6	32.3	40.9	47.8	32.6	42.5
Total	97.9	105.4	97.9	90.4	96.2	96.6	98.8	94.3
Overall Mean 97.2 ± 4.3								

na = not applicable

Table 7.1.1.1- 3: Distribution of radioactivity in [redacted] Field soil incubated under aerobic conditions
Application Rate: 2.4 kg/ha Moisture Content: 60% FC

	% of applied radioactivity							
DAT	**0**	**7**	**24**	**32**	**45**	**63**	**94**	**118**
Carbon Dioxide	na	0.1	0.8	1.4	2.8	3.1	4.6	5.2
Organic Volatiles	na	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Total Extracted	97.3	87.3	74.0	73.7	48.6	46.3	39.5	35.2
Unextracted Soil Residues	3.0	10.6	22.7	28.9	46.5	47.7	47.5	51.8
Total	100.2	97.9	97.5	104.1	96.8	97.1	91.7	92.2
Overall Mean 97.2 ± 4.0								

na = not applicable

Table 7.1.1.1- 4: Distribution of radioactivity in [redacted] soil incubated under aerobic conditions
Application Rate: 2.4 kg/h Moisture Content: 60% FC

	% of applied radioactivity							
DAT	**0**	**7**	**24**	**32**	**45**	**63**	**94**	**118**
Carbon Dioxide	na	<0.1	0.1	0.2	0.2	0.3	0.6	0.7
Organic Volatiles	na	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Total Extracted	92.4	88.7	83.1	80.0	75.9	58.5	55.9	36.8
Unextracted Soil Residues	6.0	8.2	15.1	15.9	23.3	40.3	41.6	57.6
Total	98.4	97.2	98.3	96.2	99.4	99.1	98.1	95.1
Overall Mean 97.7 ± 1.5								

na = not applicable

Table 7.1.1.1- 5: Distribution of radioactivity in [redacted] soil incubated under aerobic conditions
Application Rate: 0.25 kg/ha Moisture Content: 60% FC

	% of applied radioactivity							
DAT	**0**	**7**	**24**	**32**	**45**	**63**	**94**	**118**
Carbon Dioxide	na	0.1	1.5	1.7	1.8	2.0	2.3	2.4
Organic Volatiles	na	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Total Extracted	85.8	74.5	80.7	80.8	85.3	79.8	73.5	74.8
Unextracted Soil Residues	5.7	18.9	14.2	17.1	19.8	18.5	19.2	21.2
Total	91.5	93.5	96.3	99.6	106.9	100.4	95.2	98.5
Overall Mean 97.7 ± 4.8								

na = not applicable

Table 7.1.1.1- 6: Distribution of radioactivity in [redacted] soil incubated under aerobic conditions
Application Rate: 2.4 kg/ha Moisture Content: 30% FC

	% of applied radioactivity							
DAT	**0**	**7**	**24**	**32**	**45**	**63**	**94**	**118**
Carbon Dioxide	na	<0.1	0.1	0.1	0.1	0.1	0.1	0.7
Organic Volatiles	na	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Total Extracted	104.4	104.1	93.9	90.8	96.0	94.1	90.3	85.0
Unextracted Soil Residues	0.3	0.9	3.1	7.4	5.8	4.9	8.5	7.2
Total	104.7	105.0	97.0	98.4	102.0	99.2	99.0	93.0
Overall Mean 99.8 ± 4.0								

na = not applicable

Table 7.1.1.1- 7: Distribution of radioactivity in [redacted] soil incubated under sterile conditions
Application Rate: 2.4 kg/ha Moisture Content: 60% FC

	% of applied radioactivity				
DAT	**0**	**24**	**45**	**94**	**118**
Carbon Dioxide	na	<0.1	0.1	0.1	0.2
Organic Volatiles	na	<0.1	<0.1	<0.1	<0.1
Total Extracted	95.6	98.9	99.8	94.9	91.6
Unextracted Soil Residues	3.2	1.2	1.6	3.9	4.4
Total	98.8	97.1	101.5	98.9	96.2
Overall Mean 98.5 ± 2.0					

na = not applicable

Transformation of Parent Material

The characterisation of soil extracts is presented in Table 7.1.1.1- 8 to Table 7.1.1.1- 13.

Soils treated at an application rate of 2.4 kg/ha and incubated at 60% field capacity indicated that aclonifen was the principal radiolabelled component detected. Levels of parent accounted for 78 to 88% of applied radioactivity at day 0 and declined to 30 to 46% of applied radioactivity at termination

of the study at 118 days. In addition to parent material, the metabolite M-01 was tentatively identified at a single timepoint at 1.5 % of applied radioactivity in [redacted] soil.

Three other components, Unknowns 1, 2 and 3 (called M1, M2 and M3 in the report) were detected. Unknown 1, which represented radioactivity detected on the origin of thin layer chromatography plates, was detected in all soils at maxima of 9.9, 11.9 and 8.0% in [redacted] Farm, [redacted] Field and [redacted] soil, respectively. It was concluded that Unknown 1 was material bound to aqueous soluble soil colloids, not removed from the extracts prior to analysis. Unknown 1 was detected immediately after application at a maximum of 11.9 %. At later timepoints Unknown 1 did not exceed 10%. Representative extracts were ultra-centrifuged and the amounts of solid radioactive material detected in the pellet were comparable to the amounts of radioactive origin material (Unknown 1) detected in both normal and reverse phase TLC.

The unidentified metabolites Unknown 2 and Unknown 3 were detected in [redacted] Field and [redacted] soils at maxima of 1.0 and 1.1%, respectively and were not detected in [redacted] Farm soil.

Additional incubations with [redacted] soil

Aclonifen was the principal radiolabelled component detected in all experiments. Unknown 1, material bound to aqueous soluble soil colloids was detected in extracts of [redacted] soil tested at a reduced application rate, at reduced soil moisture content and under sterile conditions at a maximum of 10.0%, 5.1% and 4.6%, respectively. No other components or metabolites were detected in soil extracts apart from a single finding of M-01 (called RPA 407074 in the report) at 118 days (0.9 %) in soil incubated under sterile conditions.

Very little degradation of aclonifen was detected in sterile soil with 91% of applied radioactivity identified as parent at day 0 and 89% after 118 days. Chemical degradation did not therefore appear to be significant and demonstrated clearly that the compound is metabolised by soil microorganisms.

Table 7.1.1.1- 8: Characterisation of radioactivity in [redacted] Farm soil incubated under aerobic conditions Application Rate: 2.4 kg/ha Moisture Content: 60% FC

	% of applied radioactivity							
DAT	0	7	24	32	45	63	94	118
Total Extracted	86.6	92.6	77.0	57.7	54.9	48.2	65.0	50.2
Aclonifen	78.2	82.7	71.8	56.5	50.9	46.0	61.2	46.4
M-01 [A]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unknown 1 [B]	8.4	9.9	4.0	1.2	4.0	2.3	3.8	3.8
Unknown 2 [C]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unknown 3 [D]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

[A] Called RPA 407074 in the report
[B] Called M1 in the report. R_f of 0 in normal phase TLC system
[C] Called M2 in the report. R_f of 0.28 in normal phase TLC system
[D] Called M3 in the report. R_f of 0.48 in normal phase TLC system

Table 7.1.1.1- 9: Characterisation of radioactivity in [redacted] Field soil incubated under aerobic conditions Application Rate: 2.4 kg/ha Moisture Content: 60% FC

	% of applied radioactivity							
DAT	0	7	24	32	45	63	94	118
Total Extracted	97.3	87.3	74.0	73.7	48.6	46.3	39.5	35.2
Aclonifen	85.3	80.7	70.7	72.6	44.9	42.3	36.9	30.3
M-01 [A]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unknown 1 [B]	11.9	6.5	3.3	1.1	3.6	4.0	2.2	2.9
Unknown 2 [C]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.2	0.9
Unknown 3 [D]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.3	0.1

[A] Called RPA 407074 in the report
[B] Called M1 in the report. R_f of 0 in normal phase TLC system
[C] Called M2 in the report. R_f of 0.28 in normal phase TLC system
[D] Called M3 in the report. R_f of 0.48 in normal phase TLC system

Table 7.1.1.1- 10: Characterisation of radioactivity in [redacted] soil incubated under aerobic conditions Application Rate: 2.4 kg/ha Moisture Content: 60% FC

	% of applied radioactivity							
DAT	0	7	24	32	45	63	94	118
Total Extracted	92.4	88.7	83.1	80.0	75.9	58.5	55.9	36.8
Aclonifen	88.4	84.5	76.5	72.2	69.5	50.4	50.9	29.6
M-01 [A]	<0.1	<0.1	1.5	<0.1	<0.1	<0.1	<0.1	<0.1
Unknown 1 [B]	4.1	4.2	3.1	7.8	6.4	8.0	5.0	5.5
Unknown 2 [C]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	1.0
Unknown 3 [D]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.7

[A] Called RPA 407074 in the report
[B] Called M1 in the report. R_f of 0 in normal phase TLC system
[C] Called M2 in the report. R_f of 0.28 in normal phase TLC system
[D] Called M3 in the report. R_f of 0.48 in normal phase TLC system

Table 7.1.1.1- 11: Characterisation of radioactivity in [redacted] soil incubated under aerobic conditions Application Rate: 0.25 kg/ha Moisture Content: 60% FC

	% of applied radioactivity							
DAT	0	7	24	32	45	63	94	118
Total Extracted	85.8	74.5	80.7	80.8	85.3	79.8	73.5	74.8
Aclonifen	74.9	66.4	77.6	77.8	85.3	75.0	71.8	70.8
M-01 [A]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unknown 1 [B]	10.0	8.0	3.1	2.9	<0.1	4.8	1.7	4.0
Unknown 2 [C]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unknown 3 [D]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

[A] Called RPA 407074 in the report
[B] Called M1 in the report. R_f of 0 in normal phase TLC system
[C] Called M2 in the report. R_f of 0.28 in normal phase TLC system
[D] Called M3 in the report. R_f of 0.48 in normal phase TLC system

Table 7.1.1.1- 12: Characterisation of radioactivity in [redacted] soil incubated under aerobic conditions Application Rate: 2.4 kg/ha Moisture Content: 30% FC

	% of applied radioactivity							
DAT	**0**	**7**	**24**	**32**	**45**	**63**	**94**	**118**
Total Extracted	104.4	104.1	93.9	90.8	96.0	94.1	90.3	85.0
Aclonifen	99.3	99.0	93.9	89.2	91.9	91.0	89.4	80.9
M-01 [A]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unknown 1 [B]	5.1	5.1	<0.1	1.6	4.1	3.2	0.8	4.1
Unknown 2 [C]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Unknown 3 [D]	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1

[A] Called RPA 407074 in the report
[B] Called M1 in the report. R_f of 0 in normal phase TLC system
[C] Called M2 in the report. R_f of 0.28 in normal phase TLC system
[D] Called M3 in the report. R_f of 0.48 in normal phase TLC system

Table 7.1.1.1- 13: Characterisation of radioactivity in [redacted] soil incubated under sterile conditions Application Rate: 2.4 kg/ha Moisture Content: 60% FC

	% of applied radioactivity				
DAT	**0**	**24**	**45**	**94**	**118**
Total Extracted	95.6	95.9	99.8	94.9	91.6
Aclonifen	91.0	95.3	96.6	94.4	89.0
M-01 [A]	<0.1	<0.1	<0.1	<0.1	0.9
Unknown 1 [B]	4.6	0.6	3.2	0.5	1.7
Unknown 2 [C]	<0.1	<0.1	<0.1	<0.1	<0.1
Unknown 3 [D]	<0.1	<0.1	<0.1	<0.1	<0.1

[A] Called RPA 407074 in the report
[B] Called M1 in the report. R_f of 0 in normal phase TLC system
[C] Called M2 in the report. R_f of 0.28 in normal phase TLC system
[D] Called M3 in the report. R_f of 0.48 in normal phase TLC system

Aclonifen degraded at a moderate rate in the three soils. The reported DT_{50} values were 70 days, 76 days and 82 days, for [redacted] Farm, [redacted] Field and [redacted] soils respectively. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/07. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.1.1-14.

Table 7.1.1.1- 14: Degradation rate of aclonifen under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted] Farm	FOMC	97.9	α 0.23 β 4.76	8.42	n.r. n.r.	0.11 -8.40	0.45 17.9	93.3	>1000
[redacted] Field	FOMC	100.2	α 1.23 β 72.31	7.98	0.162. 0.236	-0.98 -110.3	3.44 255.0	54.6	396.3
[redacted]	SFO	98.4	k = 8.43e-03	5.7	5.55e-05	6.58e-03	0.01	82.2	273

n.r. not relevant

III. CONCLUSION

Aclonifen degraded at a moderate rate in non-sterile soils treated at an application rates of 2.4 kg/ha, incubated at 20 ºC and 60% field capacity (with 46.4%, 30.3% and 29.6% of the applied radioactivity remaining as aclonifen in [redacted] Farm, [redacted] Field and [redacted] soils, respectively, after 118 days. Less than 6% of the radioactivity was detected as $^{14}CO_2$, indicating slow mineralization of aclonifen to CO_2. Organic volatiles were not detected at significant amounts in any soil.

Aclonifen was shown to be relatively stable in sterile soil confirming that the degradation is largely microbially mediated. Microbial populations in soil will metabolise aclonifen leading to the formation of non extractable soil residues. Material bound to aqueous soluble soil colloids was observed (Unknown 1) at a range of 1 to 12%. Only very small amounts of extractable degradation products were detected. A small amount of metabolite M-01 was detected in [redacted] soil (24 days, 1.5 % of applied radioactivity) on one occasion in soil incubated under normal aerobic conditions. The radiolabelled tracer, [^{14}C], was located in the aniline ring and the presence of [^{14}C]-carbon dioxide (at up to 6%) confirmed aclonifen was ultimately mineralised with complete metabolism of the ring.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit non-normalised DT_{50} values of 93.3, 54.6 and 82.2 days for [redacted] Farm, [redacted] Field and [redacted] soils respectively.

Assessment and conclusion by applicant:

The study was conducted in accordance with Danish Requirements for Pesticide Registration No. 791 (1987). The study is considered valid to assess the aerobic degradation of [aniline-UL-^{14}C] aclonifen in soil.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.1.1/02
Report Author:	[redacted]
Report Year:	1983
Report Title:	Aerobic and anaerobic degradation of CME 127 in the soil
Report No:	R003641
Document No:	M-165109-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: OECD 307 (2002) Degradation products not quantified in all samples. Recoveries not quantitative.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Supportive only

Executive Summary

The route and rate of degradation of [aniline-UL-^{14}C]aclonifen was investigated in two soils, a European sandy loam soil freshly collected from an arable field and a German standard soil [redacted] 2.3, under laboratory aerobic conditions at 22 °C. [Aniline-UL-^{14}C] labelled aclonifen was applied to

soil samples at an application rate equivalent to 10.18 mg/kg for the [redacted] 2.3 soil and 9.98 mg/kg for the arable soil. The radiochemical purity and specific activity were ≥ 98 % and 2.80 MBq/mg respectively.

For the aerobic experiment, soil samples were incubated in the dark under static conditions at a moisture content equivalent to 40 % of the maximum soil water holding capacity, which was maintained throughout the course of the study. The production of carbon dioxide was monitored in separate 50 g samples of soil incubated under identical conditions in flasks fitted with a volatile trap (0.2 M KOH) for the collection of carbon dioxide. All flasks were incubated in the dark at 22 °C ± 2 °C. Duplicate samples of soil were taken for analysis after 0, 8, 30, 60 and 104 days from the [redacted] 2.3 soil and 0, 7, 38, 60 and 90 days from the arable soil. The potassium hydroxide in the carbon dioxide traps were changed at the same sampling times.

Soil samples incubated under aerobic conditions were exhaustively extracted with acetone, acetonitrile, methanol / water (80: 20 by volume), and water at ambient temperature. Radioactivity extracted from soil and in the volatile traps was quantified by liquid scintillation counting (LSC). The radioactivity contained in the potassium hydroxide traps was confirmed as [^{14}C]-carbon dioxide by acidification with hydrochloric acid. The remaining residue after completion of extractions was combusted to quantify non-extractable residue. Extracts of aerobic soil were analysed against reference standards by normal-phase thin-layer chromatography.

The majority of the radioactivity was initially extractable, with 96.9% to 100% of the recovered radioactivity was extracted at day 0. By the end of the study (day 104 for [redacted] 2.3 soil and day 90 for the arable soil), the levels of extractable radioactivity had declined to 56.5% and 41.0% of the recovered radioactivity, respectively. Non-extractable residues increased proportionately with the decrease in extractable radioactivity over the study period. The maximum amount of non-extractable residues was 40.9% (day 104) in the [redacted] 2.3 soil and 63.7% (day 60) in the arable soil. The levels of non-extractable soil residues at the final timepoint had declined to 56.4% in the arable soil (day 90). Radiolabelled carbon dioxide evolved accounted for a maximum of 3% of the applied radioactivity by the end of the study.

Aclonifen degraded at a moderate rate in arable soil and [redacted] 2.3 soil incubated under aerobic conditions. The degradation rates were similar to that measured in more recent, reliable studies.

The study deviates in a number of important aspects to the requirements of OECD 307 (2002). Recoveries of radioactivity were not quantitative in either soil throughout the study, ranging from 80 to 118% of applied radioactivity in the aerobic experiment and thus the study does not meet the quality criteria of OECD 307.

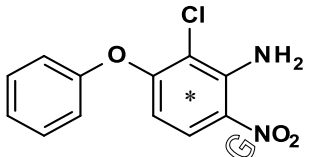
Up to 19% ([redacted] 2.3 soil after 30 days incubation) and 21% (Arable soil after 90 days incubation) of recovered radioactivity was characterised as extractable degradation products. Further degradation products could not be identified in the study (although a number of potential soil metabolites were included as analytical targets). The distribution and characterization of radioactivity was investigated only in selected extracts, one from each soil. It was established in these extracts, which contained the maximum amount of degradation products, that no single component exceeded 5% of the total radioactivity.

Consequently the study must be considered as supportive only.

I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: [U-^{14}C-aniline]-aclonifen



* Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC)	2-chloro-6-nitro-3-phenoxy-aniline
CA registry number:	74070-46-5
Lot or batch number:	Not stated
Specific activity:	2.80 MBq/mg Prior to treatment the specific activity of the radiolabelled material was adjusted to *ca.* 50 Bq/µg.
Radiochemical purity:	≥ 98% (TLC)
Stability of test compound:	Shown to be stable under the conditions of the test
Application vehicle:	Acetone

2. Soil Two soils, a European sandy loam soil collected from an arable field and a German standard sandy loam soil [redacted] 2.3 were collected from sites in Germany. Soils were air-dried and sieved to 2 mm prior to use. Specific details are shown below

Table 7.1.1.1- 15: Physico-chemical characteristics of the soils used in aerobic soil study

Characteristic / Code	Units	[redacted] 2.3 soil (Sp 382)	Arable Soil (42 82/83)
Origin	State, Country	Germany	Germany
Location	City or Township	[redacted]	[redacted]
Particle Size Analysis [1]			
>2.0 mm	%	40.8	33.9
Sand (0.02 - 2.0 mm)	%	37.8	53.9
Silt (0.002 - 0.02 mm)	%	12.6	8.1
Clay (<0.002 mm)	%	8.8	14.1
Particle Size Analysis [2]			
Sand (0.02 - 2.0 mm)	%	64	71
Silt (0.002 - 0.02 mm)	%	21	11
Clay (<0.002 mm)	%	15	19
Textural Class	DIN	Sandy loam	Sandy loam
pH		6.6	7.3
Organic Matter	%	0.92	1.48
Maximum Water Holding Capacit	%	34.2	33.5
Soil Moisture During Incubation	%	13.7	13.4
Soil Microbial Biomass (Initial)	µg microbial C / g soil	29.34	34.51

[1] Reported particle size analysis
[2] Recalculated particle size analysis excluding particles > 2mm

B. STUDY DESIGN AND METHODS

1. In-life dates:

17 January 1983 – 19 August 1983

2. Experimental design

Parameter		Description
Duration of test		104 days for the [redacted] 2.3 soil and 90 days for the arable soil
Soil condition		Soil was sieved to 2 mm and equilibrated to study conditions for 14 days.
Soil sample weight		200 g dry weight equivalents per replicate.
Concentration in test system mg/kg (ppm)		standard soil 2.3 : 10.18 arable soil: 9.98
Number of replications		Duplicate 10 g samples removed at each sampling interval.
Test apparatus		1000 mL Erlenmeyer glass flasks containing 200 g dry weight equivalent of soil.
Test material application	Identity of solvent	Acetone
	Volume of application solution used per treatment	2000 µL per 200 g soil dry weight
	Application method	Not stated
	Evaporation of application solvent	The test vessels were left open for 15 minutes to facilitate evaporation of application solvent the samples were intensively mixed with a shaking machine.

Parameter		Description
Traps for volatiles		The production of carbon dioxide was monitored in separate 50 g samples of soil incubated under identical conditions in flasks fitted with a volatile trap (0.2 M KOH) for the collection of carbon dioxide.
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	22 ± 2 °C
	Moisture content	40% MWHC
	Moisture maintenance method	Monitored every three days
	Continuous darkness (Yes/No)	Yes

Sampling

Parameter		Description
Sampling intervals		Duplicate samples of soil were taken for analysis after 0, 8, 30, 60 and 104 days from the [redacted] 2.3 soil and 0, 7, 38, 60 and 90 days from the arable soil.
Soil sampling procedures		10 g aliquots were removed at each sampling time and extracted as detailed below.
Collection of volatiles		The potassium hydroxide in the carbon dioxide traps were changed at the same sampling times.
Sampling intervals / times for	Moisture content	Monitored every three days
	Sterility checks	N/A
	Other	Soil microbial biomass was determined for untreated soil at DAT-0

Analytical procedures

Soil samples were exhaustively extracted with acetone, acetonitrile, methanol / water (80: 20 by volume), and water at ambient temperature. Radioactivity extracted from soil and in the volatile traps was quantified by liquid scintillation counting (LSC). The radioactivity contained in the potassium hydroxide traps was confirmed as [^{14}C]-carbon dioxide by acidification with hydrochloric acid. The remaining residue after completion of extractions was combusted to quantify non-extractable residue. Extracts of aerobic soil were analysed against reference standards by normal-phase thin-layer chromatography.

Degradation kinetics

Aclonifen degraded at a moderate rate in soil treated at application rates of 10 mg/kg and incubated under aerobic conditions. The reported half-life values were five to six weeks in arable soil and 14 weeks in [redacted] 2.3 soil. The rate of degradation of aclonifen was re-evaluated using KinGUI assuming simple first order kinetics for the first approval of aclonifen (EFSA (2008) 149).

II. RESULTS AND DISCUSSION

The distribution of radioactivity in a) mg/kg, b) as percentage of recovered radioactivity and c) as percentage of applied radioactivity following aerobic incubation are summarised in Table 7.1.1.1- 16 and Table 7.1.1.1- 17. Percentages of applied radioactivity have been calculated from data given in the report for this summary. The recoveries, extractable and non-extractable residue and trap contents at the beginning and end of the study are summarised below.

Mass Balance

Total radioactivity	Sum of activity in soil extracts, soil residue on combustion and that trapped as $^{14}CO_2$
Recovery at 0 DAT	86.5 and 88.5% AR [1]
Overall recovery (all samples)	80.0 – 118.1% AR and 85.4 - 99.0% AR

[1] Percentages of applied radioactivity (%AR) have been calculated from data given in the report for this summary

Extractable residues	Extractable residues declined with time	
	Total extractable residues at 0 DAT	86.7 and 85.8% AR
	Total extractable residues at end of study (90 & 104 DAT)	40.6 and 45.2% AR
Bound residues	Bound residues generally increased throughout the incubation period	
	Bound residues at end of study (90 & 104 DAT)	55.8 and 32.9% AR

Volatilisation

$^{14}CO_2$	Carbon dioxide evolution slowly increased throughout the incubation	
	$^{14}CO_2$ evolved at end of study (90 and 104 DAT)	2.1 and 2.6% AR
Other volatiles	Not collected	

For radiolabelled compounds the quality criteria in OECD 307 (2002) states mass balance recoveries should range from 90 to 110%. Although there was no systematic loss of radioactivity with time, the recovery of radioactivity at critical timepoints, such as the initial timepoint for both soils and, also at the final timepoint in the [redacted] 2.3 soil, was not quantitative. Largely for this reason the study cannot be considered as reliable

The majority of the radioactivity was initially extractable, with 96.9% to 100% of the recovered radioactivity extracted at day 0. By the end of the study (day 104 for [redacted] 2.3 soil and Day 90 for the arable soil), the levels of extractable radioactivity had declined to 56.5% and 41.0% of the recovered radioactivity, respectively.

Non-extractable residues increased proportionately with the decrease in extractable radioactivity over the study period. The maximum amount of non-extractable residues was 40.9% (day 104) in the [redacted] 2.3 soil and 63.7% (day 60) in the arable soil. The levels of non-extractable soil residues at the final timepoint had declined to 36.4% in the arable soil (day 90). Non-extractable soil residues were not solubilised with either hydrochloric acid (2N HCl, heated for 5 hours under reflux conditions) or sodium hydroxide (12.5 N NaOH, heated for 23 hours under reflux conditions). Radiolabelled carbon dioxide evolved accounted for a maximum of 3% of the applied radioactivity by the end of the study.

The findings are similar to those found in more recent, reliable studies conducted with [aniline-UL-^{14}C] labelled aclonifen.

Table 7.1.1.1- 16: Distribution of radioactivity in soil [redacted] 2.3 under aerobic conditions expressed as mg/kg (top), percentage of recovered radioactivity (middle) and percentage of applied radioactivity (bottom) [1]

A)	mg/kg				
DAT	0	8	30	60	104
Total Extracted	8.83	8.92	9.77	5.80	4.60
Unextracted Soil Residues	0	0.06	2.21	3.50	3.32
Carbon Dioxide	na	0.01	0.04	0.11	0.21
Total	8.83	8.99	12.02	9.41	8.14

na = not applicable

B)	% of recovered radioactivity				
DAT	0	8	30	60	104
Total Extracted	100	99.2	81.3	61.6	56.5
Unextracted Soil Residues	0.0	0.7	18.4	37.2	40.9
Carbon Dioxide	na	0.2	0.3	1.2	2.6
Total	100	100	100	100	100

na = not applicable

C)	% of applied radioactivity [1]				
DAT	0	8	30	60	104
Total Extracted	86.7	87.6	96.0	57.0	45.2
Unextracted Soil Residues	0.0	0.6	21.7	34.4	32.6
Carbon Dioxide	na	0.1	0.4	1.1	2.1
Total	86.7	88.3	118.1	92.4	80.0

na = not applicable
[1] Percentages of applied radioactivity have been calculated from data given in the report for this summary

Table 7.1.1.1- 17: Distribution of radioactivity in Arable soil under aerobic conditions expressed as mg/kg (top), percentage of recovered radioactivity (middle) and percentage of applied radioactivity [1]

A)	mg/kg				
DAT	0	7	38	60	90
Total Extracted	8.56	7.91	4.50	3.09	4.05
Unextracted Soil Residues	0.27	0.61	4.19	5.51	5.57
Carbon Dioxide	na	0.01	0.05	0.06	0.26
Total	8.83	8.52	8.74	8.65	9.88

na = not applicable

B)	% of recovered radioactivity				
DAT	0	7	38	60	90
Total Extracted	96.9	92.5	51.5	35.7	41.0
Unextracted Soil Residues	3.1	7.1	48.0	63.7	56.4
Carbon Dioxide	na	0.1	0.6	0.6	2.6
Total	100	100	100	100	100

na = not applicable

C)	% of applied radioactivity [1]				
DAT	0	7	38	60	90
Total Extracted	85.8	79.3	45.1	31.0	40.6
Unextracted Soil Residues	2.7	6.1	42.0	55.2	55.8
Carbon Dioxide	0.0	0.1	0.5	0.6	2.6
Total	88.5	85.4	87.6	86.7	99.0

na = not applicable
[1] Percentages of applied radioactivity have been calculated from data given in the report for this summary

Transformation of Parent Material

At the end of the aerobic incubation period at 22 °C, aclonifen degraded to 44.1% of the radioactivity recovered in the [redacted] 2.3 soil (104 days) and 20.1% in the arable soil (90 days). Further degradation products could not be identified in the study (although a number of potential soil metabolites were included as analytical targets). Up to 19% ([redacted] 2.3 soil after 30 days incubation) and 21% (Arable soil after 90 days incubation) of recovered radioactivity was characterised as extractable degradation products (see Table 7.1.1.1- 18 and Table 7.1.1.1-19).

Table 7.1.1.1- 18: Characterisation of radioactivity in soil [redacted] 2.3 under aerobic conditions expressed as mg/kg (top), percentage of recovered radioactivity (middle) and percentage of applied radioactivity (bottom) [2]

A)	mg/kg				
DAT	0	8	30	60	104
Total Extracted	8.83	8.92	9.77	5.80	4.60
Aclonifen	8.29	8.06	7.51	4.74	3.59
Degradation Products	0.54	0.86	2.26	1.06	1.01

B)	% of recovered radioactivity				
DAT	0	8	30 [1]	60	104
Total Extracted	100	99.2	81.3	61.6	56.5
Aclonifen	93.9	89.6	62.5	50.4	44.1
Degradation Products	6.1	9.6	18.8	11.2	12.4

[1] Further characterisation of degradation products

C)	% of applied radioactivity [2]				
DAT	0	8	30	60	104
Total Extracted	86.7	87.6	96.0	57.0	45.2
Aclonifen	81.4	79.2	73.8	46.6	35.3
Degradation Products	5.3	8.4	22.2	10.4	9.9

[2] Percentages of applied radioactivity have been calculated from data given in the report for this summary

Table 7.1.1.1- 19: Characterisation of radioactivity in Arable soil under aerobic conditions expressed as mg/kg (top), percentage of recovered radioactivity (middle) and percentage of applied radioactivity (bottom) [2]

A)	mg/kg				
DAT	0	7	38	60	90
Total Extracted	8.56	7.91	4.50	3.09	4.05
Aclonifen	7.52	7.30	3.68	2.39	1.98
Degradation Products	1.04	0.61	0.82	0.70	2.07

B)	% of recovered radioactivity				
DAT	0	7	38	60	90 [1]
Total Extracted	96.9	92.7	51.5	35.7	41.0
Aclonifen	85.1	85.6	42.1	27.6	20.1
Degradation Products	11.8	7.1	9.4	8.1	20.9

[1] Further characterisation of degradation products

C)	% of applied radioactivity [2]				
DAT	0	7	38	60	90
Total Extracted	85.8	79.3	45.1	31.0	40.6
Aclonifen	75.4	73.1	36.9	23.9	19.8
Degradation Products	10.4	6.1	8.2	7.0	20.7

[2] Percentages of applied radioactivity have been calculated from data given in the report for this summary

The proportion of polar material increased with polarity of the extraction solvent. The origin material isolated by analysis of methanol / water and water extracts from the 90 day Arable soil sample was combined (representing the maximum amount observed in all soil samples at 14.6%) and characterised as a number of polar degradation products by sequential TLC analysis. Initially the polar material was separated into three components (Z1, Z2 and Z3) based on their retention in a TLC solvent system using dioxane /water mobile phase. Each of the three components was quantified and two were isolated (Z1 and Z3) before further fractionation based a TLC solvent system using acetone /water mobile phase. Both of these components were further separated into three components (Z1Z1, Z1Z2, Z1Z3 and Z3Z1, Z3Z2, Z3Z3). No single component exceeded 5% of the total radioactivity (see Table 7.1.1.1- 20 and Table 7.1.1.1- 21).

Table 7.1.1.1- 20: Further characterisation of extractable radioactivity in selected samples incubated under aerobic conditions

A) [redacted] 2.3 soil, 30 days incubation (% of recovered radioactivity)

Solvent	Total Extracted	Aclonifen	Origin	Remainder [3]
Acetone	59.7	54.3	2.6	2.8
Acetonitrile	4.0	2.8	0.6	0.6
Methanol/Water	10.2	4.2	4.7	1.3
Water	7.4	1.2	6.1	<0.1

B) Arable soil, 90 days incubation (% of recovered radioactivity)

Solvent	Total Extracted	Aclonifen	Origin [2]	Remainder [3]
Acetone	22.2	18.3	3.5	0.4
Acetonitrile [1]	0.4	na	na	0.4
Methanol/Water	9.5	1.8	6.5 [2]	1.2
Water	8.9	0	8.1 [2]	0.8

[1] Not given in report, calculated from total recovery
[2] Origin material combined prior to further characterisation (see Table 7.1.1.1- 21)
[3] Background radioactivity
na = not analysed

Table 7.1.1.1- 21: Further characterisation of origin material, Arable soil, 90 days incubation (% of recovered radioactivity)

Origin	Solvent System			
	Dioxane/water		Acetone/water	
14.6 [1]	Z1	2.3%	Z1Z1	0.5
			Z1Z2	0.9
			Z1Z3	0.9
	Z2	1.8%	na	na
	Z3	10.5%	Z3Z1	4.1
			Z3Z2	4.8
			Z3Z3	1.6

[1] Combined origin material from methanol/water and water extracts (see B, Table 7.1.1.1- 20)
na = not analysed further

Aclonifen degraded at a moderate rate in soil treated at application rates of 10 mg/kg and incubated under aerobic conditions. The reported half-life values were five to six weeks in arable soil and 14 weeks in [redacted] 2.3 soil. The rate of degradation of aclonifen was re-evaluated using KinGUI assuming simple first order kinetics for the first approval of aclonifen (EFSA (2008) 149). The degradation rates are similar to those measured in more recent, reliable studies.

Table 7.1.1.1- 22: Degradation rate of aclonifen under aerobic conditions at 22 ºC

Soil	M0 (mg/kg)	DT_{50} (days)	DT_{90} (days)	chi^2	t-test
Standard Soil 2.3	8.90	78.0	259.0	4.8	0.0026, >99%
Arable	8.59	32.2	106.8	10.1	0.0068, >99%

III. CONCLUSION

The route and rate of degradation of aclonifen was investigated in two soils, a German arable soil and [redacted] 2.3 soil, under laboratory aerobic and anaerobic conditions at 22 ºC. The study was criticized during the previous EU review and was not considered acceptable to assess the anaerobic degradation of aclonifen. The aerobic investigations conducted in the study also deviate in a number of important aspects to the requirements of OECD 307 (2002) and consequently is now considered as supportive data only.

Assessment and conclusion by applicant:

This study is non-GLP and was not conducted according to any stated guidance. The study results deviate in a number of important aspects to the requirements of OECD 307 (2002) in particular the mass balances are not quantitative. The study is not considered reliable to assess the aerobic degradation of [aniline-UL-^{14}C] aclonifen in soil and is included as supporting data only.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.1.1/03
Report Author:	[redacted]
Report Year:	2007
Report Title:	Extraction efficiency in aclonifen soil studies
Report No:	M-291254-01-1
Document No:	M-291254-01-1
Guideline(s) followed in study:	not specified
Deviations from current test guideline:	Not applicable
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/officially recognised testing facilities
Acceptability/Reliability:	Supportive only

Executive Summary

This paper examines the extraction techniques used in soil aerobic laboratory studies conducted with aclonifen in which high non-extracted residues were detected, to assess the robustness of the extraction methods and the accountability of the analytical method used in field studies.

Aclonifen was fully extracted by the methods employed in soil laboratory studies. Microbial breakdown of aclonifen in soil leads to the formation of non-extractable soil residues. The formation of non-extractable residues in soil from aclonifen is a result of microbial activity, as NER is not formed in sterile soil to any significant level (≤ 4% AR throughout the sterile incubation period).

Extraction techniques used in soil studies included extractions conducted at room temperature and reflux extractions, including soxhlet extractions at high temperature. The initial extraction step was extraction with organic solvent, as this is generally very efficient at extracting aclonifen. Reflux extraction with acid and base failed to solubilised residues remaining unextracted after repeated

extraction with non-polar and polar solvents, and it is concluded the remaining residues are non-extractable, that is they cannot be extracted without significantly changing their chemical nature.

An assessment of the accountability of the analytical method used in field studies indicates > 80% of aclonifen residues will be extracted.

I. MATERIALS AND METHODS

Aclonifen was fully extracted by soil extraction methods employed in each study and the remaining residues are non extractable. The extraction techniques used in soil studies are summarised in Table 7.1.1.1- 23. For clarity, the extraction methods have been summarised as ambient extractions conducted at room temperature and reflux extractions, including soxhlet extractions. Significant efforts to extract soil residues were undertaken in the studies. In soil degradation studies the initial extraction step is extraction with organic solvent, as this is generally very efficient at extracting aclonifen.

Table 7.1.1.1- 23: Soil Extraction Methods

Report	Study Type	Ambient Extraction	Reflux Extraction
KCA 7.1.1.1/01 M-174177-02-1 [redacted], 1994	Aerobic soil	1) acetonitrile/water (4/1, v/v) (x 6)	1) acetonitrile/water (4/1, v/v) [1]
KCA 7.1.1.1/02 M-165109-01-1 [redacted], 1983	Aerobic soil Anaerobic soil	1) acetone (x 2) 2) acetonitrile (x 2) 3) methanol/water (4/1, v/v) (x 2) 4) water (x 2)	1) 2N HCl [2] 2) 12.5N NaOH [2]
KCA 7.1.2.1.1/06 M-174228-01-1 [redacted] *et al.*, 1988	Aerobic soil	1) dichloromethane/acetone (2/3, v/v) (x 2)	
KCA 7.1.2.1.1/03 M-165114-01-1 Anonymous, 1982	Aerobic soil	1) acetone 2) acetone/water 3) methanol [3] 4) methanol/dilute HCl (1/1, v/v, pH 2.5) [3] 5) methanol/1% NaOH (2/1, v/v) [3]	

[1] Day 0 and Day 7 only
[2] Selected samples
[3] Day 112 only

II. RESULTS AND DISCUSSION

Microbial breakdown of aclonifen in soil leads to the formation of non-extractable soil residues. Further metabolism or co-metabolism by soil micro-organisms, leads to the formation of carbon dioxide, with very few intermediate products observed. The levels of non-extractable residues do not exceed the EU criteria of 70% in any soil tested and levels of carbon dioxide although low, reached 5% in one soil under laboratory conditions.

It has been shown the formation of non-extractable residues in soil is a result of microbial activity, as they do not form in sterile soil (KCA 7.1.1.1/01, M-174177-02-1, [redacted], 1994). In sterile soil 92 to 100% of the applied radioactivity was extracted and ≥ 89% identified as aclonifen throughout the incubation period (118 days). Non-extracted residues were ≤ 4% of the applied radioactivity throughout the sterile incubation period.

Aclonifen was fully extracted by the methods employed in soil laboratory studies. Reflux extraction with acid and base failed to solubilised residues remaining unextracted after repeated extraction with non-polar and polar solvents, and it is concluded the remaining residues are non-extractable, that is

they cannot be extracted without significantly changing their chemical nature. Vigorous extraction procedures confirmed that these bound residues will not be readily bio-accessible and are immobile in soil.

The analytical method used to analyse soil samples from field studies involved extraction of soil with acetone in the presence of hyflosupercel. Final analysis was by gas chromatography with electron capture detection (GC/ECD) or by gas chromatography with mass spectroscopic detection (GC/MS). An assessment of the accountability of the analytical method used in field studies indicates > 80% of aclonifen residues will be extracted.

III. CONCLUSION

The extraction techniques employed in soil laboratory studies were robust and the method used to extract field soil samples was sufficiently exhaustive.

Assessment and conclusion by applicant:

The position paper is considered valid to aid assessment of the extraction efficiency of aclonifen in soil.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.1.4.04
Report Author:	[redacted]
Report Year:	2016
Report Title:	[aniline-UL-14C]aclonifen: Aerobic degradation / metabolism in one soil
Report No:	EnSa-15-0010
Document No:	M-558848-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 307 Commission Regulation (EU) No 283/2013 in accordance with Regulation (EC) No 1107/2009 US EPA OCSPP Test Guideline No. 835.4100 / 835.4200 Japanese MAFF Test Guidelines 12 Nousan 8147, No. 2-5-2
Deviations from current test guideline:	Current guideline: OECD 307 (2002) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of [aniline-UL-^{14}C]-aclonifen applied at a rate of 6.40 mg/kg, equivalent to 2.4 kg/ha, was investigated in a single soil for up to 120 days. The soil was classified (USDA classification) as a sandy loam with a pH of 4.9 and an organic carbon content of 1.7%. The soil was incubated in the dark, at a moisture content equivalent to 55 ± 5% of the maximum water

holding capacity under aerobic conditions at 20 ºC. The radiochemical purity and specific activity of [U-^{14}C-aniline] labelled aclonifen were >99 % and 6.59 MBq/mg, respectively.

The test was performed in static systems consisting of Erlenmeyer flasks each containing 100 g soil (dry weight equivalents) and equipped with traps (permeable for oxygen) for the collection of carbon dioxide and volatile organic compounds.

Duplicate samples were taken for extraction and analysis immediately after treatment (day 0) and after 2, 5, 7, 14, 35, 61, 89 and 120 days of incubation. At each sampling interval, the soil was extracted three times at ambient temperature using acetonitrile/water 4/1 (v/v). Furthermore, two microwave assisted extraction steps were performed using acetonitrile/water 4/1 (v/v) at 70 °C and methanol/aqueous 0.1 N HCl 1/1 (v/v) at 50 °C. The amounts of test item and degradation products in soil extracts were determined by liquid scintillation counting (LSC) and by TLC/HPLC/radiodetection analysis. The amounts of volatiles and non-extractable residues were determined by LSC and combustion/LSC, respectively.

Material balance was 98.4 ± 2.1% (range = 96.2 to 103.1% of applied radioactivity, % AR). Extractable [^{14}C]-residues decreased from a maximum of 104.4% AR at day 0 to a minimum of 46.5% AR at day 120. Non extractable [14C]-residues gradually increased throughout the study, to reach approximately 38.7% AR by 120 days. At study termination, evolved 14CO2 reached a maximum of 12.1% AR. Significant levels of organic volatiles were not observed.

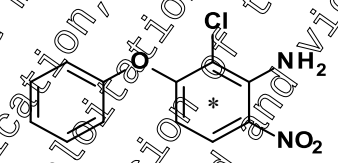
Parent compound decreased from a maximum of 102.6% AR at day 0 to a minimum of 43.1% AR at the end of the study. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a best-fit non-normalised DT_{50} value of 95.9 days and a DT_{90} value of 387.2 days for Wurmwiese soil.

Besides the formation of carbon dioxide, no major degradation products occurred in this study. The total unidentified residues amounted to a maximum of 3.4% AR and no single component exceeded 2.0% AR at any sampling interval.

I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: [U-^{14}C-aniline]-aclonifen



* Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC)	2-chloro-6-nitro-3-phenoxy-aniline
CA registry number:	74070-46-5
Lot or batch number:	KML 9794
Specific activity:	6.59 MBq/mg
Radiochemical purity:	>99% (HPLC with radioactivity detector)
Stability of test compound:	Shown to be stable under the conditions of the test
Application vehicle:	Methanol

2. Soil

A single agricultural sandy loam was collected from a site in Germany where there had been no application of plant production products in the least 5 years. The soil was collected fresh from the field (upper horizon of 0 to 20 cm) in February 2015 and sieved to 2 mm prior to use. Specific details are shown below.

Table 7.1.1.1- 24: Physico-chemical characteristics of the soil used in aerobic soil study

Characteristic / Code	Units	[redacted] Hof Wurmwiese	
Origin	Country	Germany	
Location	City or Township	[redacted], North Rhine-Westphalia	
GPS Coordinates		[redacted]	
Soil Taxonomic Classification (USDA)		Loamy, mixed, mesic Typic Argudalf	
Particle Size Analysis			
Sand (50 - 2 mm)	%	35	
Silt (2 - 50 µm)	%	27	
Clay (< 2 µm)	%	18	
Textural Class	USDA	Sandy loam	
pH			
soil/0.01 M $CaCl_2$ 1/2		4.9	
soil/water 1/1		5.1	
saturated paste		5.2	
soil/1 N KCl 1/1		4.5	
Organic Carbon	%	1.7	
Cation Exchange Capacity	meq/100g	9.6	
Bulk density (disturbed)	g/cm³	1.03	
Water Holding Capacity Maximum			
maximum (MWHC)		64.1 g H_2O ad 100 g DW	
at 1/10 bar (pF 2.0)	%	30.2	
Soil Microbial Biomass		[1]BIO-	[2]BIO+
Initial	mg microbial C / kg soil	505	
DAT - 61		339[3]	402
DAT - 120		529[4]	662

[1] BIO- samples were left untreated.
[2] BIO+ samples were applied with solvent of application solution (200 µL methanol).

B. STUDY DESIGN AND METHODS

1. In-life dates:

09 March 2016 – 30 June 2016

2. Experimental design

Parameter	Description
Duration of test	120 Days
Soil condition	Fresh field samples sieved to 2 mm and equilibrated to study conditions for 8 days.

Parameter		Description
Soil sample weight		100 g dry weight equivalents per replicate.
Concentration in test system	g test item/ha	Nominal: 2400 (based on maximum single field application rate of 2400 g/ha)
	µg test item/kg soil DW	Nominal: 6400 (actual:6286)
Control conditions (if used)		Samples for determination of soil microbial biomass: native soil with and without application solvent.
Number of replications		Duplicate samples for each sampling interval.
Test apparatus		300 mL Erlenmeyer glass flasks containing 100 g dry weight equivalent of soil.
Test material application	Identity of solvent	Methanol
	Volume of application solution used per treatment	204 µL per 100 g soil dry weight
	Application method	Dropwise application to the soil surface using an adjustable pipette.
	Evaporation of application solvent	The test vessels were left open for 15 minutes to facilitate evaporation of application solvent.
Traps for volatiles		The traps were filled with soda lime and polyurethane foam plug. The traps were permeable for oxygen.
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	20.39 °C
	Moisture content	53.8% MWHC
	Moisture maintenance method	Re-weighing and addition of lost water.
	Continuous darkness (Yes/No)	Yes

Sampling

Parameter		Description
Sampling intervals		Duplicate samples were processed and analysed 0, 2, 5, 7, 14, 35, 61, 89 and 120 days after treatment (DAT).
Soil sampling procedures		Complete treated samples were removed at each sampling time and extracted as detailed below.
Collection of volatiles		Soda lime for absorption of carbon dioxide and polyurethane foam for adsorption of volatile organic compounds.
Sampling intervals / times for	Moisture content	Each sampling interval.
	Sterility checks	N/A.
	Other	Soil microbial biomass was determined for untreated soil at DAT-0, DAT-61 and DAT-120, as well as for soil treated with application solvent at DAT-61 and DAT-120
Sample storage before analysis		The soils were processed immediately after sampling; first HPLC/radiodetection analysis of soil extracts was performed within three days.

Analytical procedures

At each sampling interval, the soil was extracted three times at ambient temperature using acetonitrile/water 4/1 (v/v). Furthermore, two microwave assisted extraction steps were performed using acetonitrile/water 4/1 (v/v) at 70 °C and methanol/aqueous 0.1 N HCl 1/1 (v/v) at 50 °C. The

amounts of test item and degradation products in soil extracts were determined by liquid scintillation counting (LSC) and by TLC and HPLC analysis with radiodetection. The amounts of volatiles and non-extractable residues were determined by LSC and combustion followed by LSC, respectively.

Following extraction, soil samples were lyophilised and homogenised, and the remaining unextracted radioactivity quantified by combustion.

With the exception of the zero time samples, trap attachments were removed for analysis at each sampling time. Carbon dioxide absorbed by soda lime was liberated and trapped in a scintillation cocktail selective for binding of carbon dioxide using an air-tight assembly. For this purpose, the soda lime of the trap attachments was transferred into an Erlenmeyer flask which was then attached with a dropping funnel, containing aqueous hydrochloric acid (18%), and connected to a series of two trapping vessels, each filled with ice-cooled scintillation cocktail. The aqueous hydrochloric acid was added dropwise under continuous stirring and the liberated carbon dioxide was purged into the trapping vessels by a stream of nitrogen. The radioactivity contents of these vessels were determined by LSC and summed to determine the total radioactivity liberated from soda lime.

The PU foam plug was extracted with ethyl acetate for approximately 30 minutes in an ultrasonic bath to desorb any volatile organic compounds. The radioactivity content was determined by LSC.

Degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. Additionally, DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/07. A brief summary of the approach for trigger endpoints is provided below.

The non-normalised data was best fitted by the Double First Order in Parallel (DFOP) model in Wurmweise soil with a $\chi 2$ error of 2%.

II. RESULTS AND DISCUSSION

The total recoveries and distribution of radioactivity are shown in detail in Table 7.1.1.1- 25. The recoveries, extractable and non-extractable residue and trap contents at the beginning and end of the study are summarised below.

Mass Balance

Total radioactivity	Sum of activity in soil extracts, soil residue on combustion and that trapped as $^{14}CO_2$ and volatile organics in the volatile traps.
Recovery at 0 DAT	101.6 and 104.5% AR
Overall recovery (all samples)	Range 95.8 to 104.5%, mean 98.4% AR

Extractable residues	Extractable residues declined with time.	
	Total extractable residues at 0 DAT	101.2 and 104.0% AR
	Total extractable residues at end of study (120 DAT)	46.2 and 46.8% AR
Bound residues	Bound residues generally increased throughout the incubation period.	
	Bound residues at end of study (120 DAT)	37.9 and 39.5% AR

Volatilisation

$^{14}CO_2$	Carbon dioxide evolution slowly increased throughout the incubation.	
	$^{14}CO_2$ evolved at end of study (120 DAT)	12.1% AR
Other volatiles	No other volatiles were observed (≤ 0.1% AR)	

Extractable residues gradually decreased from 102.6% at zero time to 46.5% AR at day 120 in [redacted] Hof Wurmwiese sandy loam soil. Non-extractable residues concurrently increased from 0.5 % at zero time to 38.7% at day 120 in the [redacted] Hof Wurmwiese sandy loam soil. The maximum amount of carbon dioxide was 12.1% AR at study end (day 120) in [redacted] Hof Wurmwiese soil. Formation of volatile organic compounds was insignificant as demonstrated by values of < 0.1% AR at all sampling intervals.

Table 7.1.1.1- 25: Distribution of radioactivity in soil [redacted] Hof Wurmwiese under aerobic conditions expressed as percentage of applied radioactivity

	Replicate No.	DAT								
		0	**2**	**5**	**7**	**14**	**35**	**61**	**89**	**120**
Volatiles										
Carbon Dioxide	A	n.a.	0.1	0.3	0.3	1.1	3.5	6.1	7.6	12.2
	B	n.a.	0.1	0.2	0.4	1.0	3.1	4.5	8.4	11.9
	Mean	**n.a.**	**0.1**	**0.3**	**0.4**	**1.1**	**3.3**	**5.3**	**8.0**	**12.1**
Volatile Organic Compounds	A	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	B	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mean	**n.a.**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**
Total Volatiles	A	n.a.	0.1	0.3	0.3	1.1	3.5	6.1	7.6	12.2
	B	n.a.	0.1	0.2	0.4	1.0	3.1	4.5	8.4	11.9
	Mean	**n.a.**	**0.1**	**0.3**	**0.4**	**1.1**	**3,3**	**5.3**	**8.0**	**12.1**
Extractable Residues										
Ambient Extract 1	A	101.2	89.3	84.1	84.9	77.3	49.3	55.7	48.8	34.2
	B	98.4	94.1	85.5	84.9	76.5	49.0	63.4	45.0	35.8
	Mean	99.8	91.7	84.8	84.9	76.9	49.1	59.4	46.9	35.0
Ambient Extract 2	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
Microwave Extract 1	A	2.2	4.4	29	3.3	3.8	12.9	2.3	3.6	4.6
	B	2.2	3.1	2.9	3.5	3.4	14.5	2.9	4.2	3.8
	Mean	**2.2**	**3.7**	**2.9**	**3.4**	**3.6**	**13.7**	**2.6**	**3.9**	**4.2**
Microwave Extract 2	A	0.6	1.6	1.9	1.8	3.0	6.8	5.6	6.0	7.4
	B	0.6	1.3	1.8	2.2	3.0	6.9	4.8	5.8	7.2
	Mean	**0.6**	**1.4**	**1.9**	**2.0**	**3.0**	**6.9**	**5.2**	**5.9**	**7.3**
Microwave Extract 3	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**
Total Extractable Residues	A	104.0	95.3	88.9	90.0	84.1	69.0	63.6	58.3	46.2
	B	101.2	98.5	90.3	90.6	82.9	70.4	71.1	55.0	46.8
	Mean	**102.6**	**96.9**	**89.6**	**90.3**	**83.5**	**69.7**	**67.3**	**56.7**	**46.5**
Non - Extractable Residues	A	0.5	3.4	6.7	7.3	12.9	24.6	28.8	30.9	39.5
	B	0.4	3.3	6.0	9.1	13.2	23.3	22.8	32.8	37.9
	Mean	**0.5**	**3.3**	**6.3**	**8.2**	**13.1**	**24.0**	**25.8**	**31.8**	**38.7**
Material Balance	A	104.5	98.7	95.8	97.6	98.2	97.1	98.5	96.8	97.9
	B	101.6	101.9	96.5	100.1	97.1	96.8	98.4	96.3	96.6
	Mean	**103.1**	**100.3**	**96.2**	**98.9**	**97.7**	**96.9**	**98.4**	96.6	**97.3**
Overall Mean 98.4 ± 2.1										

DAT: days after treatment, n.a. : not applicable

Transformation of Parent Material

The characterisation of soil extracts is presented in Table 7.1.1.1- 26. Analysis indicated that aclonifen was the principal radiolabelled component detected. Levels of parent accounted for 102.6% of applied radioactivity at day 0 and declined to 43.1% of applied radioactivity at termination of the study at 120 days. The total unidentified residues amounted to a maximum of 3.4% AR and no single component exceeded 2.0% AR at any sampling interval.

Table 7.1.1.1- 26: Characterisation of radioactivity in soil [redacted] Hof Wurmwiese under aerobic conditions expressed as percentage of applied radioactivity

Compound		DAT								
		0	2	5	7	14	35	61	89	120
Aclonifen	Mean	102.6	96.9	89.9	90.1	82.9	68.6	65.5	54.2	43.1
	SD	± 1.4	± 1.6	± 0.8	± 0.3	± 0.6	± 0.7	± 4.1	± 1.7	± 0.6
Unknown 1	Mean	n.d.	n.d.	< LOD	0.2	0.3	0.6	0.6	0.9	2.0
	SD				± 0.1	± 0.0	± 0.0	± 0.1	± 0.0	± 0.0
Unknown 2	Mean	n.d.	n.d.	n.d.	n.d.	0.3	0.5	0.6	0.9	1.2
	SD					± 0.0	± 0.0	± 0.1	± 0.0	± 0.1
Diffuse Residues	Mean	n.d.	n.d.	n.d.	n.d.	< LOD	n.d.	0.6	0.7	0.2
	SD							± 0.2	± 0.0	± 0.0
Sum of Unid./Diff. Residues[1]	Mean	n.d.	n.d.	< LOD	0.2	0.6	1.1	1.8	2.5	3.4
	SD				± 0.1	± 0.1	± 0.0	± 0.4	± 0.0	± 0.3
Total Extractable Residues[2]	Mean	102.6	96.9	89.6	90.3	83.5	69.7	67.3	56.7	46.5
	SD	± 1.4	± 1.6	± 0.7	± 0.3	± 0.7	± 0.7	± 3.7	± 1.7	± 0.3
Carbon Dioxide[3]	Mean	n.a.	0.1	0.3	0.4	1.1	3.3	5.3	8.0	12.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.2	± 0.8	± 0.4	± 0.2
Volatile Organic Compounds[3]	Mean	n.a.	0.1	0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues[3]	Mean	0.5	3.3	6.3	8.2	13.1	24.0	25.8	31.8	38.7
	SD	± 0.0	± 0.1	± 0.4	± 0.9	± 0.1	± 0.6	± 3.0	± 1.0	± 0.8
Total Recovery	Mean	103.1	100.3	96.2	98.8	97.7	96.9	98.4	96.5	97.2
	SD	± 1.4	± 1.7	± 0.3	± 1.3	± 0.6	± 0.2	± 0.1	± 0.3	± 0.6

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[1] Minor components are summed up to sum of unidentified / diffuse residues.
[2] Difference to Material Balance values due to rounding errors as well as clean up and chromatographic losses.
[3] Values taken from Material Balance.

Aclonifen degraded at a moderate rate in [redacted] Hof Wurmwiese soil. The reported best-fit non-normalised DT_{50} value was 93.9 days (DFOP model with a $\chi 2$ error of 2%). The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/07. The resulting best-fit DT_{50} value for the trigger endpoint is summarised below in Table 7.1.1.1- 27 (and is the same as the reported values).

Table 7.1.1.1- 27: Degradation rate of aclonifen under aerobic conditions at 20 ºC (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Wurmwiese	DFOP	104.5	k1 1.73e-01 k2 5.52e-03 g 1.509e-01	2.213	0.0205 2.54e-08 -	- - 8.76e-02	- - 0.214	95.86	387.2

III. CONCLUSION

Aclonifen was moderately degraded and mineralised (formation of carbon dioxide was up to 12.1% AR at study end) in soil under aerobic conditions in the laboratory in the dark. Besides carbon dioxide, no major degradation products occurred during the study. Non-extractable residues (NER) increased from 0.5% AR at time zero to 38.7% AR by day 120, which is an indication for biotic degradation of aclonifen.

The best-fit non-normalised DT_{50} value was 95.9 days, derived in accordance with FOCUS guidance document on degradation kinetics (2014).

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002) and is considered valid to assess the aerobic degradation of [aniline-UL-^{14}C] aclonifen in soil.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.1.1/05
Report Author:	[redacted]; [redacted]
Report Year:	2019
Report Title:	Aclonifen: Aerobic degradation / metabolism in four soils
Report No:	EnSa-19-0046
Document No:	M-674036-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 307 (2002); Commission Regulation (EU) No 283/2013 in accordance with Regulation (EC) No 1107/2009; US EPA OCSPP Test Guideline No. 835.4100 / 835.4200 (2008); Japanese MAFF Test Guidelines 12 Nousan 8147, No. 2-5-2 (2008)
Deviations from current test guideline:	Current Guideline: OECD 307 (2002) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of [aniline-UL-^{14}C]aclonifen applied at a rate of 4.0 µg/kg, equivalent to 1.5 g/ha, was investigated in four soils for up to 120 days.

Soil	Source	Texture (USDA)	pH *	OC [%]
[redacted] Farm	[redacted], Essex, England, UK	sandy loam	4.6	1.7
[redacted] Field	[redacted] Farm, Essex, England, UK	loam	5.9	2.5
[redacted]	[redacted] Road, England, UK	loamy sand	5.3	1.5
[redacted]	[redacted] Farm, Essex, England, UK	loam	7.0	2.3

* pH values were derived from aqueous 0.01 M $CaCl_2$ suspensions

The soil was incubated in the dark, at a moisture content equivalent to 55 ± 5% of the maximum water holding capacity under aerobic conditions at 20 °C. The radiochemical purity and specific activity of [U-^{14}C-aniline] labelled aclonifen were >99 % and 6.59 MBq/mg, respectively.

The test was performed in static systems consisting of Erlenmeyer flasks each containing 100 g soil (dry weight equivalents) and equipped with traps (permeable for oxygen) for the collection of carbon dioxide and volatile organic compounds.

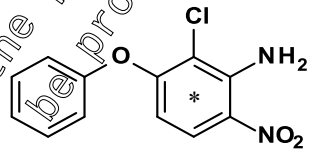
Duplicate samples were taken for extraction and analysis immediately after treatment (day 0) and after 4, 7, 14, 32, 60, 90 and 120 days of incubation. At each sampling interval, the soil was extracted three times at ambient temperature using acetonitrile/water 4/1 (v/v). Furthermore, two microwave assisted extraction steps were performed using acetonitrile/water 4/1 (v/v) at 70 °C and methanol/aqueous 0.1 N HCl 1/1 (v/v) at 50 °C. The amounts of test item and degradation products in soil extracts were determined by liquid scintillation counting (LSC) and by TLC and HPLC analysis with radiodetection. The amounts of volatiles and non-extractable residues were determined by LSC and combustion followed by LSC, respectively.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit non-normalised DT_{50} values ranging from 55.0 to 282.6 days and DT_{90} values from 237 to >1000 days.

I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: [U-^{14}C-aniline]-aclonifen



* Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC) 2-chloro-6-nitro-3-phenoxy-aniline
CA registry number: 74070-46-5
Lot or batch number: KML 10434
Specific activity: 6.59 MBq/mg

Radiochemical purity: 97.7% (HPLC with radioactivity detector)

Stability of test compound: Shown to be stable under the conditions of the test

Application vehicle: (400 µL acetone/water 1/1 v/v)

2. Soil

Four agricultural soils were freshly collected from four sites in the United Kingdom where there had been no application of plant production products in the least 5 years. The soils were collected fresh from the field (upper horizon of 0 to 20 cm) in January 2019 and sieved to 2 mm prior to use. Specific details are shown below.

Table 7.1.1.1- 28: Physico-chemical characteristics of the soil used in aerobic soil study

Characteristic / Code	Units	[redacted] Farm		[redacted] Field		[redacted]		[redacted]	
Origin	Country	UK		UK		UK		UK	
Location	City or Township	[redacted] Essex, UK		[redacted] Farm, [redacted] Essex, UK		[redacted] Road, [redacted] Suffolk, UK		[redacted] Farm, [redacted], Essex, UK	
GPS Coordinates		Lat [redacted]		[redacted]		[redacted]		[redacted]	
Particle Size Analysis									
Sand (50 - 2 mm)	%	65		37		83		33	
Silt (2 - 50 µm)	%	30		40		16		42	
Clay (< 2 µm)	%	5		23		1		25	
Textural Class	USDA	sandy loam		loam		loamy sand		loam	
pH									
soil/0.01 M $CaCl_2$ 1/2		4.6		5.9		5.3		7.0	
soil/water 1/1		4.7		5.9		5.4		7.1	
saturated paste		4.7		5.9		5.5		7.0	
soil/1 N KCl 1/1		4.1		5.4		5.0		6.6	
Organic Carbon	%	1.7		2.5		1.5		2.3	
Organic Matter[1]	%	2.9		4.3		2.6		4.0	
Cation Exchange Capacit	meq/100g	8.6		16.6		8.7		21.0	
Bulk density (disturbed)	g/cm³	1.16		1.04		1.16		1.06	
Water Holding Capacity									
Maximum WHC		45.5 g H_2O ad 100 g DW		54.5 g H2O ad 100 g DW		46.5 g H2O ad 100 g DW		56.8 g H2O ad 100 g DW	
at 1/10 bar (pF 2.0)	%	15.1		24.2		12.4		26.4	
Soil Microbial Biomass	mg microbial C / kg soil	[2]BIO-	[3]BIO+	[2]BIO-	[3]BIO+	[2]BIO-	[3]BIO+	[2]BIO-	[3]BIO+
DAT - 04		226	-	777	-	631	-	374	-
DAT - 63		135	219	484	621	558	691	483	556
DAT - 123		121	195	412	503	245	317	203	281

[1] % organic matter = % organic carbon x 1.724
[2] BIO- samples were left untreated.
[3] BIO+ samples were applied with solvent of application solution (400 µL acetone/water 1/1 v/v).

B. STUDY DESIGN AND METHODS

1. In-life dates:

23 January 2019 - 29 October 2019

2. Experimental design

Parameter		Description
Duration of test		120 Days
Soil condition		Fresh field samples sieved to 2 mm and equilibrated to study conditions for 4 days.
Soil sample weight		100 g dry weight equivalents per replicate.
Concentration in test system	kg test item/ha	Nominal: 1.5
	mg test item/kg soil DW	Nominal: 4.0 (actual:3.5)
Control conditions (if used)		Samples for determination of soil microbial biomass: native soil with and without application solvent.
Number of replications		Duplicate samples for each sampling interval.
Test apparatus		300 mL Erlenmeyer glass flasks containing 100 g dry weight equivalent of soil.
Test material application	Identity of solvent	acetone/water 1/4 (v/v)
	Volume of application solution used per treatment	400 µL per 100 g soil dry weight
	Application method	Dropwise application to the soil surface using an adjustable pipette.
	Evaporation of application solvent	No
Traps for volatiles		The traps were filled with soda lime and polyurethane foam plug. The traps were permeable for oxygen.
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	19.3 °C
	Moisture content	54.3% MWHC
	Moisture maintenance method	Re-weighing and addition of lost water /(no moisture maintenance was necessary).
	Continuous darkness (Yes/No)	Yes

Sampling

Parameter		Description
Sampling intervals		Duplicate samples were processed and analysed 0, 4, 7, 14, 32, 60, 90 and 120 days after treatment (DAT).
Soil sampling procedures		Complete treated samples were removed at each sampling time and extracted as detailed below.
Collection of volatiles		Soda lime for absorption of carbon dioxide and polyurethane foam for adsorption of volatile organic compounds.
Sampling intervals times for	Moisture content	Each sampling interval and after 36, 56, 84 and 113 days of incubation.
	Sterility checks	N/A.
	Other	Soil microbial biomass was determined for untreated soil at DAT-

Parameter		Description
		4, DAT-63 and DAT-125, as well as for soil treated with application solvent at DAT-63 and DAT-125
Sample storage before analysis		The soils were processed immediately after sampling, first HPLC/radiodetection analysis of soil extracts was performed within two days.

Analytical procedures

At each sampling interval, the soil was extracted three times at ambient temperature using acetonitrile/water 4/1 (v/v). Furthermore, two microwave assisted extraction steps were performed using acetonitrile/water 4/1 (v/v) at 70 °C and methanol/aqueous 0.1 N HCl 1/1 (v/v) at 50 °C. The amounts of test item and degradation products in soil extracts were determined by liquid scintillation counting (LSC) and by TLC/HPLC/radiodetection analysis. The amounts of volatiles and non-extractable residues were determined by LSC and combustion/LSC, respectively.

Following extraction, soil samples were air-dried and homogenised, and the remaining unextracted radioactivity quantified by combustion.

With the exception of the zero time samples, trap attachments were removed for analysis at each sampling time. Carbon dioxide absorbed by soda lime was liberated and trapped in a scintillation cocktail selective for binding of carbon dioxide using an air-tight assembly. For this purpose the soda lime of the trap attachments was transferred into an Erlenmeyer flask, which was then attached with a dropping funnel, containing aqueous hydrochloric acid (18%), and connected to a series of two trapping vessels, each filled with ice-cooled scintillation cocktail. The aqueous hydrochloric acid was added dropwise under continuous stirring and the liberated carbon dioxide was purged into the trapping vessels by a stream of nitrogen. The radioactivity contents of these vessels were determined by LSC and summed to determine the total radioactivity liberated from soda lime.

The PU foam plug was extracted with ethyl acetate for approximately 20 minutes in an ultrasonic bath to desorb any volatile organic compounds. The radioactivity content was determined by LSC.

Degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. Additionally, DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/07. A brief summary of the approach for trigger endpoints is provided below.

The non-normalised data was best fitted by the Double First Order in Parallel (DFOP) model in [redacted] Farm, [redacted] and [redacted] soils with $\chi 2$ errors of 1, 0.4 and 1% and the First Order Multiple Compartment (FOMC) model in [redacted] Field soils with a $\chi 2$ error of 3%.

II. RESULTS AND DISCUSSION

The total recoveries and distribution of radioactivity are shown in detail in Table 7.1.1.1- 29 to Table 7.1.1.1- 32. The recoveries, extractable and non-extractable residue and trap contents at the beginning and end of the study are summarised below.

Mass Balance

Total radioactivity	Sum of activity in soil extracts, soil residue on combustion and that trapped as $^{14}CO_2$ and volatile organics in the volatile traps.
Recovery at 0 DAT	96.8 to 101.4% AR
Overall recovery (all samples)	Range 91.8 to 102.1%, mean 98.9% AR

Bound and Extractable Residues

From the 120 DAT timepoint, duplicate soil samples post extraction were subjected to soil organic matter fractionation into humic acids, fulvic acids and humin fractions. The results indicated that the majority of the non-extractable radioactivity was associated with the humin and humin acids fraction. The recoveries and distribution of radioactivity from humic substance fractionation are shown in Table 7.1.1.1- 33.

Extractable residues	Extractable residues declined with time.	
	Total extractable residues at 0 DAT	96.8 to 101.4% AR
	Total extractable residues at end of study (120 DAT)	26.6 to 74.8% AR
Bound residues	Bound residues generally increased throughout the incubation period.	
	Bound residues at end of study (120 DAT)	18.8 to 60.0% AR

Volatilisation

$^{14}CO_2$	Carbon dioxide evolution gradually increased throughout the incubation.	
	$^{14}CO_2$ evolved at end of study (120 DAT)	10.5% AR
Other volatiles	No other volatiles were observed (< 0.1% AR)	

Extractable residues decreased from DAT-0 to DAT-120 from 99.3 to 73.0% AR in the [redacted] Farm soil, from 98.7 to 33.3% AR in the [redacted] Field soil, from 99.2 to 50.0% AR in the [redacted] soil and from 90.8 to 28.3% AR in the [redacted] soil. Non-extractable residues concurrently increased from DAT-0 to DAT-120 from 0.5 to 19.9% AR in [redacted] Farm soil, from 1.0 to 56.9% AR in the [redacted] Field soil, from 0.7 to 38.0% AR in the [redacted] soil and from 0.9 to 57.7% AR in the [redacted] soil. Formation of volatile organic compounds was insignificant as demonstrated by values of < 0.1% AR at all sampling intervals. The maximum amount of carbon dioxide was 6.3, 10.5, 8.1 and 9.3% AR at study end (DAT-120) in the [redacted] Farm, [redacted] and [redacted] soils, respectively, and 10.5% at DAT-90 in the [redacted] Field soil.

Table 7.1.1.1- 29: Distribution of radioactivity in soil [redacted] Farm under aerobic conditions expressed as percentage of applied radioactivity

	Replicate No.	DAT							
		0	4	7	14	32	60	90	120
Volatiles									
Carbon Dioxide	A	n.a.	0.3	0.5	0.9	1.6	n.a	5.4	6.7
	B	n.a.	0.3	0.5	0.8	1.7	2.9	5.4	5.9
	Mean	**n.a.**	**0.3**	**0.5**	**0.9**	**1.7**	**2.9**	**5.4**	**6.3**
Volatile Organic Compounds	A	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	B	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mean	**n.a.**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**
Total Volatiles	A	n.a.	0.4	0.5	0.9	1.6	<0.1	5.5	6.7
	B	n.a.	0.3	0.5	0.8	1.7	2.9	5.4	5.9
	Mean	**n.a.**	**0.3**	**0.5**	**0.9**	**1.7**	**1.4**	**5.4**	**6.3**
Extractable Residues									
Ambient Extract 1	A	97.1	95.3	92.7	87.8	81.8	79.3	70.2	66.6
	B	95.3	94.7	93.5	88.9	85.7	79.7	70.0	70.0
	Mean	**96.2**	**95.0**	**93.1**	**88.3**	**83.7**	**79.5**	**70.1**	**68.3**
Microwave Extract 1	A	2.7	3.2	3.1	3.4	3.1	3.5	3.5	3.1
	B	2.6	3.3	3.1	3.4	3.2	3.5	3.6	3.2
	Mean	**2.6**	**3.3**	**3.1**	**3.4**	**3.2**	**3.6**	**3.5**	**3.2**
Microwave Extract 2	A	0.5	0.7	0.9	1.0	1.0	1.2	1.1	1.4
	B	0.5	0.7	0.8	1.0	1.0	1.2	1.2	1.5
	Mean	**0.5**	**0.7**	**0.8**	**1.0**	**1.0**	**1.2**	**1.2**	**1.5**
Total Extractable Residues	A	100.3	99.3	96.6	92.1	85.9	84.2	74.9	71.2
	B	98.4	98.6	97.3	93.4	90.0	84.3	74.7	74.8
	Mean	**99.3**	**98.9**	**97.0**	**92.8**	**88.0**	**84.3**	**74.8**	**73.0**
Non-Extractable Residues	A	0.5	2.2	3.5	5.8	10.0	13.9	19.0	21.0
	B	0.5	2.2	3.0	5.9	10.3	13.7	18.9	18.8
	Mean	**0.5**	**2.2**	**3.2**	**5.8**	**10.2**	**13.8**	**19.0**	**19.9**
Material Balance	A	100.8	101.8	100.6	98.8	97.6	98.1	99.3	98.9
	B	98.9	101.2	100.8	100.1	102.1	100.9	99.0	99.5
	Mean	**99.9**	**101.5**	**100.7**	**99.5**	**99.8**	**99.5**	**99.2**	**99.2**
Overall Mean 99.9 ± 0.7									

DAT: days after treatment, n.a. : not applicable

Table 7.1.1.1- 30: Distribution of radioactivity in soil [redacted] Field under aerobic conditions expressed as percentage of applied radioactivity

	Replicate No.	DAT							
		0	4	7	14	32	60	90	120
Volatiles									
Carbon Dioxide	A	n.a.	0.4	0.7	1.4	2.9	n.a	15.1	8.0
	B	n.a.	0.4	0.6	1.2	2.5	4.5	5.8	9.6
	Mean	**n.a.**	**0.4**	**0.6**	**1.3**	**2.7**	**4.5**	**10.5**	**8.8**
Volatile Organic Compounds	A	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	B	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mean	**n.a.**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**
Total Volatiles	A	n.a.	0.4	0.7	1.4	2.9	<0.1	15.1	8.0
	B	n.a.	0.4	0.6	1.2	2.5	4.5	5.8	9.6
	Mean	**n.a.**	**0.4**	**0.6**	**1.3**	**2.7**	**2.3**	**10.5**	**8.8**
Extractable Residues									
Ambient Extract 1	A	93.4	88.6	81.9	71.0	56.7	47.3	21.9	29.4
	B	96.9	87.3	82.6	72.4	59.7	44.6	38.6	26.4
	Mean	**95.1**	**87.9**	**82.3**	**71.7**	**58.2**	**46.1**	**30.2**	**27.9**
Microwave Extract 1	A	2.8	4.3	3.7	4.4	3.9	3.3	2.5	3.7
	B	3.0	4.1	3.8	4.1	3.4	3.3	3.3	3.6
	Mean	**2.9**	**4.2**	**3.7**	**4.2**	**3.6**	**3.3**	**2.9**	**3.6**
Microwave Extract 2	A	0.6	1.0	1.1	1.4	1.5	1.4	1.5	1.6
	B	0.6	1.0	1.1	1.5	1.3	1.6	1.5	1.9
	Mean	**0.6**	**1.0**	**0.1**	**1.4**	**1.4**	**1.5**	**1.5**	**1.7**
Total Extractable Residues	A	96.9	93.8	87.7	76.8	62.1	52.3	25.9	34.7
	B	100.5	92.4	87.4	78.0	64.4	49.5	43.3	31.8
	Mean	**98.7**	**93.1**	**87.1**	**77.4**	**63.2**	**50.9**	**34.6**	**33.3**
Non-Extractable Residues	A	1.0	6.7	11.5	20.06	33.2	41.7	54.2	53.8
	B	1.0	6.4	10.8	19.6	31.8	43.7	47.3	60.0
	Mean	**1.0**	**6.5**	**11.2**	**20.1**	**32.5**	**42.7**	**50.8**	**56.9**
Material Balance	A	97.8	100.9	98.9	98.7	98.2	93.9	95.2	96.6
	B	101.4	99.2	98.8	98.9	98.7	97.7	96.5	101.5
	Mean	**99.6**	**100.1**	**98.9**	**98.8**	**98.4**	**95.8**	**95.9**	**99.0**
Overall Mean 98.3 ± 1.5									

DAT: days after treatment; n.a. : not applicable

Table 7.1.1.1- 31: Distribution of radioactivity in soil [redacted] under aerobic conditions expressed as percentage of applied radioactivity

	Replicate No.	DAT							
		0	4	7	14	32	60	90	120
Volatiles									
Carbon Dioxide	A	n.a.	0.5	0.7	1.2	3.1	n.a	7.2	8.6
	B	n.a.	0.5	0.7	1.2	3.3	5.5	7.0	7.7
	Mean	**n.a.**	**0.5**	**0.7**	**1.2**	**3.2**	**5.5**	**7.1**	**8.1**
Volatile Organic Compounds	A	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	B	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mean	**n.a.**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**
Total Volatiles	A	n.a.	0.5	0.7	1.3	3.1	<0.1	7.2	8.6
	B	n.a.	0.5	0.7	1.2	3.3	5.5	7.0	7.7
	Mean	**n.a.**	**0.5**	**0.7**	**1.2**	**3.2**	**2.8**	**7.1**	**8.2**
Extractable Residues									
Ambient Extract 1	A	95.5	89.7	87.4	80.7	65.7	57.7	49.2	38.9
	B	96.4	90.5	88.7	81.3	66.7	58.7	51.1	48.3
	Mean	**96.0**	**90.1**	**88.0**	**81.0**	**66.2**	**58.2**	**50.2**	**43.6**
Microwave Extract 1	A	2.8	4.0	3.7	4.3	4.1	4.4	4.2	3.7
	B	2.7	4.1	3.6	4.2	4.2	4.5	4.2	4.0
	Mean	**2.8**	**4.0**	**3.7**	**4.2**	**4.2**	**4.4**	**4.2**	**3.8**
Microwave Extract 2	A	0.5	1.0	1.2	1.6	1.9	2.0	2.1	2.7
	B	0.5	0.9	1.2	1.6	1.9	2.2	2.3	2.6
	Mean	**0.5**	**0.9**	**1.2**	**1.6**	**1.9**	**2.1**	**2.2**	**2.6**
Total Extractable Residues	A	98.8	94.6	92.3	86.6	71.8	64.1	55.6	45.3
	B	99.6	95.5	93.5	87.1	72.9	65.4	57.6	54.8
	Mean	**99.2**	**95.1**	**92.9**	**86.8**	**72.3**	**64.8**	**56.6**	**50.0**
Non-Extractable Residues	A	0.7	3.4	7.3	12.4	23.5	30.8	36.3	39.5
	B	0.7	4.6	7.0	12.3	23.6	30.3	36.0	36.5
	Mean	**0.7**	**4.0**	**7.2**	**12.4**	**23.6**	**30.6**	**36.1**	**38.0**
Material Balance	A	99.6	98.5	100.3	100.2	98.3	94.9	99.0	93.4
	B	100.3	100.6	101.2	100.6	99.9	101.2	100.6	99.0
	Mean	**99.9**	**99.5**	**100.7**	**100.4**	**99.1**	**98.1**	**99.8**	**96.2**
Overall Mean 99.2 ± 1.4									

DAT: days after treatment; n.a. : not applicable

Table 7.1.1.1- 32: Distribution of radioactivity in soil [redacted] under aerobic conditions expressed as percentage of applied radioactivity

	Replicate No.	DAT							
		0	4	7	14	32	60	90	120
Volatiles									
Carbon Dioxide	A	n.a.	0.4	0.6	1.0	2.5	n.a	6.0	7.6
	B	n.a.	0.4	0.6	1.0	2.1	4.0	5.9	11.0
	Mean	**n.a.**	**0.4**	**0.6**	**1.0**	**2.3**	**4.0**	**6.0**	**9.3**
Volatile Organic Compounds	A	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	B	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mean	**n.a.**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**
Total Volatiles	A	n.a.	0.4	0.6	1.0	2.5	<0.1	6.0	7.6
	B	n.a.	0.4	0.6	1.0	2.1	4.0	5.9	11.0
	Mean	**n.a.**	**0.4**	**0.6**	**1.0**	**2.3**	**2.0**	**6.0**	**9.3**
Extractable Residues									
Ambient Extract 1	A	92.3	85.5	82.2	74.0	55.9	42.8	31.7	24.6
	B	94.1	87.9	82.0	73.3	59.9	44.5	31.3	21.5
	Mean	**93.2**	**86.7**	**82.1**	**73.6**	**57.9**	**43.7**	**31.5**	**23.1**
Microwave Extract 1	A	2.9	4.2	3.9	4.0	4.4	4.0	3.9	3.7
	B	3.2	4.1	3.9	4.0	4.2	4.2	3.4	3.6
	Mean	**3.0**	**4.2**	**3.9**	**4.0**	**4.1**	**4.1**	**3.6**	**3.7**
Microwave Extract 2	A	0.6	0.9	1.2	1.3	1.3	1.6	1.4	1.7
	B	0.6	1.0	1.1	1.4	1.4	1.7	1.7	1.5
	Mean	**0.6**	**0.9**	**1.1**	**1.3**	**1.3**	**1.6**	**1.5**	**1.6**
Total Extractable Residues	A	95.8	90.6	87.3	79.3	61.7	48.4	37.1	30.0
	B	97.9	93.0	87.0	78.7	65.6	50.3	36.4	26.6
	Mean	**96.8**	**91.8**	**87.2**	**79.0**	**63.6**	**49.4**	**36.7**	**28.3**
Non-Extractable Residues	A	0.9	6.8	10.8	19.2	32.5	43.3	54.2	57.7
	B	0.9	7.0	11.6	19.1	29.5	44.6	51.2	57.7
	Mean	**0.9**	**6.9**	**11.2**	**19.2**	**31.0**	**43.9**	**52.7**	**57.7**
Material Balance	A	96.8	97.8	98.7	99.5	96.6	91.8	97.3	95.4
	B	98.8	100.4	99.2	98.8	97.2	98.9	93.5	95.3
	Mean	**97.8**	**99.1**	**99.0**	**99.2**	**96.9**	**95.4**	**95.4**	**95.4**
Overall Mean 97.3 ± 1.7									

DAT: days after treatment, n.a. : not applicable

Table 7.1.1.1- 33: Humic substance fractionation (as % applied radioactivity)

Soil	Replicate	Humin fraction [% AR]	Humic acid fraction [% AR]	Fulvic acid fraction [% AR]	Total [% AR]
[redacted] Farm	A	7.3	9.4	2.6	19.4
	B	6.9	9.0	2.4	18.2
[redacted] Field	A	28.7	18.7	2.9	50.3
	B	31.2	19.8	3.5	54.4
[redacted]	A	13.7	17.9	3.3	35.0
	B	13.1	16.9	3.0	32.9
[redacted]	A	44.0	12.9	2.6	59.5
	B	42.2	12.1	3.1	57.4

Transformation of Parent Material

The characterisation of soil extracts is presented in Table 7.1.1.1- 34 to Table 7.1.1.1- 37. The amount of aclonifen in the soil extracts decreased from DAT-0 to DAT-120 from 99.3 to 73.0% AR in the [redacted] Farm soil, from 98.7 to 33.3% AR in soil [redacted] Field, from 99.2 to 50.0% AR in the [redacted] soil and from 96.8 to 28.3% AR in the [redacted] soil. Apart from carbon dioxide, no degradation products were observed above > LOD.

Table 7.1.1.1- 34: Characterisation of radioactivity in soil [redacted] Farm under aerobic conditions expressed as percentage of applied radioactivity

Compound		DAT (Mean % AR ± SD)							
		0	4	7	14	32	60	90	120
Aclonifen	Mean	99.3	98.9	97.0	92.8	88.0	84.3	74.8	73.0
	SD	± 0.9	± 0.3	± 0.4	± 0.6	± 2.0	± 0.1	± 0.1	± 1.8
Unknown 1	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Unknown 2	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Unknown 3	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Sum of Unid./Diff. Residues[1]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Total Extractable Residues[2]	Mean	99.3	98.9	97.0	92.8	88.0	84.3	74.8	73.0
	SD	± 0.9	± 0.3	± 0.4	± 0.6	± 2.0	± 0.1	± 0.1	± 1.8
Carbon Dioxide[3]	Mean	n.a.	0.3	0.5	0.9	1.7	2.9	5.4	6.3
	SD		± 0.0	± 0.0	± 0.0	± 0.1	± 0.0	± 0.0	± 0.4
Volatile Organic Compounds[3]	Mean	n.a.	< 0.1	0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues[3]	Mean	0.5	2.2	3.2	5.8	10.2	13.8	19.0	19.9
	SD	± 0.0	± 0.0	± 0.2	± 0.0	± 0.1	± 0.1	± 0.1	± 1.1
Total Recovery[2]	Mean	99.9	101.4	100.7	99.4	99.8	99.5	99.2	99.2
	SD	± 1.0	± 0.3	± 0.1	± 0.6	± 2.3	± 1.4	± 0.1	± 0.3

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[1] Minor components are summed up to sum of unidentified / diffuse residues.
[2] Difference to Material Balance values due to rounding errors as well as clean up and chromatographic losses.
[3] Values taken from Material Balance.

Table 7.1.1.1- 35: Characterisation of radioactivity in soil [redacted] Field under aerobic conditions expressed as percentage of applied radioactivity

Compound		DAT (Mean % AR ± SD)							
		0	4	7	14	32	60	90	120
Aclonifen	Mean SD	98.7 ± 1.8	93.1 ± 0.7	87.1 ± 0.3	77.4 ± 0.6	63.2 ± 1.2	50.9 ± 1.4	32.9 ± 10.1	33.3 ± 1.5
Unknown 1	Mean SD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	LOD	n.d.
Unknown 2	Mean SD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	< LOD	n.d.
Unknown 3	Mean SD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	< LOD	n.d.
Sum of Unid./Diff. Residues[1]	Mean SD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	< LOD	n.d.
Total Extractable Residues[2]	Mean SD	98.7 ± 1.8	93.1 ± 0.7	87.1 ± 0.3	77.4 ± 0.6	63.2 ± 1.2	50.9 ± 1.4	32.9 ± 10.1	33.3 ± 1.5
Carbon Dioxide[3]	Mean SD	n.a.	0.4 ± 0.0	0.6 ± 0.0	1.3 ± 0.1	2.5 ± 0.2	4.5 ± 0.0	10.5 ± 4.6	8.8 ± 0.8
Volatile Organic Compounds[3]	Mean SD	n.a.	< 0.1 ± 0.0	< 0.1 ± 0.0	< 0.1 ± 0.0	< 0.1 ± 0.0	< 0.1 ± 0.0	< 0.1 ± 0.0	< 0.1 ± 0.0
Non-Extractable Residues[3]	Mean SD	1.0 ± 0.0	6.5 ± 0.2	11.2 ± 0.4	20.1 ± 0.5	32.5 ± 0.7	42.7 ± 1.0	50.8 ± 3.5	56.9 ± 3.1
Total Recovery[2]	Mean SD	99.6 ± 1.8	100.1 ± 0.9	98.9 ± 0.1	98.8 ± 0.1	98.4 ± 0.3	95.8 ± 1.9	95.1 ± 1.4	99.0 ± 2.5

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[1] Minor components are summed up to sum of unidentified / diffuse residues.
[2] Difference to Material Balance values due to rounding errors as well as clean-up and chromatographic losses.
[3] Values taken from Material Balance.

Table 7.1.1.1- 36: Characterisation of radioactivity in soil [redacted] under aerobic conditions expressed as percentage of applied radioactivity

Compound		DAT (Mean % AR ± SD)							
		0	4	7	14	32	60	90	120
Aclonifen	Mean	99.2	95.1	92.9	86.8	72.3	64.8	56.6	50.0
	SD	± 0.4	± 0.4	± 0.6	± 0.3	± 0.6	± 0.6	± 1.0	± 4.8
Unknown 1	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Unknown 2	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Unknown 3	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Sum of Unid./Diff. Residues[1]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Total Extractable Residues[2]	Mean	99.2	95.1	92.9	86.8	72.3	64.8	56.6	50.0
	SD	± 0.4	± 0.4	± 0.6	± 0.3	± 0.6	± 0.6	± 1.0	± 4.8
Carbon Dioxide[3]	Mean	n.a.	0.5	0.7	1.2	3.2	5.5	7.1	8.1
	SD		± 0.0	± 0.0	± 0.0	± 0.1	± 0.0	± 0.1	± 0.5
Volatile Organic Compounds[3]	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues[3]	Mean	0.7	4.0	7.2	12.4	23.6	30.6	36.1	38.0
	SD	± 0.0	± 0.0	± 0.2	± 0.0	± 0.1	± 0.2	± 0.1	± 1.5
Total Recovery[2]	Mean	99.9	99.5	100.7	100.4	99.1	98.1	99.8	96.2
	SD	± 0.4	± 1.0	± 0.5	± 0.2	± 0.8	± 3.2	± 0.8	± 2.8

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[1] Minor components are summed up to sum of unidentified / diffuse residues.
[2] Difference to Material Balance values due to rounding errors as well as clean-up and chromatographic losses.
[3] Values taken from Material Balance.

Table 7.1.1.1- 37: Characterisation of radioactivity in soil [redacted] under aerobic conditions expressed as percentage of applied radioactivity

Compound		DAT (Mean % AR ± SD)							
		0	4	7	14	32	60	90	120
Aclonifen	Mean	96.8	91.8	87.2	79.0	63.6	49.4	36.7	28.3
	SD	± 1.0	± 1.2	± 0.2	± 0.3	± 2.0	± 0.9	± 0.3	± 1.7
Unknown 1	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Unknown 2	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Unknown 3	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Sum of Unid./Diff. Residues[1]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	SD								
Total Extractable Residues[2]	Mean	96.8	91.8	87.2	79.0	63.6	49.4	36.7	28.3
	SD	± 1.0	± 1.2	± 0.2	± 0.3	± 2.0	± 0.9	± 0.3	± 1.7
Carbon Dioxide[3]	Mean	n.a.	0.4	0.6	1.0	2.3	4.0	6.0	9.3
	SD		± 0.0	± 0.0	± 0.0	± 0.2	± 0.0	± 0.0	± 1.7
Volatile Organic Compounds[3]	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues[3]	Mean	0.9	6.9	11.2	19.2	31.0	43.9	52.7	57.37
	SD	± 0.0	± 0.1	± 0.4	± 0.0	± 1.8	± 0.6	± 1.5	± 0.0
Total Recovery[2]	Mean	97.8	99.1	99.0	99.2	96.9	95.3	95.4	95.3
	SD	± 1.0	± 1.3	± 0.2	± 0.4	± 0.3	± 3.6	± 1.9	± 0.0

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[1] Minor components are summed up to sum of unidentified / diffuse residues. unidentified metabolites and diffuse residues.
[2] Difference to Material Balance values due to rounding errors as well as clean-up and chromatographic losses.
[3] Values taken from Material Balance.

Aclonifen degraded at a moderate rate in [redacted] Field and [redacted] soils and more slowly in [redacted] Farm and [redacted] soils. The reported best-fit non-normalised DT_{50} values ranging from 55 to 561 days. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/07. The resulting best-fit DT_{50} values for the trigger endpoints are summarised below in Table 7.1.1.1- 38.

Table 7.1.1.1- 38: Degradation rate of aclonifen under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, α, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted] Farm	DFOP	100.8	k1 2.222e-03 k2 6.939e-02 g 9.367e-01	1.235	6.92e-05 0.153 -	- - 8.701e-01	- - 1.003	282.6	>1000
[redacted] Field	FOMC	101.4	α 0.9664 β 52.4101	2.934	- -	0.2028 -11.5297	1.73 116.35	54.97	515.4
[redacted]	DFOP	98.8	k1 0.070432 k2 0.09021 g 00.148474	0.4214	0.01091 2.91e-09 -	- - 0.062343	- - 0.235	59.52	237.4

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted]	DFOP	100.3	k1 4.393e-02 k2 3.502e-03 g 2.369e-01	1.104	0.03705 0.00744 -	- - 4.123e-02	- - 0.432	121.4	580.2

III. CONCLUSION

Aclonifen was moderately degraded and mineralised (formation of carbon dioxide was up to 10.5% AR at study end) in soil under aerobic conditions in the laboratory in the dark. The best-fit non-normalised DT_{50} values were between 55.0 to 282.6 days and DT_{90} values from 237 to >1000 days in the tested soils.

Besides carbon dioxide and non-extractable residues (NER), no metabolites were detected during the study. Non-extractable residues (NER) increased to a maximum of 60.0% AR at the end of the study, which is an indication for biotic degradation of aclonifen.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002) and is considered valid to assess the aerobic degradation of [aniline-UL-^{14}C] aclonifen in soil.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.1.1/06
Report Author:	[redacted]; [redacted]
Report Year:	2019
Report Title:	Aclonifen: Aerobic degradation / metabolism in four soils
Report No:	EnSa-19-0251
Document No:	M-674477-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 307, Commission Regulation (EU) N0 283/2013 in accordance with Regulation (EC) No 1107/2009; US EPA OCSSP Test Guideline No. 835.4100 / 835.4200; Japanese MAFF Test Guidelines 12 Nousan 8147, No. 2-5-2
Deviations from current test guideline:	Current Guideline: OECD 307 (2002) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of [phenoxy-UL-^{14}C]aclonifen applied at a rate of 4.8 mg/kg, equivalent to 1.8 kg/ha, was investigated in four soils for up to 120 days.

Soil	Source	Texture (USDA)	pH *	OC [%]
[redacted] Hof AXXa	[redacted], North Rhine-Westphalia, Germany	sandy loam	6.7	2.0
[redacted] am [redacted]	[redacted], North Rhine-Westphalia, Germany	silt loam	6.1	1.9
[redacted] Hof	[redacted], North Rhine-Westphalia, Germany	silt loam	5.5	2.7
[redacted] II	[redacted], North Rhine-Westphalia, Germany	clay loam	7.2	5.6

* pH values were derived from aqueous 0.01 M $CaCl_2$ suspensions

The soil was incubated in the dark, at a moisture content equivalent to 35 ± 5% of the maximum water holding capacity under aerobic conditions at 20 °C. The radiochemical purity and specific activity of [phenoxy-U-^{14}C] labelled aclonifen were >98 % and 4.52 MBq/mg, respectively.

The test was performed in static systems consisting of Erlenmeyer flasks each containing 100 g soil (dry weight equivalents) and equipped with traps (permeable for oxygen) for the collection of carbon dioxide and volatile organic compounds.

Duplicate samples were taken for extraction and analysis immediately after treatment (day 0) and after 2, 7, 14, 27, 57, 90 and 120 days of incubation. At each sampling interval, the soil was extracted three times at ambient temperature using acetonitrile/water 4/1 (v/v). Furthermore, two microwave assisted extraction steps were performed using acetonitrile/water 4/1 (v/v) at 70 °C and methanol/aqueous 0.1 N HCl 1/1 (v/v) at 50 °C. The amounts of test item and degradation products in soil extracts were determined by liquid scintillation counting (LSC) and by TLC and HPLC analysis with radiodetection. The amounts of volatiles and non-extractable residues were determined by LSC and combustion followed by LSC, respectively.

Extractable residues decreased from DAT-0 to DAT-120 from 97.0 to 15.4% AR in the [redacted] Hof AXXa soil, from 100.8 to 30.0% AR in the [redacted] am [redacted] soil, from 102.0 to 44.1% AR in the [redacted] Hof soil and from 99.3 to 13.0% AR in the [redacted] II soil. Non-extractable residues concurrently increased from DAT-0 to DAT-120 from 0.9 to 56.3% AR in [redacted] Hof AXXa soil, from 1.3 to 53.1% AR in the [redacted] am [redacted] soil, from 1.8 to 42.1% AR in the [redacted] Hof soil and from 2.0 to 70.9% AR in the [redacted] II soil.

Formation of volatile organic compounds was insignificant as demonstrated by values of ≤ 0.1% AR at all sampling intervals. The maximum amount of carbon dioxide was 29.4, 14.4, 13.8 and 15.1% AR at study end (DAT-120) in the [redacted] Hof AXXa, [redacted] am [redacted], [redacted] Hof and [redacted] II soils, respectively.

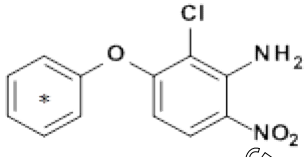
The amount of aclonifen in the soil extracts decreased from DAT-0 to DAT-120 from 97.0 to 14.2% AR in the [redacted] Hof AXXa, soil, from 100.8 to 28.7% AR in soil [redacted] am [redacted], from 102.0 to 43.5% AR in the [redacted] Hof soil and from 99.3 12.2% AR in the [redacted] II soil.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit non-normalised DT_{50} values ranging from 26.4 to 85.8 days and DT_{90} values from 147 to 331 days.

I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: [phenoxy-UL-^{14}C]-aclonifen



* Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC)	2-chloro-6-nitro-3-phenoxy-aniline
CA registry number:	74070-46-5
Lot or batch number:	KML 1039
Specific activity:	4.52 MBq/mg
Radiochemical purity:	> 98% (HPLC with radioactivity detector)
Stability of test compound:	Shown to be stable under the conditions of the test
Application vehicle:	Acetone/water 1:1 (v/v), made alkaline with triethylamine

2. Soil Four agricultural soils were freshly collected from four sites in the Germany where there had been no application of plant production products in the last 5 years. The soils were collected fresh from the field (upper horizon of 0 to 20 cm) in April 2018 and sieved to 2 mm prior to use. The main characteristics of the soils are shown below.

Table 7.1.1.1- 39: Physico-chemical characteristics of the soil used in aerobic soil study

Characteristic / Code	Units	[redacted] Hof AXXa		[redacted] am [redacted]		[redacted] Hof		[redacted] II	
Origin	Country	Germany		Germany		Germany		Germany	
Location	City or Township	[redacted], North Rhine-Westphalia, Germany		[redacted], North Rhine-Westphalia, Germany		[redacted], North Rhine-Westphalia, Germany		[redacted], North Rhine-Westphalia, Germany	
GPS Coordinates		[redacted]		[redacted]		[redacted]		[redacted]	
Soil Taxonomic Classification (USDA)		Sandy, mixed, active, nonacid, mesic Inceptic Hapludalf		Coarse-silty, mixed, active, nonacid, mesic Typic Dystrudept		Loamy-skeletal, mixed, semiactive, mesic Dystric Eutrudept		Fine-loamy, mixed, active, frigid Typic Eutrudept	
Particle Size Analysis									
Sand (50 - 2 mm)	%	70		18		28		30	
Silt (2 - 50 µm)	%	20		68		54		42	
Clay (< 2 µm)	%	10		14		18		28	
Textural Class	USDA	sandy loam		silt loam		silt loam		clay loam	
pH									
soil/0.01 M $CaCl_2$ 1/2		6.7		6.1		5.5		7.2	
soil/water 1/1		6.9		6.3		5.7		7.4	
saturated paste		7.0		6.4		5.8		7.3	
soil/1 N KCl 1/1		6.6		5.8		5.2		7.0	
Organic Carbon	%	2.0		1.9		2.7		5.6	
Organic Matter[1]	%	3.4		3.3		4.7		9.7	
Cation Exchange Capacity	meq/100g	9.3		11.3		10.4		19.6	
Bulk density (disturbed)	g/cm³	1.2		1.1		1.0		1.0	
Water Holding Capacity									
Maximum WHC		58.4 g H_2O ad 100 g DW		55.3 g H_2O ad 100 g DW		64.3 g H2O ad 100 g DW		90.5 g H2O ad 100 g DW	
at 1/10 bar (pF 2.0)	%	14.6		27.0		27.4		37.9	
Soil Microbial Biomass		[2]BIO-	[3]BIO+	[2]BIO-	[3]BIO+	[2]BIO-	[3]BIO+	[2]BIO-	[3]BIO+
DAT - 04	mg microbial C / kg soil	921	-	944	-	846	-	2732	-
DAT - 61		506	485	528	527	579	558	1879	1936
DAT - 124		329	313	410	393	445	408	1618	1644

[1] % organic matter = % organic carbon x 1.724
[2] BIO- samples were left untreated.
[3] BIO+ samples were applied with solvent of application solution (200 µL acetone).

B. STUDY DESIGN AND METHODS

1. In-life dates:

12 April 2017 – 11 October 2019

2. Experimental design

Parameter	Description
Duration of test	120 Days

Parameter		Description
Soil condition		Fresh field samples sieved to 2 mm and equilibrated to study conditions for 2 days.
Soil sample weight		100 g dry weight equivalents per replicate.
Concentration in test system	kg test item/ha	Nominal: 1.8
	mg test item/kg soil DW	Nominal: 4.8 (actual 5.1)
Control conditions (if used)		Samples for determination of soil microbial biomass: native soil with and without application solvent.
Number of replications		Duplicate samples for each sampling interval.
Test apparatus		300 mL Erlenmeyer glass flasks containing 100 g dry weight equivalent of soil.
Test material application	Identity of solvent	Acetone/water 1/1 (*v*/*v*), made alkaline with triethylamine
	Volume of application solution used per treatment	400 µL per 100 g soil dry weight
	Application method	Dropwise application to the soil surface using an adjustable pipette.
	Evaporation of application solvent	No
Traps for volatiles		The traps were filled with soda lime and polyurethane foam plug. The traps were permeable for oxygen.
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	19.4 °C
	Moisture content	54.0% MWHC
	Moisture maintenance method	Re-weighing and addition of lost water
	Continuous darkness (Yes/No)	Yes

Sampling

Parameter		Description
Sampling intervals		Duplicate samples were processed and analysed 0, 2, 7, 14, 27, 57, 90 and 120 days after treatment (DAT).
Soil sampling procedures		Complete treated samples were removed at each sampling time and extracted as detailed below.
Collection of volatiles		Soda lime for absorption of carbon dioxide and polyurethane foam for adsorption of volatile organic compounds.
Sampling intervals / times for	Moisture content	Each sampling interval
	Sterility checks	N/A
	Other	Soil microbial biomass was determined for untreated soil at DAT-0, DAT-61 and DAT-124, as well as for soil treated with application solvent at DAT-61 and DAT-124
Sample storage before analysis		The soils were processed immediately after sampling; first HPLC/radiodetection analysis of soil extracts was performed within three days.

Analytical procedures

At each sampling interval, the soil was extracted three times at ambient temperature using acetonitrile/water 4/1 (v/v). Furthermore, two microwave assisted extraction steps were performed

using acetonitrile/water 4/1 (v/v) at 70 °C and methanol/aqueous 0.1 N HCl 1/1 (v/v) at 50 °C. The amounts of test item and degradation products in soil extracts were determined by liquid scintillation counting (LSC) and by TLC/HPLC/radiodetection analysis. The amounts of volatiles and non-extractable residues were determined by LSC and combustion/LSC, respectively.

Following extraction, soil samples were lyophilised and homogenised, and the remaining unextracted radioactivity quantified by combustion.

With the exception of the zero time samples, trap attachments were removed for analysis at each sampling time. Carbon dioxide absorbed by soda lime was liberated and trapped in a scintillation cocktail selective for binding of carbon dioxide using an air-tight assembly. For this purpose the soda lime of the trap attachments was transferred into an Erlenmeyer flask, which was then attached with a dropping funnel, containing aqueous hydrochloric acid (18%), and connected to a series of two trapping vessels, each filled with ice-cooled scintillation cocktail. The aqueous hydrochloric acid was added dropwise under continuous stirring and the liberated carbon dioxide was purged into the trapping vessels by a stream of nitrogen. The radioactivity contents of these vessels were determined by LSC and summed to determine the total radioactivity liberated from soda lime.

The PU foam plug was extracted with ethyl acetate to desorb any volatile organic compounds. The radioactivity content was determined by LSC.

Degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. Additionally, DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details are provided in Document KCA 7.1.2.1.1/07. A brief summary of the approach for trigger endpoints is provided below.

The non-normalised data was best fitted by the Simple First Order (SFO) model in [redacted] Hof AXXa soil and Double First Order in Parallel (DFOP) model in [redacted] am [redacted], [redacted] Hof and [redacted] II soils with χ2 errors of 2%.

II. RESULTS AND DISCUSSION

The total recoveries and distribution of radioactivity are shown in detail in Table 7.1.1.1- 40 to Table 7.1.1.1- 43. The recoveries, extractable and non-extractable residue and trap contents at the beginning and end of the study are summarised below.

Mass Balance

Total radioactivity	Sum of activity in soil extracts, soil residue on combustion and that trapped as $^{14}CO_2$ and volatile organics in the volatile traps
Recovery at 0 DAT	97.6 to 104.6% AR
Overall recovery (all samples)	Range 92.0 to 104.6% mean 99.1% AR

Bound and Extractable Residues

From the 120 DAT timepoint soil samples post extraction were subjected to soil organic matter fractionation into humic acids, fulvic acids and humin fractions. The results are shown in Table 7.1.1.1- 44.

Extractable residues	Extractable residues declined with time.	
	Total extractable residues at 0 DAT	96.7 to 102.7% AR
	Total extractable residues at end of study (120 DAT)	9.4 to 44.1% AR
Bound residues	Bound residues generally increased throughout the incubation period.	

	Bound residues at end of study (120 DAT)	41.8 to 72.6% AR

Volatilisation

$^{14}CO_2$	Carbon dioxide evolution gradually increased throughout the incubation.	
	$^{14}CO_2$ evolved at end of study (120 DAT)	29.4% AR
Other volatiles	No other volatiles were observed (≤ 0.1% AR)	

Extractable residues decreased from DAT-0 to DAT-120 from 97.0 to 15.4% AR in the [redacted] Hof AXXa soil, from 100.8 to 30.0% AR in the [redacted] am [redacted] soil, from 102.0 to 44.1% AR in the [redacted] Hof soil and from 99.3 to 13.0% AR in the [redacted] II soil. Non-extractable residues concurrently increased from DAT-0 to DAT-120 from 0.9 to 56.3% AR in [redacted] Hof AXXa soil, from 1.3 to 53.1% AR in the [redacted] am [redacted] soil, from 1.8 to 42.1% AR in the [redacted] Hof soil and from 2.0 to 70.9% AR in the [redacted] II soil. Formation of volatile organic compounds was insignificant as demonstrated by values of ≤ 0.1% AR at all sampling intervals. The maximum amount of carbon dioxide was 29.4, 14.4, 13.8 and 15.1% AR at study end (DAT-120) in the [redacted] Hof AXXa, [redacted] am [redacted], [redacted] Hof and [redacted] II soils, respectively.

Table 7.1.1.1- 40: Distribution of radioactivity in soil [redacted] Hof AXXa under aerobic conditions expressed as percentage of applied radioactivity

	Replicate No.	DAT							
		0	2	7	14	27	57	90	120
Volatiles									
Carbon Dioxide	A	n.a.	0.1	0.6	1.5	2.5	6.2	28.8	37.6
	B	n.a.	0.2	0.5	1.3	2.2	4.6	8.7	21.1
	Mean	**n.a.**	**0.1**	**0.5**	**1.4**	**2.3**	**5.4**	**18.7**	**29.4**
Volatile Organic Compounds	A	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	B	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mean	**n.a.**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**
Total Volatiles	A	n.a.	0.1	0.6	1.5	2.5	6.2	28.9	37.6
	B	n.a.	0.2	0.5	1.5	2.2	4.7	8.8	21.1
	Mean	**n.a.**	**0.1**	**0.5**	**1.5**	**2.4**	**1.4**	**18.8**	**29.4**
Extractable Residues									
Ambient Extract 1	A	93.0	91.4	78.5	73.4	60.2	36.7	10.5	7.0
	B	93.8	95.2	88.0	74.6	63.1	44.7	36.4	21.1
	Mean	**93.4**	**93.3**	**83.3**	**74.0**	**61.7**	**40.7**	**23.4**	**12.5**
Microwave Extract 1	A	2.8	3.0	4.8	3.4	3.1	3.2	1.7	1.5
	B	2.7	2.8	3.5	3.7	2.8	3.8	2.4	2.3
	Mean	**2.8**	**2.9**	**4.1**	**3.6**	**2.9**	**3.5**	**3.5**	**1.9**
Microwave Extract 2	A	0.9	1.1	1.6	1.3	1.3	1.5	1.3	0.9
	B	0.9	1.2	1.6	1.3	1.3	1.6	1.8	1.2
	Mean	**0.9**	**1.2**	**1.6**	**1.3**	**1.3**	**1.6**	**1.5**	**1.0**
Total Extractable Residues	A	96.7	95.5	84.9	78.1	64.6	41.3	13.4	9.4
	B	97.4	98.2	93.1	79.6	67.3	50.1	40.6	21.4
	Mean	**97.0**	**97.4**	**89.0**	**78.9**	**65.9**	**45.7**	**27.0**	**15.4**
Non-Extractable Residues	A	0.9	4.2	10.3	19.3	29.5	48.0	53.7	53.9
	B	0.8	4.2	9.8	17.1	27.5	41.8	52.2	58.7
	Mean	**0.9**	**4.2**	**10.0**	**18.2**	**28.5**	**44.9**	**52.9**	**56.3**
Material Balance	A	97.6	99.8	95.8	98.9	96.7	95.5	96.0	101.0
	B	98.2	103.5	103.4	98.2	96.9	96.5	101.5	101.2
	Mean	**97.9**	**101.7**	**99.6**	**98.6**	**96.8**	**96.0**	**98.7**	**101.1**
Overall Mean 98.8 ± 1.9									

DAT: days after treatment; n.a. : not applicable

Table 7.1.1.1- 41: Distribution of radioactivity in soil [redacted] am [redacted] under aerobic conditions expressed as percentage of applied radioactivity

	Replicate No.	DAT							
		0	2	7	14	27	57	90	120
Volatiles									
Carbon Dioxide	A	n.a.	0.2	1.0	2.2	4.7	7.4	11.7	14.3
	B	n.a.	0.3	0.9	2.3	4.4	9.0	14.4	
	Mean	**n.a.**	**0.3**	**0.9**	**2.3**	**4.6**	**5.4**	**10.4**	**14.4**
Volatile Organic Compounds	A	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	B	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mean	**n.a.**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**
Total Volatiles	A	n.a.	0.2	1.0	2.2	4.7	7.4	11.7	14.3
	B	n.a.	0.3	0.9	2.3	4.5	9.6	9.2	14.6
	Mean	**n.a.**	**0.3**	**0.9**	**2.3**	**4.6**	**4.0**	**10.5**	**14.4**
Extractable Residues									
Ambient Extract 1	A	97.7	90.2	79.5	68.5	59.8	40.9	33.3	25.3
	B	95.3	87.9	81.2	67.4	62.6	38.7	29.3	26.0
	Mean	**96.5**	**89.1**	**80.3**	**67.9**	**61.2**	**39.8**	**31.3**	**25.7**
Microwave Extract 1	A	3.3	3.2	4.2	3.6	2.9	3.8	2.9	2.7
	B	3.0	4.4	4.0	3.6	3.0	3.6	2.4	2.7
	Mean	**2.9**	**3.8**	**4.1**	**3.6**	**2.9**	**3.7**	**2.7**	**2.7**
Microwave Extract 2	A	0.9	1.1	1.7	1.4	1.6	1.4	1.9	1.6
	B	1.0	1.3	1.4	1.3	1.5	1.5	2.3	1.7
	Mean	**0.9**	**1.2**	**1.5**	**1.4**	**1.5**	**1.4**	**2.1**	**1.7**
Total Extractable Residues	A	102.0	94.5	85.3	73.5	64.4	46.1	38.1	29.6
	B	99.7	93.6	86.5	72.3	67.2	43.8	34.0	30.4
	Mean	**100.8**	**94.0**	**85.9**	**72.9**	**65.7**	**45.0**	**36.0**	**30.0**
Non-Extractable Residues	A	1.5	4.6	11.9	20.9	31.3	43.5	47.5	52.7
	B	1.0	5.9	12.9	21.7	31.5	45.6	52.5	53.5
	Mean	**1.3**	**5.2**	**12.4**	**21.3**	**31.4**	**44.6**	**50.0**	**53.1**
Material Balance	A	103.5	99.1	98.2	96.7	100.2	97.1	97.3	96.6
	B	100.7	99.8	100.3	96.3	103.2	90.0	95.7	98.3
	Mean	**102.1**	**99.5**	**99.2**	**96.5**	**101.7**	**93.6**	**96.5**	**97.6**
Overall Mean 101.7 ± 1.9									

DAT: days after treatment, n.a. : not applicable

Table 7.1.1.1- 42: Distribution of radioactivity in soil [redacted] Hof under aerobic conditions expressed as percentage of applied radioactivity

	Replicate No.	DAT							
		0	2	7	14	27	57	90	120
Volatiles									
Carbon Dioxide	A	n.a.	0.2	0.8	1.7	3.2	8.0	11.6	14.1
	B	n.a.	0.2	0.7	1.5	3.3	6.9	10.7	13.4
	Mean	**n.a.**	**0.2**	**0.7**	**1.6**	**3.3**	**7.4**	**11.2**	**13.8**
Volatile Organic Compounds	A	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	B	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mean	**n.a.**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**
Total Volatiles	A	n.a.	0.2	0.8	1.8	3.3	8.0	11.7	14.2
	B	n.a.	0.2	0.7	1.6	3.4	6.9	10.8	13.5
	Mean	**n.a.**	**0.2**	**0.8**	**1.7**	**3.3**	**7.5**	**11.2**	**13.8**
Extractable Residues									
Ambient Extract 1	A	97.4	86.1	78.6	75.6	67.7	50.5	44.9	38.7
	B	96.4	87.8	83.8	79.9	68.7	54.9	45.9	38.7
	Mean	**96.9**	**87.0**	**81.2**	**77.7**	**68.2**	**52.7**	**45.4**	**38.7**
Microwave Extract 1	A	4.2	4.1	5.2	4.7	5.8	4.5	3.6	3.2
	B	3.8	3.4	5.0	4.6	3.8	5.2	3.3	3.3
	Mean	**4.0**	**3.7**	**5.1**	**4.6**	**4.8**	**4.9**	**3.5**	**3.3**
Microwave Extract 2	A	1.1	1.4	2.1	1.7	2.6	2.3	1.8	2.2
	B	1.1	1.2	1.9	1.5	2.0	1.8	2.3	2.2
	Mean	**1.1**	**1.3**	**2.0**	**1.6**	**2.3**	**2.0**	**2.1**	**2.2**
Total Extractable Residues	A	102.7	91.6	86.0	82.0	76.1	57.2	50.3	44.1
	B	101.3	92.4	90.7	86.0	74.6	61.9	51.5	44.1
	Mean	**102.0**	**92.0**	**88.4**	**84.0**	**75.3**	**59.6**	**50.9**	**44.1**
Non-Extractable Residues	A	1.8	4.2	9.8	14.9	21.7	33.5	41.2	42.4
	B	1.7	4.5	10.7	13.8	22.2	32.2	39.8	41.8
	Mean	**1.8**	**4.4**	**10.2**	**14.3**	**22.0**	**32.9**	**40.5**	**42.1**
Material Balance	A	104.6	96.1	96.6	98.7	101.1	98.8	103.2	100.7
	B	103.0	97.1	102.1	101.3	100.2	101.1	102.1	99.4
	Mean	**103.8**	**96.6**	**99.3**	**100.0**	**100.6**	**99.9**	**102.7**	**100.1**
Overall Mean 100.4 ± 2.0									

DAT: days after treatment, n.a. : not applicable

Table 7.1.1.1- 43: Distribution of radioactivity in soil [redacted] II under aerobic conditions expressed as percentage of applied radioactivity

	Replicate No.	DAT							
		0	2	7	14	27	57	90	120
Volatiles									
Carbon Dioxide	A	n.a.	0.2	1.1	2.4	4.9	2.0	7.5	14.8
	B	n.a.	0.3	1.0	2.3	4.7	6.5	9.3	15.4
	Mean	**n.a.**	**0.3**	**1.0**	**2.4**	**4.8**	**4.2**	**8.4**	**15.1**
Volatile Organic Compounds	A	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	B	n.a.	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mean	**n.a.**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**
Total Volatiles	A	n.a.	0.2	1.1	2.4	4.9	2.1	7.6	14.9
	B	n.a.	0.3	1.0	2.4	4.8	6.6	9.3	15.4
	Mean	**n.a.**	**0.3**	**1.1**	**2.4**	**4.8**	**4.3**	**8.5**	**15.1**
Extractable Residues									
Ambient Extract 1	A	94.2	84.7	74.3	57.8	44.7	28.8	22.8	11.9
	B	93.1	86.5	72.2	59.6	42.9	32.7	16.4	9.8
	Mean	**93.7**	**85.6**	**73.3**	**58.7**	**43.8**	**28.2**	**19.6**	**10.8**
Microwave Extract 1	A	4.4	4.1	5.3	4.1	3.2	3.9	1.6	1.1
	B	4.3	4.2	4.4	4.1	3.2	4.5	1.7	1.4
	Mean	**4.3**	**4.2**	**4.8**	**4.1**	**3.2**	**3.9**	**1.7**	**1.3**
Microwave Extract 2	A	1.4	1.2	1.7	1.1	1.3	1.3	1.0	0.9
	B	1.2	1.4	1.8	0.9	1.6	1.7	1.1	0.9
	Mean	**1.3**	**1.3**	**1.8**	**1.0**	**1.5**	**1.5**	**1.1**	**0.9**
Total Extractable Residues	A	100.0	90.0	81.3	62.9	49.5	28.4	25.5	14.0
	B	98.5	92.2	78.4	64.9	47.7	38.8	19.2	12.1
	Mean	**99.3**	**91.1**	**79.9**	**63.7**	**48.5**	**33.6**	**22.4**	**13.0**
Non-Extractable Residues	A	2.0	7.6	19.4	31.0	44.4	61.5	67.8	72.6
	B	2.0	8.0	19.7	31.5	46.9	54.2	70.8	69.3
	Mean	**2.0**	**7.8**	**19.5**	**31.3**	**45.7**	**57.9**	**69.3**	**70.9**
Material Balance	A	102.0	97.8	101.8	96.4	98.6	92.0	101.0	101.4
	B	100.5	100.5	99.1	98.4	99.4	99.6	99.3	96.7
	Mean	**101.3**	**99.1**	**100.4**	**97.4**	**99.0**	**95.8**	**100.1**	**99.1**
Overall Mean 99.0 ± 1.6									

DAT: days after treatment, n.a. : not applicable

Table 7.1.1.1- 44: Humic substance fractionation (as % applied radioactivity)

Soil	Humin fraction [% AR]	Humic acid fraction [% AR]	Fulvic acid fraction [% AR]	Total [% AR]
[redacted] Hof AXXa	13.0	27.0	9.8	49.8
[redacted] am [redacted]	12.5	20.4	28.4	61.3
[redacted] Hof	16.5	19.0	3.8	39.3
[redacted] II	31.9	8.9	25.6	66.5

Transformation of Parent Material

The characterisation of soil extracts is presented in Table 7.1.1.1- 45 to Table 7.1.1.1- 48. The amount of aclonifen in the soil extracts decreased from DAT-0 to DAT-120 from 97.0 to 14.2% AR in the [redacted] Hof AXXa, soil, from 100.8 to 28.7% AR in soil [redacted] am [redacted], from 102.0 to 43.5% AR in the [redacted] Hof soil and from 99.3 12.2% AR in the [redacted] II soil. Apart from carbon dioxide, no degradation products were observed above > LOD.

Table 7.1.1.1- 45: Characterisation of radioactivity in soil [redacted] Hof AXXa under aerobic conditions expressed as percentage of applied radioactivity

Compound		DAT (Mean % AR ± SD)							
		0	2	7	14	27	57	90	120
Aclonifen	Mean	97.0	97.4	89.0	78.9	65.9	45.7	27.0	14.2
	SD	± 0.3	± 1.8	± 4.1	± 0.8	± 1.3	± 4.4	± 13.6	± 6.1
Sum of Unid./Diff. Residues[1]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.2
	SD								± 0.1
Total Extractable Residues[2]	Mean	97.0	97.4	89.0	78.9	65.9	45.7	27.0	15.4
	SD	± 0.3	± 1.8	± 4.1	± 0.8	± 1.3	± 4.4	± 13.6	± 6.0
Carbon Dioxide[3]	Mean	n.a.	0.1	0.5	1.4	2.3	5.4	18.7	29.4
	SD		± 0.1	± 0.0	± 0.1	± 0.2	± 0.8	± 10.0	± 8.3
Volatile Organic Compounds[3]	Mean	n.a.	< 0.1	0.1	0.1	< 0.1	0.1	0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.1	± 0.1	± 0.0	± 0.0	± 0.0
Non-Extractable Residues[3]	Mean	0.9	4.2	10.0	18.2	28.5	44.9	52.9	56.3
	SD	± 0.0	± 0.0	± 0.3	± 1.1	± 1.0	± 3.1	± 0.8	± 2.4
Total Recovery[2]	Mean	97.9	101.6	99.6	98.6	96.8	96.0	98.7	101.1
	SD	± 0.3	± 1.9	± 3.8	± 0.3	± 0.1	± 0.55	± 2.7	± 0.1

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[1] Minor components are summed up to sum of unidentified / diffuse residues.
[2] Difference to Material Balance values due to rounding errors as well as clean up and chromatographic losses.
[3] Values taken from Material Balance.

Table 7.1.1.1- 46: Characterisation of radioactivity in soil [redacted] am [redacted] under aerobic conditions expressed as percentage of applied radioactivity

Compound		DAT (Mean % AR ± SD)							
		0	2	7	14	27	57	90	120
Aclonifen	Mean	100.8	94.0	85..9	72.9	65.3	45.0	36.0	28.7
	SD	± 1.1	± 0.4	± 0.6	± 0.6	± 1.9	± 1.2	± 2.1	± 0.5
Sum of Unid./Diff. Residues[1]	Mean	n.d.	n.d.	n.d.	n.d.	< LOD	n.d.	n.d.	1.3
	SD								± 0.0
Total Extractable Residues[2]	Mean	100.8	94.0	85.9	72.9	65.3	45.0	36.0	30.0
	SD	± 1.1	± 0.4	± 0.6	± 0.6	± 1.9	± 1.2	± 2.1	± 0.4
Carbon Dioxide[3]	Mean	n.a.	0.3	0.9	2.3	4.6	7.0	10.4	14.4
	SD		± 0.0	± 0.0	± 0.0	± 0.1	± 3.4	± 1.3	± 0.1
Volatile Organic Compounds[3]	Mean	n.a.	< 0.1	< 0.1	0.1	< 0.1	0.1	0.1	0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.1	± 0.1
Non-Extractable Residues[3]	Mean	1.3	5.2	12.4	21.3	31.4	44.6	50.0	53.1
	SD	± 0.3	± 0.7	± 0.5	± 0.4	± 0.1	± 2.1	± 2.5	± 0.4
Total Recovery[2]	Mean	102.1	99.5	99.2	96.5	101.2	93.5	96.5	97.6
	SD	± 1.4	± 0.3	± 1.0	± 0.2	± 2.0	± 3.5	± 0.8	± 0.9

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[1] Minor components are summed up to sum of unidentified / diffuse residues.
[2] Difference to Material Balance values due to rounding errors as well as clean up and chromatographic losses.
[3] Values taken from Material Balance.

Table 7.1.1.1- 47: Characterisation of radioactivity in soil [redacted] Hof under aerobic conditions expressed as percentage of applied radioactivity

Compound		DAT (Mean % AR ± SD)							
		0	2	7	14	27	57	90	120
Aclonifen	Mean	102.0	92.0	88.4	84.0	75.3	59.6	50.9	43.5
	SD	± 0.7	± 0.4	± 2.4	± 2.0	± 0.8	± 2.3	± 0.6	± 0.1
Sum of Unid./Diff. Residues[1]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	< LOD
	SD								
Total Extractable Residues[2]	Mean	102.0	92.0	88.4	84.0	75.3	59.6	50.9	43.5
	SD	± 0.7	± 0.4	± 2.4	± 2.0	± 0.8	± 2.3	± 0.6	± 0.1
Carbon Dioxide[3]	Mean	n.a.	0.2	0.7	1.6	3.3	7.4	11.2	13.8
	SD		± 0.0	± 0.0	± 0.1	± 0.1	± 0.5	± 0.5	± 0.4
Volatile Organic Compounds[3]	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues[3]	Mean	1.8	4.4	10.2	14.3	22.0	32.9	40.5	42.1
	SD	± 0.1	± 0.2	± 0.4	± 0.6	± 0.3	± 0.6	± 0.7	± 0.3
Total Recovery[2]	Mean	103.8	96.6	99.3	100.0	100.6	99.9	102.7	99.4
	SD	± 0.8	± 0.5	± 2.8	± 1.3	± 0.5	± 1.1	± 0.5	± 0.5

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[1] Minor components are summed up to sum of unidentified / diffuse residues.
[2] Difference to Material Balance values due to rounding errors as well as clean up and chromatographic losses.
[3] Values taken from Material Balance.

Table 7.1.1.1- 48: Characterisation of radioactivity in soil [redacted] II under aerobic conditions expressed as percentage of applied radioactivity

Compound		DAT (Mean % AR ± SD)							
		0	2	7	14	27	57	90	120
Aclonifen	Mean	99.3	91.1	79.9	63.7	48.5	33.6	22.4	12.2
	SD	± 0.8	± 1.1	± 1.5	± 0.8	± 0.8	± 5.2	± 3.2	± 0.9
Sum of Unid./Diff. Residues[1]	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	< LOD
	SD								
Total Extractable Residues[2]	Mean	99.3	91.1	79.9	63.7	48.5	33.6	22.2	12.2
	SD	± 0.8	± 1.1	± 1.5	± 0.8	± 0.8	± 5.2	± 3.2	± 0.9
Carbon Dioxide[3]	Mean	n.a.	0.3	1.0	2.4	4.8	4.2	8.4	15.1
	SD		± 0.0	± 0.0	± 0.0	± 0.1	± 2.3	± 0.9	± 0.3
Volatile Organic Compounds[3]	Mean	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	0.1	0.1	< 0.1
	SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues[3]	Mean	2.0	7.8	19.5	31.3	45.7	57.9	69.3	80.9
	SD	± 0.0	± 0.2	± 0.1	± 0.2	± 1.2	± 2.6	± 1.5	± 1.6
Total Recovery[2]	Mean	101.3	99.1	100.4	97.4	99.0	95.8	100.1	98.3
	SD	± 0.7	± 1.3	± 1.3	± 1.0	± 0.4	± 3.8	± 0.8	± 2.3

n.d.: not detected, n.a.: not analysed, DAT: days after treatment, SD: standard deviation
[1] Minor components are summed up to sum of unidentified / diffuse residues.
[2] Difference to Material Balance values due to rounding errors as well as clean up and chromatographic losses.
[3] Values taken from Material Balance.

Aclonifen degraded at a moderate rate in the four soils. The reported best-fit non-normalised DT_{50} values ranging from 26.4 to 85.8 days. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1) along with all other aerobic soil data relied on. Full details of the evaluation are provided in the summary for KCA 7.1.2.1.1/07. The resulting best-fit DT_{50} value for the trigger endpoint is summarised below in Table 7.1.1.1- 49.

Table 7.1.1.1- 49: Degradation rate of aclonifen under aerobic conditions at 20 °C (best-fit DT_{50} values for trigger endpoints)

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob > t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted] Hof AXXa	SFO	98.2	k = 0.0146	2.04	2.83e-09	n.r.	n.r.	47.4	157.5
[redacted] am [redacted]	DFOP	103.5	k1 8.874e-02 k2 8.246e-03 g 2.546e-01	2.202	0.00479 5.44e-07 -	- - 1.504e-01	- - 0.359	49.22	243.6
[redacted] Hof	DFOP	104.6	k1 6.366e-03 k2 9.068e-01 g 8.785e-01	1.812	2.77e-11 0.0454 -	- - 8.76e-01	- - 0.916	85.83	331
[redacted] II	DFOP	102.0	k1 1.020e-01 k2 1.302e-02 g 3.268e-01	2.318	0.00954 6.41e-06 -	- - 1.633e-01	- - 0.490	26.35	146.5

n.r. not relevant

III. CONCLUSION

Aclonifen was moderately degraded and mineralised in soil under aerobic conditions in the laboratory in the dark. Carbon dioxide was formed at maxima of between 13.8 to 29.4% AR in soil treated the

phenoxy label. The best-fit non-normalised DT_{50} values were between 26.4 and 85.8 days and DT_{90} values from 146.5 to 331 days in the tested soils.

Besides carbon dioxide and non-extractable residues (NER), no metabolites were detected during the study. Non-extractable residues (NER) increased to a maximum of 42.1 to 70.9% AR at the end of the study, which is an indication for biotic degradation of aclonifen.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002) and is considered valid to assess the aerobic degradation of [phenoxy-UL-^{14}C] aclonifen in soil.

Assessment and conclusion by RMS:

CA 7.1.1.2 Anaerobic degradation

The route of anaerobic degradation of aclonifen in soil has been investigated in three ~~two~~ studies (CA 7.1.1.2/01, CA 7.1.1.2/02 and CA 7.1.1.2/04).

Report reference	Author, Year	Aniline Label	Phenoxy Label	Comment
KCA 7.1.1.2/01 M-165109-01-1	[redacted], 1989	✓	×	Submitted for first approval of aclonifen. Considered not acceptable in EFSA (2008) 149.
KCA 7.1.1.2/02 M-404038-01-1	[redacted] & [redacted] 2011	✓	×	New data not yet reviewed under UP.
KCA 7.1.1.2/07 M-687746-01-1	[redacted], 2020	×	✓	New data not yet reviewed under UP.

Study KCA 7.1.1.2/01 was evaluated during the previous EU review and was considered acceptable to assess the aerobic degradation of aclonifen but not the anaerobic degradation. Study KCA 7.1.1.2/02 and KCA 7.1.1.2/04 are provided as new data not yet reviewed under uniform principles.

Cleavage of aclonifen or its minor soil metabolite M-01 could lead to the formation of phenol (or hydroquinone from cleavage of M-01). A review of the published information on phenol and hydroquinone under anaerobic conditions is provided in KCA 7.1.1.2/03 ([redacted], 2019; M-676285-02-1).

~~An additional study is ongoing to investigate the anaerobic degradation of [^{14}C-UL-phenoxy] aclonifen ([redacted] Study Title: "[Phenoxy-U-^{14}C] Aclonifen: Route of Degradation in Anaerobic Soil", Study No. VC/19/070). A final report for this study was not available in time to be included in this dossier. As agreed with the RMS, an updated dossier will be submitted by the notifier which will include the final report and its OECD summary.~~

Data Point:	KCA 7.1.1.2/01
Report Author:	[redacted]
Report Year:	1983
Report Title:	Aerobic and anaerobic degradation of CME 127 in the soil
Report No:	R003641
Document No:	M-165109-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: OECD 307 (2002) Degradation products not quantified in all samples. Recoveries not quantitative.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Supportive only

In the previous submission (EFSA (2008) 149), this study was evaluated and accepted as valid for aerobic soil but not for anaerobic soil. The PRAPeR meeting of experts found the study not acceptable to evaluate the anaerobic degradation of aclonifen due to the low recovery of radioactivity. Hence a summary of this study is not presented in this part of the dossier.

Data Point:	KCA 7.1.1.2/02
Report Author:	[redacted]; [redacted]
Report Year:	2011
Report Title:	[Aniline-14C] aclonifen: Anaerobic soil metabolism
Report No:	MEF-11/126
Document No:	M-404038-01-1
Guideline(s) followed in study:	OECD 307; US EPA OPPTS 835.4100 and 835.4200
Deviations from current test guideline:	Current guideline: OECD 307 (2002) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The anaerobic biotransformation of radiolabeled [aniline-^{14}C]aclonifen was studied in a silt loam (pH 6.6 in water, 6.3 in $CaCl_2$, organic carbon 2.6%) from [redacted] am [redacted] 4a, Germany. Aclonifen was applied at a nominal rate of 640 µg a.i./ 100 g soil, equivalent to 2400 g a.i./ha.

The test systems consisted of glass Erlenmeyer flasks, each containing 100 g of soil (dry weight equivalent). During the aerobic study phase, air-permeable traps were attached for the collection of CO_2 and volatile organics (static test systems). The soil was maintained under aerobic conditions for 30 days in the dark at 20 ± 2 °C and at a soil moisture of 55% maximum water holding capacity.

Following the aerobic phase, the samples were flooded with oxygen-free water and maintained in the dark at 20 ± 2°C for 161 days after soil flooding (DASF-161, DAT-191). The flasks were equipped with sealable double-valve glass stoppers connected to plastic gas sampling bags for the collection of CO_2 and organic volatiles. Then, the vessels were placed in a nitrogen flooded box within an incubation chamber.

Samples were analysed after 0, 7, 14 and 30 days of aerobic incubation (DAT) and after 0, 2, 7, 14, 21, 42, 63, 91, 125 and 161 days of anaerobic incubation. During the aerobic phase, the soil samples were extracted with 3 x 80 mL acetonitrile/ water (80/20, v/v) at ambient conditions and with 80 mL acetonitrile/water (80/20, v/v) and 100 mL methanol/ 0.1 N HCl (50/50, v/v) each under reflux conditions. At each sampling interval during the anaerobic incubation phase, the water was decanted from each test system. Then, the soil was extracted as described for the aerobic samples, except that for the second aggressive extraction step 80 mL of methanol/ 0.1 N HCl (50/50, v/v) were used. each of the extracts were separately analysed for volume and radioactivity. The residues of [^{14}C]aclonifen and its transformation products were analysed by HPLC.

The average total material balance in the soil/water systems was 99.3% of the applied radioactivity. In the aerobic phase, extractable [^{14}C] residues in soil decreased from 97.6% at day 0 to 73.3% on day 30. Non-extractable (bound) residues increased from 0.8% at day 0 to 26.0% at day 30. At the end of the aerobic phase, 1.7% or less of the applied radioactivity was present as CO_2. Volatile organic compounds were not detected in significant amounts (<0.1% of AR).

The concentration of aclonifen in the aerobic phase decreased from 97.6% of the applied amount at day 0 to 72.8% at day 30. Soil was flooded at this point in order to establish anaerobic conditions. During the anaerobic phase, radioactivity in the water layer and the ambient and aggressive organic extracts decreased from 77.0% at flooding to 9.2% by the end of the study.

Non-extractable residues in soil increased from 24.2% at flooding to 88.3% of the applied amount at day 161. They were further characterized for one sample taken on day 161 in which the non-extractable residues (NER) accounted for 89.4% of AR. Through fractionation into the fulvic acid, humic acid and humin components, 4.8, 35.8, and 49.4% of the NER were associated with these fractions, respectively. Especially the formation of the humin fraction shows the irreversible binding of the NER to soil.

No significant amounts of CO_2 and volatile organic compounds were produced during the anaerobic phase.

During the anaerobic phase, the concentration of aclonifen in soil decreased from 77.0% at flooding to 0.3% of AR at study termination. Besides aclonifen, up to 22 minor peaks were detected in the HPLC-chromatograms. Most of them appeared from 42 days after flooding onwards when the redox potential in both, the water layer and the soil, reached finally anaerobic conditions. The maximum amount of a single minor transformation was 3.8% of AR (single value). The unidentified radioactivity, containing further non-characterized minor peaks, accounted for ≤ 0.8% of AR throughout the study period.

The disappearance of aclonifen in the entire systems (water layer and soil extracts) during the anaerobic phase could be best described using a single first order model. The calculated DT_{50} and DT_{90} values of Aclonifen in soil [redacted] am [redacted] 4a were 21.4 and 70.9 days, respectively. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit DT_{50} values of 70.53 days for the aerobic phase and 17.96 days for the anaerobic phase.

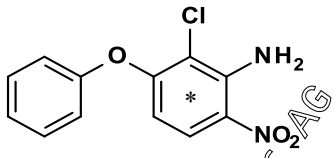
I. MATERIALS AND METHODS

A. Materials

1. Test material:

[Aniline-UL-^{14}C] Aclonifen

Chemical structure:



* labelling position

Chemical name (IUPAC):	2-chloro-6-nitro-3-phenoxy-aniline
Batch no.:	KATH 6486
Specific radioactivity:	6.59 MBq/mg
Radiochemical purity:	> 99%
CA registry number:	74070-46-5

2. Soil: A silt loam soil from Germany was used. The soil was freshly collected from the the upper soil horizon (0-20cm) slightly moistened and homogenized by sieving (< 2 mm). The main characteristics of the soils are shown below.

Table 7.1- 4: Physico-chemical characteristics of the soil used in anaerobic soil study

Characteristic / Code	Units	[redacted] am [redacted] 4a
Origin	State, Country	Germany
Location	Geographic Location (City / State / Country)	D-[redacted] / Northrhine-Westfalia / Germany
Particle Size Analysis		
Sand (0.05 - 2.0 mm)	%	28
Silt (0.002 - 0.05 mm)	%	55
Clay (<0.002 mm)	%	17
Textural Class	USDA	Silt loam
pH	0.01 M $CaCl_2$ (1:2)	6.3
	1M KCl	6.0
	Water (1:1)	6.6
Organic Matter	%	4.48
Organic Carbon	%	2.6
Cation Exchange Capacity	mEq/100 g	14.4
Bulk Density (disturbed)	g/cm^3	1.08
Water Holding Capacity at 0.33 bar	%	24.2
Maximum Water Holding Capacity	%	58.6
Soil Microbial Biomass (Aerobic Initial)	mg microbial C/kg soil	1148 (Day 0)
Soil Microbial Biomass (Anaerobic Final)	CFU/ g soil	12 x 10^3 - 12.7 x 10^4 (Day 191)

B. Study design and methods

In-life dates:

15 July 2010 – 14 March 2011

1. Experimental conditions: The degradation of [U-^{14}C-aniline] labelled aclonifen was investigated under aerobic / anaerobic conditions in a silt loam soil at 20 °C in the dark. [^{14}C]-aclonifen was applied to soil samples at an application rate of 640 µg per 100 g of soil, which is equivalent to a field rate of 2.4 kg/ha.

The test systems consisted of glass Erlenmeyer flasks, each containing 100 g of soil (dry weight equivalent). During the aerobic phase of the study, soil samples were incubated under static conditions in flasks equipped with a combined solid phase trap for the collection of carbon dioxide (soda lime) and volatile organic compounds (polyurethane bung). The soil was maintained under aerobic conditions for 30 days at a soil moisture content equivalent to 55% of maximum water holding capacity.

Following the aerobic phase, the samples were flooded with oxygen-free water to an approximate depth of 1-3 cm above the soil surface and maintained in the dark for 161 days post flooding (191 days in total). During the anaerobic phase, the soil samples were placed in a nitrogen flooded box within an incubation chamber. The combined solid phase traps were replaced with plastic gas sampling bags for the collection of carbon dioxide (CO_2) and organic volatiles.

2. Sampling: Duplicate samples were analysed after 0, 7, 14 and 30 days of aerobic incubation and after 0, 2, 7, 14, 21, 42, 63, 91, 125 and 161 days post flooding.

3. Description of analytical procedures: At analysis, samples incubated under anaerobic conditions were first separated into soil and water phases. Soil samples were initially extracted with acetonitrile/ water (4/1, v/v) three times under ambient conditions, then with acetonitrile/water (4/1, v/v) under reflux conditions and finally with methanol/ 0.1N HCl (1/1, v/v) under reflux conditions. Radioactivity in the water phase and extracted from soil was quantified by liquid scintillation counting (LSC). The remaining residue after completion of extractions was combusted to quantify non-extractable residue (NER). NER was further characterised by fractionation of the organic material. This analysis was conducted on an extracted soil sample from the final time-point. This radioactive material was separated into three fractions:

- Soluble in 0.5M sodium hydroxide solution and remaining in solution on acidification with hydrochloric acid (fulvic acid fraction).
- Soluble in 0.5M sodium hydroxide solution but insoluble on acidification with hydrochloric acid (humic acid fraction).
- Insoluble in 0.5M sodium hydroxide solution (humin fraction).

The quantity of radioactive volatiles generated during the study was determined by processing the elements that made up each of the volatile traps. Polyurethane foam was extracted with ethyl acetate. CO_2 adsorbed on soda lime was released by digesting the soda lime with hydrochloric acid and re-trapped in an appropriate scintillation cocktail. Volatile radioactivity contained in plastic gas sampling bags was oxidized in a muffle furnace and trapped in an appropriate scintillation cocktail.

Components present in the water phase, the combined ambient soil extracts, and the two reflux soil extracts were separately characterised and quantified by HPLC. In addition, selected ambient soil extracts were analysed by TLC.

4. Degradation kinetics: DT_{50} and DT_{90} values for the degradation of aclonifen have been calculated in the report following the recommendations of the FOCUS work group (2005) using the software KinGUI (version v1.1). Additionally, DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the current recommendations of the FOCUS work group (2006, 2014) using the software KinGUI (version 2.1).

II. RESULTS AND DISCUSSIONS

A. Mass balance

The mean material balance was 99.3% (range from 97.5 to 102.9% AR). A summary of the recoveries at each sampling time interval is provided in Table 7.1.1.2- 1.

B. Findings

In the aerobic phase, extractable [^{14}C] residues in soil decreased from 97.6% at day 0 to 73.3% on day 30. Non-extractable (bound) residues increased from 0.8% at day 0 to 26.0% at day 30. At the end of the aerobic phase, 1.7% or less of the applied radioactivity was present as CO_2. Volatile organic compounds were not detected in significant amounts (< 0.1% of AR).

During the anaerobic phase, radioactivity in the water layer and the ambient and aggressive organic extracts decreased from 77.0% at flooding to 9.2% by the end of the study.

Non-extractable residues in soil increased from 24.2% at flooding to 88.3% of the applied amount at day 161. They were further characterized for one sample taken on day 161 in which the non-extractable residues (NER) accounted for 89.4% of AR. Through fractionation into the fulvic acid, humic acid and humin components, 4.8, 35.8, and 49.4% of the NER were associated with these fractions, respectively. Especially the formation of the humin fraction shows the irreversible binding of the NER to soil.

No significant amounts of CO_2 and volatile organic compounds were produced during the anaerobic phase.

Table 7.1.1.2- 1: Distribution of radioactivity in [redacted] am [redacted] soil incubated under aerobic / anaerobic conditions

Extract	Replicate	Time (days)						
		Aerobic phase				Anaerobic phase		
		0	7	14	30	0	2	7
Water phase	A	-	-	-	-	0.4	0.4	0.5
	B	-	-	-	-	0.4	0.4	0.4
	Mean	**-**	**-**	**-**	**-**	**0.4**	**0.4**	**0.5**
Ambient extract	A	92.9	83.5	78.5	68.6	70.5	65.8	63.5
	B	94.2	82.6	79.4	67.9	72.2	63.9	59.3
	Mean	93.6	83.0	79.0	68.2	71.4	64.9	61.4
Reflux Extract 1	A	3.1	4.0	3.7	3.7	4.1	3.8	3.7
	B	3.3	3.9	4.2	3.6	4.1	3.7	3.8
	Mean	**3.2**	**4.0**	**4.0**	**3.6**	**4.1**	**3.7**	**3.8**
Reflux Extract 2	A	0.8	1.0	1.2	1.4	1.2	1.3	1.2
	B	0.8	1.0	1.3	1.4	1.2	1.3	1.3
	Mean	**0.8**	**1.0**	**1.2**	**1.4**	**1.2**	**1.3**	**1.3**
Total extracted	A	96.9	88.5	83.4	73.7	76.3	71.1	69.0
	B	98.3	87.6	84.9	72.9	77.8	69.3	64.9
	Mean	**97.8**	**88.0**	**84.2**	**73.3**	**77.0**	**70.3**	**66.9**
Total volatiles	A	-	0.2	0.6	1.7	1.7	1.7	1.7
	B	-	0.3	0.6	1.8	1.5	1.7	1.7
	Mean	**-**	**0.2**	**0.6**	**1.7**	**1.6**	**1.7**	**1.7**
Unextracted soil residue	A	0.8	9.0	13.9	25.3	24.9	27.8	28.1
	B	0.8	9.3	13.2	26.7	23.4	28.7	30.9
	Mean	**0.8**	**9.2**	**13.6**	**26.0**	**24.2**	**28.3**	**29.5**
Total	A	97.8	97.8	97.9	100.8	102.9	100.8	98.8
	B	99.1	97.2	98.8	101.4	102.8	99.7	97.5
	Mean	**98.4**	**97.5**	**98.4**	**101.1**	**102.9**	**100.3**	**98.1**

Table 7.1.1.2-1 cont: Distribution of radioactivity in [redacted] am [redacted] soil incubated under aerobic / anaerobic conditions continued

Extract	Replicate	Time (days)						
		Anaerobic phase (continued)						
		14	**21**	**42**	**63**	**91**	**125**	**161**
Water phase	A	0.4	0.5	0.5	0.4	0.2	0.2	0.2
	B	0.4	0.4	0.6	0.4	0.3	0.2	0.2
	Mean	**0.4**	**0.5**	**0.6**	**0.4**	**0.3**	**0.2**	**0.2**
Ambient extract	A	53.9	46.8	11.3	7.7	6.6	5.8	6.0
	B	56.6	49.1	12.9	7.8	6.6	5.7	6.0
	Mean	**55.3**	**47.9**	**12.1**	**7.8**	**6.6**	**5.8**	**6.0**
Reflux Extract 1	A	3.9	3.8	2.9	3.1	2.6	3.0	2.1
	B	3.7	3.8	2.9	3.2	2.6	3.0	2.1
	Mean	**3.8**	**3.8**	**2.9**	**3.2**	**2.6**	**3.0**	**2.1**
Reflux Extract 2	A	1.1	1.4	1.1	1.2	1.0	1.1	0.9
	B	1.3	1.4	1.2	1.2	1.0	1.0	0.9
	Mean	**1.2**	**1.4**	**1.2**	**1.2**	**1.0**	**1.1**	**0.9**
Total extracted	A	59.3	52.5	15.9	12.4	10.5	10.2	9.2
	B	62.0	54.6	17.5	12.6	10.4	9.9	9.1
	Mean	60.6	53.5	16.7	12.5	10.4	10.1	9.2
Total volatiles	A	1.7	1.7	1.7	1.7	1.7	1.7	1.7
	B	1.7	1.7	1.7	1.7	1.7	1.7	1.7
	Mean	**1.7**	**1.7**	**1.7**	**1.7**	**1.7**	**1.7**	**1.7**
Unextracted soil residue	A	37.6	43.6	80.4	83.5	88.6	89.2	89.4
	B	34.4	41.9	78.5	83.7	89.3	89.5	87.2
	Mean	**36.0**	**42.8**	**79.5**	**83.5**	**88.9**	**89.3**	**88.3**
Total	A	98.5	97.8	98.0	97.5	100.7	101.0	100.3
	B	98.1	98.2	97.7	98.0	101.4	101.1	98.1
	Mean	**98.3**	**98.0**	**97.8**	**97.8**	**101.0**	**101.0**	**99.2**

The concentration of aclonifen in the aerobic phase decreased from 97.6% of the applied amount at day 0 to 72.8% at day 30. During the anaerobic phase, the concentration of aclonifen in soil decreased from 77.0% at flooding to 0.3% of AR at study termination. Besides aclonifen, up to 22 minor peaks were detected in the HPLC-chromatograms. Most of them appeared from 42 days after flooding onwards when the redox potential in both the water layer and the soil, reached finally anaerobic conditions. The maximum amount of a single minor transformation was 3.8% of AR (single value). The unidentified radioactivity, containing further non-characterized minor peaks, accounted for ≤ 0.8% of AR throughout the study period.

Table 7.1.1.2- 2: Characterisation of radioactivity in [redacted] am [redacted] soil incubated under aerobic / anaerobic conditions

Extract	Replicate	Time (days)						
		Aerobic				Anaerobic		
		0	7	14	30	0	2	7
Aclonifen	Mean	97.6	88.0	84.2	72.8	77.0	70.2	66.2
	SD (±)	1.0	0.6	1.1	0.4	1.1	1.4	3.0
Sum of unknown degradation products	Mean	-	-	-	0.5	-	0.1	0.1
	SD (±)	-	-	-	0.1	-	-	0.1
Number	Total	-	-	-	1	-	1	1
Maximum for single metabolite	Mean	-	-	-	0.5	-	0.1	0.1
	SD (±)	-	-	-	0.1	-	-	0.1
Total extracted	Mean	97.6	88.0	84.2	73.3	77.0	70.3	66.9
	SD (±)	1.0	0.6	1.1	0.6	1.1	1.4	2.9
Total volatiles	Mean	0.2	0.2	0.6	1.7	1.6	1.7	1.7
	SD (±)	-	0.1	0.0	0.1	0.1	0.0	0.0
Unextracted soil residue	Mean	0.8	9.2	13.6	26.0	24.2	28.3	29.5
	SD (±)	0.0	0.2	0.5	1.0	1.1	0.6	2.0
Total	Mean	98.4	97.5	98.4	101.1	102.9	100.3	98.1
	SD (±)	0.9	0.4	0.6	0.4	0.1	0.8	0.9

Table 7.1.1.2-2 cont: Characterisation of radioactivity in [redacted] am [redacted] soil incubated under aerobic / anaerobic conditions continued

Extract	Replicate	Time (days)						
		Anaerobic						
		14	21	42	63	91	125	161
Aclonifen	Mean	59.7	51.9	10.1	3.2	0.3	0.4	0.3
	SD (±)	1.9	1.7	1.8	0.3	0.0	0.1	0.1
Sum of unknown degradation products	Mean	0.9	1.6	6.5	8.9	10.1	9.4	9.0
	SD (±)	0.1	0.2	0.6	0.2	0.3	0.1	0.3
Number	Total	2	5	7	14	17	15	16
Maximum for single metabolite	Mean	0.9	1.2	3.4	1.7	1.5	1.5	1.5
	SD (±)	0.1	0.2	0.6	0.8	0.1	0.5	0.1
Total extracted	Mean	60.6	53.5	16.7	12.5	10.4	10.1	9.2
	SD (±)	1.9	1.5	1.1	0.1	0.1	0.2	0.1
Total volatiles	Mean	1.7	1.7	1.7	1.7	1.7	1.7	1.7
	SD (±)	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Unextracted soil residue	Mean	36.0	42.8	79.5	83.5	88.9	89.3	88.3
	SD (±)	2.3	1.2	1.3	0.1	0.5	0.2	1.6
Total	Mean	98.3	98.0	97.8	97.8	101.0	101.0	99.2
	SD (±)	0.3	0.3	0.2	0.4	0.5	0.1	1.6

When considering the data set from the moment at which the soils were flooded, aclonifen degraded at a moderately fast rate in soil under anaerobic conditions. The reported best fit DT_{50} value was 21.4 days assuming simple first order kinetics.

Table 7.1.1.2- 3: Degradation rate of aclonifen under anaerobic conditions at 20 ºC

Soil	Model	DT_{50} (days)	DT_{90} (days)	Min Chi² error (%)
[redacted] am [redacted] 4a	SFO	21.4	70.9	15.4

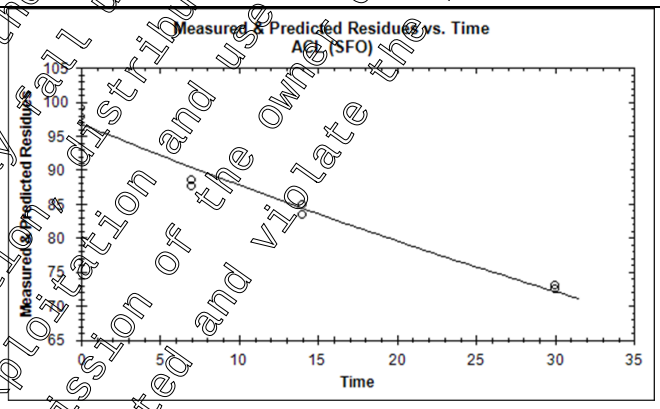
A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit DT_{50} values of 70.53 days for the aerobic phase and 17.96 days for the anaerobic phase.

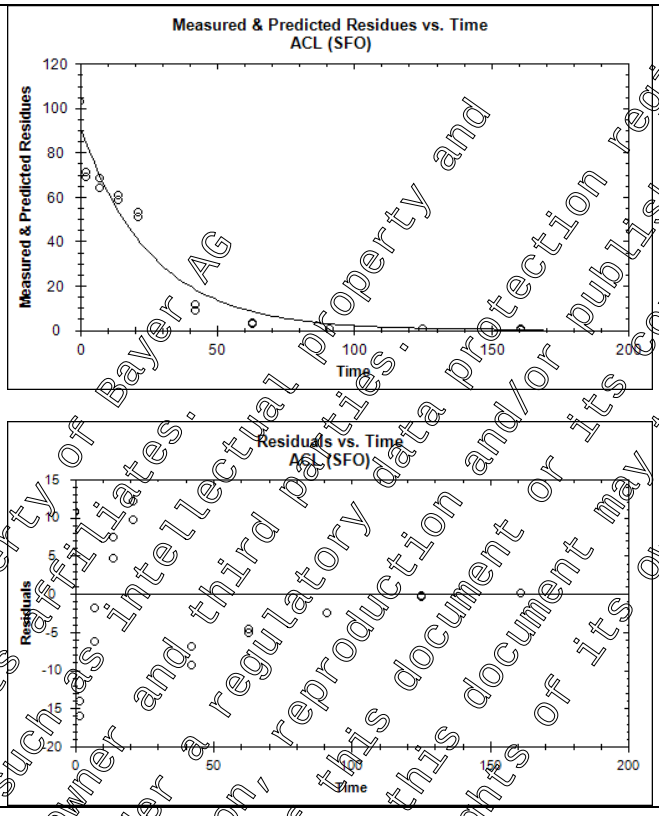
Table 7.1.1.2- 4: Degradation rate of aclonifen under aerobic and anaerobic conditions at 20 °C

Soil Phase	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Aerobic	SFO	99.1	k = 9.827e-03	1.398	4.77e-06	8.418e-03	0.011	70.53	334.3
Anaerobic		102.8	k = 0.038594	17.2	4.81e-08	0.029732	0.047	17.96	59.66

The degradation of aclonifen with time is graphed below. The data upon which this is based has been presented previously in Table 7.1.1.2- 3.

Table 7.1.1.2- 5: Degradation of aclonifen under aerobic / anaerobic conditions in [redacted] soil at 20 ºC with time

Trial / Best Fit Model	Graphical Representations
Aerobic / SFO	

Trial / Best Fit Model	Graphical Representations
Anaerobic / SFO	

III. CONCLUSION

Aclonifen degraded at a moderately fast rate in soil incubated under anaerobic conditions, with less than 0.5% of radioactivity remaining as aclonifen in [redacted] and [redacted] soil after 161 days. The principal degradation route was the formation of unextractable soil bound residues. Anaerobic metabolism of aclonifen led to the formation of a number of unidentified minor degradation products, none of which exceeded 4% and non extractable soil residues which reached a maximum of 89% after 91 days incubation and remained at this level until the end of the incubation period.

The best-fit DT$_{50}$ values, derived in accordance with FOCUS guidance document on degradation kinetics (2014), were 70.33 days for the aerobic phase and 17.96 days for the anaerobic phase.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002) and is considered valid to assess the anaerobic degradation of [aniline-UL-^{14}C] aclonifen in soil.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.1.2/03
Report Author:	[redacted]
Report Year:	2020
Report Title:	Aclonifen - Statement on the fate of phenol and hydroquinone in the environment
Report No:	VC/19/025H
Document No:	M-676285-02-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	not applicable
Acceptability/Reliability:	

Executive Summary

Any cleavage of the diphenyl ether linkage would result in the formation of phenol and hydroquinone from the phenyl ring. This document summarizes the published information on the fate of phenol and hydroquinone in anaerobic soil and photodegradation in aqueous solutions. The information on the fate of phenol and hydroquinone in anaerobic soil is summarised here.

Phenol and hydroquinone can be readily degraded anaerobically by many denitrifying, iron- or sulphate- reducing bacteria and by methanogenic consortia to carbon dioxide and methane. The metabolic pathways have been elucidated using isolated bacterial or fungal strains. Degradation rates were slower under anaerobic conditions than under aerobic conditions. Detailed summaries are provided for three publications which report the rate of degradation of phenol in soil and paddy soils incubated under anaerobic conditions.

Both compounds would be slowly degraded under flooded conditions if soil is anaerobic, but once it is no longer flooded and soil conditions become aerobic, phenol and hydroquinone will rapidly degrade. Neither compound will persist or accumulate in the environment under anaerobic conditions.

I. MATERIALS AND METHODS

First an overview of the route and rate of anaerobic degradation of phenol and hydroquinone, including metabolic pathways elucidated with isolated bacterial or fungal strains obtained from published literature is provided in the statement. Then fully detailed summaries of three publications are provided; one publication investigated the fate of phenol in soil collected from a uncultivated grassland site, while remaining two describe the fate of phenol in Japanese paddy soil. In all three publications the investigations included anaerobic laboratory soil incubations.

Reference 1.	Baker, M. D.; Mayfield, C. I.; Microbial and nonbiological decomposition of chlorophenols and phenol in soil Water, Air, and Soil Pollution (1980), 13(4), 411-24
Reference 2.	Shibata Atsushi; Inoue Yasushi; Katayama Arata Aerobic and anaerobic biodegradation of phenol derivatives in various paddy soils The Science of the total environment, (2006 Aug 31) Vol. 367, No. 2-3, pp. 979-87. Electronic Publication Date: 13 Mar 2006

Reference 3.	Shibata Atsushi; Toyota Koki; Miyake Katsuhide; Katayama Arata (lead author) Anaerobic biodegradation of 4-alkylphenols in a paddy soil microcosm supplemented with nitrate Chemosphere (2007), 68(11), 2096-2103

A short overview of the results pertinent to the fate of phenol in anaerobic soil is provided below.

II. RESULTS AND DISCUSSION

Baker & Mayfield (1980) examined the fate of phenol and selected chlorophenols in a clay loam soil under aerobic and anaerobic conditions. Flasks were incubated at 23 °C under aerobic or anaerobic conditions for up to 40 days. An additional experiment was conducted in sterile sand under aerobic conditions at 4, 26 and 60°C for up to 32 days. Analysis of soil extracts with UV spectrophotometry showed that phenol was rapidly degraded by microorganisms in aerobically incubated soil at 23 °C. Degradation by microorganisms in anaerobically incubated soil at 23 °C was very much slower. Direct microscopic observation revealed that phenol stimulated aerobic and to a lesser extent, anaerobic microbial growth in soil. Phenol underwent rapid non-biological degradation in sterile silica sand.

Shibata *et al*. (2006) examined microbiological degradation of phenol and some of its alkyl derivatives under both aerobic and anaerobic conditions in seven Japanese paddy soils. Aerobic biodegradation of phenol and its alkyl derivatives was detected in all the paddy soils examined. The half-lives for phenol ranged from 2 to 7 days. Anaerobic biodegradation of phenol was detected in three soils with the half-lives ranging from 24 to 260 days. The three soils were characterized by low contents of nitrate and iron oxides. Other soil properties did not show any significant correlations with the anaerobic degradation rates. These results suggest that phenol will be rapidly degraded when paddy soil is not flooded and under aerobic conditions, while under flooded anaerobic conditions, degradation will be slower.

Shibata *et al.* (2007) observed the anaerobic degradation of phenol and some of its alkyl-derivatives in a paddy soil supplemented with nitrate. Complete degradation of 0.3 mM of phenol was observed within six days in paddy soil supplemented with 5 mM of sodium nitrate. Compared with the original soils, Betaproteobacteria became predominant in the microcosm during the degradation of phenol. Paddy soil supplemented with sulfate or iron (III) as electron acceptors did not degrade phenol.

III. CONCLUSION

Any cleavage of the diphenyl ether linkage would result in the formation of phenol and hydroquinone from the phenyl ring. A review of the published data on phenol and hydroquinone enabled conclusions on their fate and behaviour in the environment to be made. Neither compound will persist or accumulate in the environment under anaerobic conditions.

Assessment and conclusion by applicant:
The position paper is considered valid to aid assessment of the fate of phenol and hydroquinone in anaerobic soil.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.1.2/07
Report Author:	[redacted]
Report Year:	2020
Report Title:	[Phenoxy-U-14C] aclonifen: Route of degradation in anaerobic soil
Report No:	VC/19/070
Document No:	M-687746-01-1
Guideline(s) followed in study:	OECD 307
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary

The anaerobic biotransformation of radiolabeled [phenoxy-UL-^{14}C]aclonifen was studied in a silt loam (pH 6.0 in water, 5.7 in $CaCl_2$, organic carbon 2.6%) from [redacted] am [redacted] 4a, Germany. Aclonifen was applied at a nominal rate of 400 µg a.i./ 100 g soil, equivalent to 1500 g a.i./ha.

The test systems consisted of glass flasks, each containing 100 g of soil (dry weight equivalent). During the aerobic study phase, soil samples were incubated under flow-through conditions with a continuous supply of humidified air pumped across the soil surface. The air flow exiting the flasks was passed through a series of traps for the collection of CO_2 and volatile organics. The soil was maintained under aerobic conditions for 30 days in the dark at 20 ± 2 °C and at a soil moisture of 55% maximum water holding capacity.

Following the aerobic phase, the samples were flooded with oxygen-free water and maintained in the dark at 20 ± 2°C for 122 days after soil flooding (DASF-122, DAT 152). During the anaerobic phase, the soil samples were incubated under flow-through conditions with a continuous supply of nitrogen pumped through the surface of the water. Gas flow exiting the flasks was passed through a series of volatile traps.

Samples were analysed after 0, 14 and 30 days of aerobic incubation (DAT) and after 0, 3, 7, 14, 21, 31, 45 and 122 days of anaerobic incubation. During the aerobic phase, the soil samples were extracted with three times with acetonitrile/ water (4/1, v/v) at ambient conditions and then with acetonitrile/water (4/1, v/v) and methanol/ 0.1 N HCl (1/1, v/v) under reflux conditions. At each sampling interval during the anaerobic incubation phase, the water was decanted from each test system. Then, the soil was extracted as described for the aerobic samples. Water and soil extracts were pooled prior to analysis by HPLC.

The average total material balance in the soil/water systems was 98.2% of the applied radioactivity. In the aerobic phase, extractable [^{14}C] residues in soil decreased from 97.0% at day 0 to 65.6% on day 30. Non-extractable (bound) residues increased from 0.7% at day 0 to 27.1% at day 30. At the end of the aerobic phase, 3.5% of the applied radioactivity was present as CO_2. Volatile organic compounds were not detected.

The concentration of aclonifen in the aerobic phase decreased from 97.0% of the applied amount at day 0 to 65.2% at day 30. Soil was flooded at this point in order to establish anaerobic conditions. During the anaerobic phase, radioactivity in the water layer, ambient and reflux organic extracts decreased from 64.1% 3 days after flooding to 10.5% by the end of the study.

Non-extractable residues in soil increased from 29.0% AR 3 days after flooding to 92.6% of the applied amount at day 122 of the anaerobic phase. They were further characterized in samples taken

on day 45 of the anaerobic phase in which the non-extractable residues (NER) accounted for 78.3% of AR. Through fractionation into the fulvic acid, humic acid and humin components, it was shown 8.4, 42.0, and 49.6% of the NER were associated with these fractions, respectively. The formation of large amounts of humin fraction, in particular, shows the irreversible binding of the NER to soil.

Amounts of CO_2 remained low during the anaerobic phase reaching a maximum of 4.9% AR by the end of the study. Volatile organic compounds were not detected in significant amounts (≤0.1% of AR).

During the anaerobic phase, the concentration of aclonifen in soil decreased from 63.6% 3 days after flooding to 1.2% of AR at study termination. Besides aclonifen, up to 45 minor peaks were detected in the HPLC-chromatograms. Most of them appeared from 21 days after flooding onwards when the redox potential of the system became anaerobic. The maximum amount of a single minor transformation was 2.3% of AR (single value).

The degradation rate of aclonifen under anaerobic conditions was faster than under aerobic conditions. The slower aerobic rate continued for a few days post-flooding but once anaerobic conditions were properly established in the soil, the rate of degradation accelerated. The degradation rate under aerobic conditions was determined using a simple first order kinetic model, due to the limited number of time points. The disappearance of aclonifen in the entire systems (water layer and soil extracts) during the anaerobic phase could be best described using a biphasic Hockey Stick model.

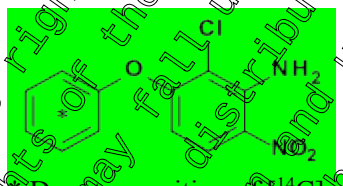
Evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014) resulted in best-fit DT_{50} values were 53.5 days for the aerobic phase and 24.9 days for the anaerobic phase.

I. MATERIALS AND METHODS

A. Materials

1. Test material: [phenoxy-UL-^{14}C]-aclonifen

Chemical structure:



* Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC): 2-chloro-6-nitro-3-phenoxy-aniline

Batch no.: RML 10726

Specific radioactivity: 4.52 MBq/mg

Radiochemical purity: 97.8%

CA registry number: 74070-46-5

2. Soil: A silt loam soil from Germany was used. The soil was freshly collected from the upper soil horizon (0-20 cm) slightly moistened and homogenized by sieving (≤ 2 mm). The main characteristics of the soils are shown below.

Table 7.1.1.2- 6: Physico-chemical characteristics of the soil used in anaerobic soil study

Characteristic / Code	Units	[redacted] am [redacted] 4a
Origin	State, Country	Germany
Location	Geographic Location (City / State / Country)	D-[redacted] / Northrhine-Westfalia / Germany

Particle Size Analysis		
Sand (0.05 - 2.0 mm)	%	15
Silt (0.002 - 0.05 mm)	%	70
Clay (<0.002 mm)	%	15
Textural Class	USDA	Silt loam
pH	0.01 M $CaCl_2$ (1:2)	5.7
	Water (1:1)	6.0
Organic Carbon	%	2.6
Cation Exchange Capacity	mEq/100 g	10.5
Maximum Water Holding Capacity	%	67.4
Water Holding Capacity at 0.1 bar (pF 2)	%	33.5
Water Holding Capacity at 0.33 bar (pF 2.5	%	23.8
Soil Microbial Biomass (DAT 0)	mg microbial C /kg soil	545
Soil Microbial Biomass (DAT 152)		288

B. Study design and methods

In-life dates:

October 2019 – April 2020

1. Experimental conditions: The degradation of [phenoxy-UL-^{14}C]-labelled aclonifen was investigated under aerobic / anaerobic conditions in a silt loam soil at 20 °C in the dark. [^{14}C]-aclonifen was applied to soil samples at an application rate of 416.4 µg per 100 g of soil, which is equivalent to a field rate of 1.561 kg/ha.

The test systems consisted of glass flasks, each containing 100 g of soil (dry weight equivalent). During the aerobic phase of the study, soil samples were incubated under flow-through conditions with a continuous supply of humidified air pumped across the soil surface. The air flow exiting the flasks was passed through a series of traps containing ethylene glycol (x1) and 2 M KOH (x2) to trap volatiles and CO_2. A polyurethane bung was placed in the soil flask headspace. The soil was maintained under aerobic conditions for 30 days at a soil moisture content equivalent to 55% of maximum water holding capacity.

Following the aerobic phase, the samples were flooded with oxygen-free water to 3 cm above the soil surface and maintained in the dark for 122 days post flooding (152 days in total). During the anaerobic phase, the soil samples were incubated under flow-through conditions with a continuous supply of nitrogen pumped through the surface of the water. The gas flow exiting the flasks was passed through a series of traps containing ethylene glycol (x1) and 2 M KOH (x2) to trap volatiles and CO_2.

2. Sampling: Duplicate samples were analysed after 0, 14 and 30 days of aerobic incubation and after 0, 3, 7, 14, 21, 31, 45, 80 and 122 days post flooding.

3. Description of analytical procedures: At analysis, samples incubated under anaerobic conditions were first separated into soil and water phases. Soil samples were initially extracted with acetonitrile/ water (4/1, v/v) three times under ambient conditions, then with acetonitrile/water (4/1, v/v) under reflux conditions and finally with methanol/ 0.1N HCl (1/1, v/v) under reflux conditions. Radioactivity in the water phase and extracted from soil was quantified by liquid scintillation counting (LSC). The remaining residue after completion of extractions was combusted to quantify non-extractable residue (NER). NER was further characterised by fractionation of the organic material. This analysis was conducted on an extracted soil sample from DASF 45 (DAT 85). This radioactive material was separated into three fractions:

- Soluble in 0.5M sodium hydroxide solution and remaining in solution on acidification with hydrochloric acid (fulvic acid fraction).

- Soluble in 0.5M sodium hydroxide solution but insoluble on acidification with hydrochloric acid (humic acid fraction).
- Insoluble in 0.5M sodium hydroxide solution (humin fraction).

The quantity of radioactive volatiles generated during the study was determined by LSC of the volatile traps. Polyurethane foam was extracted with acetonitrile.

Components present in the water phase and combined soil extracts were characterised and quantified by HPLC. In addition, the identity of the parent substance was confirmed in selected sample extracts by LC/MS.

4. Degradation kinetics: DT_{50} and DT_{90} values for the degradation of aclonifen in the soil extracts were determined following the recommendations of the FOCUS work group, with calculations performed according to the FOCUS guidance document on degradation kinetics. The aerobic and anaerobic phases were analysed separately. The Day 30 aerobic results were used as time zero for the anaerobic phase. The kinetic evaluations and the statistical calculations for the quality checks were implemented in the numerical software package CAKE 3.3.

II. RESULTS AND DISCUSSIONS

A. Mass balance

The mean material balance was 98.2% (range from 91.5 to 110.6% AR). A summary of the recoveries at each sampling time interval is provided in Table 7.1.1.2- 7.

B. Findings

In the aerobic phase, extractable [^{14}C] residues in soil decreased from 97.0% at day 0 to 65.6% on day 30. Non-extractable (bound) residues increased from 0.7% at day 0 to 27.1% at day 30. At the end of the aerobic phase, 3.5% of applied radioactivity was present as CO_2. Volatile organic compounds were not detected.

During the anaerobic phase, radioactivity in the water layer, ambient and reflux organic extracts decreased from 64.1% 3 days after flooding to 10.5% by the end of the study.

Non-extractable residues in soil increased from 29.0% AR 3 days after flooding to 92.6% of the applied amount at day 122 of the anaerobic phase. They were further characterized in samples taken on day 45 of the anaerobic phase in which the non-extractable residues (NER) accounted for 78.3% of AR. Through fractionation into the fulvic acid, humic acid and humin components, it was shown 8.4, 42.0, and 49.6% of the NER were associated with these fractions, respectively. Especially the formation of the humin fraction shows the irreversible binding of the NER to soil.

Amounts of CO_2 remained low during the anaerobic phase reaching a maximum of 4.9% AR by the end of the study. Volatile organic compounds were not detected in significant amounts (≤0.1% of AR).

Table 7.1.1.2- 7: Distribution of radioactivity in [redacted] am [redacted] soil incubated under aerobic / anaerobic conditions

Extract	Rep	Time (days)										
		Aerobic phase			Anaerobic phase							
		0	14	30	3	7	14	21	31	45	80	122
Water phase	A	-	-	-	1.6	1.7	1.2	1.3	1.8	0.9	0.7	0.6
	B	-	-	-	1.9	1.6	1.2	1.3	2.4	0.9	0.6	0.5
	Mean	-	-	-	**1.7**	**1.7**	**1.2**	**1.3**	**2.0**	**0.9**	**0.6**	**0.5**
Ambient extract	A	72.6	60.8	72.6	58.5	56.4	54.5	42.7	21.5	9.9	6.9	6.7
	B	73.0	62.3	73.0	58.3	55.1	49.5	40.5	21.3	9.9	7.1	6.5
	Mean	72.8	61.6	72.8	58.3	55.7	52.0	41.6	21.3	9.9	7.0	6.6
Reflux Extract 1	A	2.4	2.9	2.2	2.3	4.2	3.4	3.7	4.8	3.8	3.0	2.4
	B	2.5	3.5	2.8	3.1	5.0	3.2	4.2	4.4	4.0	3.1	2.3
	Mean	**2.4**	**3.2**	**2.5**	**2.7**	**4.6**	**3.3**	**3.9**	**4.6**	**3.9**	**3.1**	**2.3**
Reflux Extract 2	A	0.5	1.3	1.6	1.3	1.7	1.6	1.9	1.9	1.5	1.1	1.1
	B	0.6	1.2	1.4	1.5	2.9	1.7	1.7	1.7	1.4	1.1	1.0
	Mean	**0.6**	**1.3**	**1.5**	**1.4**	**1.8**	**1.7**	**1.8**	**1.8**	**1.5**	**1.1**	**1.1**
Total extracted	A	96.7	76.8	64.6	63.7	64.0	60.7	49.6	29.7	16.1	11.7	10.8
	B	97.3	77.7	66.5	64.8	63.6	58.6	47.7	29.8	16.2	11.9	10.3
	Mean	**97.0**	**77.3**	**65.6**	64.1	63.8	58.2	48.6	29.7	16.2	11.8	10.5
Total volatiles	A	-	1.5	3.4	3.2	3.8	3.8	4.1	4.3	3.5	4.8	4.2
	B	-	1.6	3.7	4.0	3.5	4.3	4.6	4.6	4.4	3.9	4.8
	Mean	-	**1.6**	**3.5**	3.6	3.6	4.1	4.4	4.4	4.0	4.3	4.5
Unextracted soil residue	A	0.7	21.0	23.8	28.3	27.2	30.3	46.2	67.2	77.9	81.9	95.5
	B	0.8	18.6	30.6	29.7	31.7	37.6	45.0	57.6	78.7	78.6	89.7
	Mean	**0.7**	**19.8**	**27.1**	**29.0**	**29.5**	**33.9**	**45.6**	**62.4**	**78.3**	**80.2**	**92.6**
Total	A	97.4	99.4	91.5	95.2	95.0	95.0	99.7	101.3	97.3	98.3	110.6
	B	98.1	97.9	100.9	98.4	98.8	97.4	97.3	92.0	99.3	94.4	104.9
	Mean	**97.7**	**98.6**	**96.2**	**96.8**	**96.9**	**96.2**	**98.5**	**96.6**	**98.3**	**96.3**	**107.7**

The concentration of aclonifen in the aerobic phase decreased from 97.0% of the applied amount at day 0 to 65.2% at day 30. During the anaerobic phase, the concentration of aclonifen in soil decreased from 63.6% 3 days after flooding to 1.2% of AR at study termination. Besides aclonifen, up to 45 minor peaks were detected in the HPLC-chromatograms. Most of them appeared from 21 days after flooding onwards when the redox potential of the system became anaerobic. The maximum amount of a single minor transformation was 2.3% of AR (single value).

Table 7.1.1.2- 8: Characterisation of radioactivity in [redacted] am [redacted] soil incubated under aerobic / anaerobic conditions

Extract	Rep	Time (days)										
		Aerobic phase			Anaerobic phase							
		0	14	30	3	7	14	21	31	45	80	122
Aclonifen	Mean	97.0	77.3	65.2	63.6	61.8	57.6	43.3	22.0	5.8	1.8	1.2
	SD (±)	0.3	0.5	0.6	0.5	0.1	2.0	0.7	0.7	0.3	0.1	0.0
Sum of minor metabolites	Mean	0.0	0.0	0.4	0.6	2.0	0.6	5.3	7.8	10.1	10.1	9.5
	SD (±)	0.0	0.0	0.4	0.0	0.2	0.6	0.2	0.7	0.4	0.1	0.3
Number	Total	0	0	1	1	6	3	37	39	36	32	45
Max single metabolite		-	-	0.7	0.6	0.9	0.4	0.7	1.6	1.9	2.3	1.9
Total extracted	Mean	97.0	77.3	65.6	64.1	63.8	58.2	48.6	29.7	16.2	11.8	10.5
	SD (±)	0.3	0.5	1.0	0.5	0.2	2.6	0.9	0.1	0.0	0.1	0.3
Total volatiles	Mean	-	1.6	3.5	3.6	3.6	4.1	4.4	4.4	4.0	4.3	4.5
	SD (±)		0.1	0.2	0.4	0.2	0.3	0.3	0.2	0.5	0.5	0.3
Unextracted soil residue	Mean	0.7	19.8	27.1	29.0	29.5	33.9	45.6	62.4	78.3	80.2	92.6
	SD (±)	0.1	1.2	3.6	0.7	2.3	3.7	0.6	4.8	0.4	1.7	2.9
Total	Mean	97.7	98.6	96.2	96.8	96.9	96.2	98.5	96.6	98.3	96.3	107.7
	SD (±)	0.3	0.8	4.7	1.6	1.9	1.2	1.2	4.7	1.0	2.0	2.8

The degradation rate under aerobic conditions was determined using a simple first order kinetic model, due to the limited number of time points.

Day 30 aerobic results were used as time zero for the anaerobic phase. Hockey Stick (HS) kinetics best described the degradation of aclonifen under anaerobic conditions, due to the initial lag phase in degradation whilst anaerobic conditions were established.

The degradation rate under anaerobic conditions was faster than under aerobic conditions. The slower aerobic rate continued for a few days post-flooding but once anaerobic conditions were properly established in the soil, the rate of degradation accelerated.

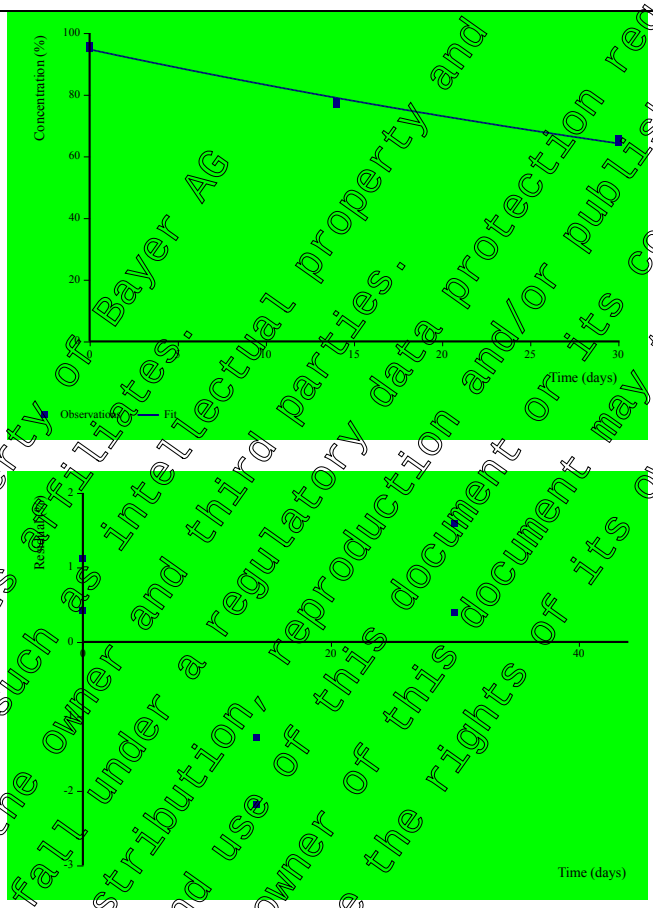
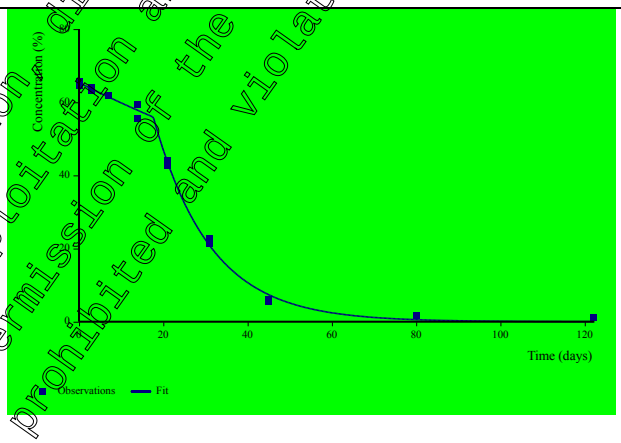
The best-fit DT_{50} values were 53.5 days for the aerobic phase and 24.9 days for the anaerobic phase.

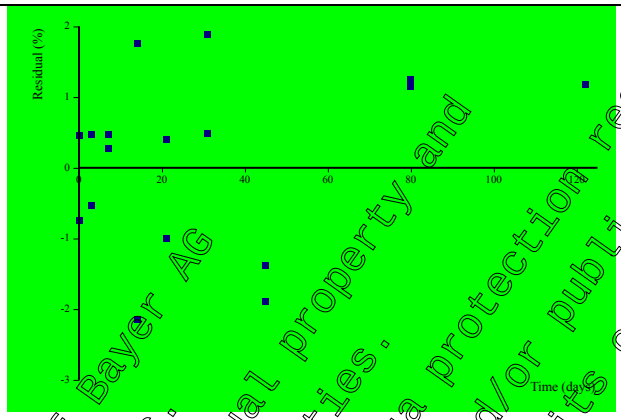
Table 7.1.1.2- 9: Degradation rate of aclonifen under aerobic and anaerobic conditions at 20 °C

Soil Phase	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	χ^2; %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Aerobic	SFO	94.68	k 0.01295	1.38	2.63E-005	0.01098	0.015	53.5	178
Anaerobic	HS	65.34	k1 0.008826 k2 0.07398 tb 17.64	2.25	1.94E-005 8.26E-012 -	0.005617 0.06579 13.01	0.012 0.082 22.26	24.9	46.7

The degradation of aclonifen with time is graphed below. The data upon which this is based has been presented previously in Table 7.1.1.2- 10.

Table 7.1.1.2- 10: Degradation of aclonifen under aerobic / anaerobic conditions in [redacted] am [redacted] soil at 20 °C with time

Trial / Best Fit Model	Graphical Representations
Aerobic / SFO	
Anaerobic / HS	

Trial / Best Fit Model	Graphical Representations
	

III. CONCLUSION

Aclonifen degraded at a moderately fast rate in soil incubated under anaerobic conditions, with less than 2% of radioactivity remaining as aclonifen in [redacted] an [redacted] soil after 122 days. The principal degradation route was the formation of unextractable soil bound residues. Anaerobic metabolism of aclonifen led to the formation of a number of unidentified minor degradation products, none of which exceeded 3%, and non extractable soil residues, which reached a maximum of 93% after 122 days anaerobic incubation.

The best-fit DT_{50} values, derived in accordance with FOCUS guidance document on degradation kinetics (2014), were 53.5 days for the aerobic phase and 24.9 days for the anaerobic phase.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 307 (2002) and is considered valid to assess the anaerobic degradation of [phenoxy-UL-^{14}C] aclonifen in soil.

Assessment and conclusion by RMS:

CA 7.1.1.3 Soil photolysis

The soil photolysis of aclonifen has been investigated in Study KCA 7.1.1.3/01 which was evaluated during the previous EU review and is still considered acceptable.

Report reference	Author, Year	Aniline Label	Phenoxy Label	Comment
KCA 7.1.1.3/01 M-174175-01-1	[redacted] & [redacted], 1994	✓	×	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.

Cleavage of aclonifen or its minor soil metabolite M-01 could lead to the formation of phenol (or hydroquinone from cleavage of M-01). A review of the published information on phenol and

hydroquinone did not locate work on the photolytic degradation of phenol and hydroquinone on soil surfaces in the presence of light. However, the rate of degradation of phenol at levels derived from the degradation of aclonifen in soil would be expected to be in the order of a few hours. The rate of degradation of hydroquinone in soil is also very rapid at ca. 100% degradation after 1 day. Thus there is no additional environmental risk from the exposure of any residues formed from aclonifen being exposed to sunlight.

Data Point:	KCA 7.1.1.3/01
Report Author:	[redacted]; [redacted]
Report Year:	1994
Report Title:	Photodegration study of [14C]-Aclonifen on soil
Report No:	R007084
Document No:	M-174175-01-1
Guideline(s) followed in study:	USEPA (=EPA): N,161-3
Deviations from current test guideline:	Not applicable
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The photolytic degradation of aclonifen on soil surfaces was investigated on moist [redacted] loamy sand soil (pH 6.4, 1.5% organic carbon), incubated at 75 % field moisture capacity and 22 °C, using [aniline-UL-^{14}C]-aclonifen. An application rate of 27 mg a.s./kg soil (dry weight) was used, equivalent to 2.7 kg a.s./ha. The treated samples were exposed to artificial irradiation from a Xenon lamp (with < 290 nm cut-off filter) with light and dark cycles of 12 hours each for a period of 30 days. A set of dark control soils treated at the same application rate for each radiolabel was incubated in the dark at 22 °C.

The mean recoveries of radioactivity were 96.6% ± 3.3 % and 97.6% ± 5.6 % for the irradiated and dark controls, respectively.

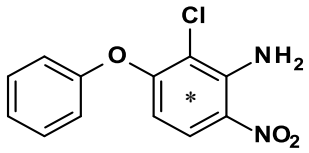
In the irradiated samples, aclonifen slowly degraded with a reported half-life of 75.3 days. Mineralization was low at 5 % AR. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit DT_{50} values of 74.02 days. The degradation rate of aclonifen was slightly enhanced in the presence of light leading to the formation of polar material and other minor photodegradation products. No degradation of aclonifen occurred during incubation in the dark.

I. MATERIALS AND METHODS

A. Materials

1. Test material: [Aniline-UL-^{14}C] Aclonifen

Chemical structure:



* labelling position

Chemical name (IUPAC): 2-chloro-6-nitro-3-phenoxy-aniline
Batch no.: 9XR302A
Specific radioactivity: 3.46 MBq/mg
Radiochemical purity: ≥ 99 %
CA registry number: 74070-46-5

2. Soil: A loamy sand soil from the UK was used. The soil was air-dried and homogenized by sieving (≤ 2 mm). The main characteristics of the soil are shown below.

Table 7.1.1.3- 1: Physico-chemical characteristics of the soil used in soil photolysis soil study

Characteristic / Code	Units	[redacted] 94/10/2
Origin	Country	Suffolk, UK
Location	City or Township	[redacted], [redacted][2]
Particle Size Analysis		
Total Sand (0.063 - 2.0 mm)	%	82.9
Silt (0.002 - 0.063 mm)	%	11.7
Clay (<0.002 mm)	%	5.4
Textural Class	USDA	Loamy sand
pH[1]		6.8
Organic Carbon	%	1.5
Cation Exchange Capacity	meq/100g	4.5
Maximum Water Holding Capacity	%	47.7
Soil Moisture During Incubation 75% FC	%	24.90
Soil Microbial Biomass Initial	Total plate counts	5.5 x 10^6 /g soil
Final (Light)		12.8 x 10^6 /g soil
Final (Dark)		17.0 x 10^7 /g soil

[1] 5 5
[2] Same location as Oglivie's Farm on [redacted] Road, [redacted].

B. STUDY DESIGN AND METHODS

1. In-life dates:

In-life dates: 10 May 1994 to 24 August 1994

2. Experimental design

The photolytic degradation of [UL-^{14}C-aniline] labelled aclonifen was studied following application to a loamy sand soil under artificial sunlight for a period of 30 days. The soil characteristics are summarised above. The soil was collected on 11 February 1994 and sieved to 2 mm prior to use. The same soil was also used to investigate the aerobic degradation of aclonifen (KCA 7.1.1.1/01, M-174177-02-1, [redacted], 1994; KCA 7.1.1.1/05, M-674036-01-1, [redacted], 2019; KCA

7.1.2.1.1/06, M-174228-01-1, England et al., 1988). The artificial sunlight was provided by a xenon arc lamp with filters to cut off any radiation below 290 nm.

The radiochemical purity and specific activity of [U-^{14}C-aniline] labelled aclonifen were ≥ 99 % and 3.46 MBq/mg, respectively.

Soil thin layer plates were prepared by mixing sieved soil and water and applying the slurry to glass plates using a TLC plate coater to give a soil depth of *ca*. 1 mm. The plates were air dried at ambient temperature. The soil samples were treated at a rate of 27 mg/kg, equivalent to 2.7 kg/ha and placed in an incubation chamber with air continuously drawn over the soil surface. Out-going air was passed through volatile traps to trap carbon dioxide (sodium hydroxide) and volatile organic products (ethylene glycol). The samples were incubated at 22 ºC under a 12 hour light / 12 hour dark cycle (irradiated samples) or in the dark (non-irradiated samples).

2. Sampling: Single soil samples were taken from both irradiated and non-irradiated systems at intervals throughout the study duration.

3. Description of analytical procedures: Soil samples from Day 0 until Day 14 were extracted at ambient temperature with acetonitrile / water (8/2, v/v), followed by a reflux extraction at 70 ºC with acetonitrile / water (8/2, v/v). At later timepoints additional ambient extraction steps were included. At 21 days soil samples were extracted with acetonitrile / water (8/2, v/v), followed by acetonitrile / water (2/8, v/v) at ambient temperature, and then with a reflux extraction at 70 ºC with acetonitrile / water (8/2, v/v). At the final timepoint (30 days) soil samples were extracted with acetonitrile / water (8/2, v/v), acetonitrile / water (2/8, v/v) and acetonitrile / 0.1M sodium bicarbonate (2/8, v/v) at ambient temperature followed by a reflux extraction at 70 ºC with acetonitrile / water (8/2, v/v). Radioactivity extracted from soil and in the volatile traps was quantified by liquid scintillation counting (LSC). The radioactivity contained in the sodium hydroxide traps was confirmed as $^{14}CO_2$ by precipitation as [^{14}C] barium carbonate. Radioactivity remaining unextracted from soil was quantified by combustion and LSC. Ambient soil extracts were pooled and concentrated prior to analysis by TLC. Selected extracts were also analysed by HPLC. Reflux extracts were not analysed except for the Day 30 irradiated sample reflux extract which was analysed by TLC.

4. Degradation kinetics: The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the current recommendations of the FOCUS work group (2006, 2014) using the software KinGUI (version 2.1).

II. RESULTS AND DISCUSSIONS

A. Data

Table 7.1.1.3- 2: Distribution of radioactivity in soil following irradiation of [aniline-UL-14C]-aclonifen on thin soil layers (as % of applied radioactivity)

Extract	Time (days)					
		Irradiated				
	Day 0	3	7	14	21	30
Ambient	93.8	92.2	97.9	84.6	80.2	77.7
Aclonifen	93.8	90.9	95.3	81.6	76.3	73.3
Polar material R_F 0.01 [A]	nd	1.3	2.6	3	3.9	4.4
Reflux	0.6	1.8	2.9	4	5.2	7.1
Aclonifen	-	-	-	-	-	4.5
Polar material R_F 0.01	-	-	-	-	-	2.2
M-04 R_F 0.30 [B]	-	-	-	-	-	0.2
D4 R_F 0.16	-	-	-	-	-	0.2
Unextracted soil residue	0.1	2	1.5	5.7	6.6	5.3

Carbon dioxide	na	0.2	0.8	1.8	2.8	4.1
Organic volatiles	na	<0.05	<0.05	<0.05	<0.05	<0.05
Total	94.5	96.2	103.1	96.1	94.8	94.2

[A] Called D2 in the report. R_f of 0.01 in normal phase TLC systems but not detected by HPLC. In an aerobic soil study with the same soil the radioactivity was established as polar radioactive material bound to aqueous soluble soil colloids (see KCA 7.1.1.1/01).
[B] Called RPA 407288 in the report

Table 7.1.1.3- 3: Distribution of radioactivity in dark control samples treated with [aniline-UL-14C]-aclonifen on thin soil layers (as % of applied radioactivity)

Extract	Time (days)					
	Day 0	Dark Controls				
		3	7	14	21	30
Ambient	93.8	93.2	104.6	92.2	91.7	91.7
Aclonifen	93.8	93.2	104.6	92.2	91.7	91.7
Reflux	0.6	1	1.7	2.1	3.1	3.4
Unextracted soil residue	0.1	0.3	2.5	0.9	1.3	1.3
Carbon dioxide	na	<0.05	<0.05	<0.05	<0.05	<0.05
Organic volatiles	na	<0.05	<0.05	<0.05	<0.05	<0.05
Total	94.5	94.5	108.8	95.2	96.1	96.4

B. Mass balance: The mean recoveries of radioactivity were 96.5% ± 3.3 % and 97.6% ± 5.6 % for the irradiated and dark controls, respectively.

C. Bound and Extractable: In irradiated samples the radioactivity recovered in the soil extracts accounted for 94.4 % at Day 0 and declined slightly to 84.8% at Day 30. The amounts of unextractable radioactivity were low, reaching a maximum of 7% of applied radioactivity.

D. Volatilization: Radiolabelled carbon dioxide evolved accounted for 4% in the irradiated samples. No organic volatiles were detected throughout the study.

E. Transformation of Parent Compound: Aclonifen was the principal radiolabelled component detected. Levels of parent accounted for all extracted radioactivity in Day 0 soil extracts and in non-irradiated samples throughout the study Levels of parent in irradiated samples declined over the incubation period to 73% of applied radioactivity after 30 days. A minor unidentified metabolite (D2) was detected as origin material in two normal phase TLC systems, at a maximum of 4% in ambient extracts and 2% in reflux extracts of the irradiated samples but was not detected by HPLC. In an aerobic soil study with the same soil the radioactivity was established as polar radioactive material bound to aqueous soluble soil colloids (see KCA 7.1.1.1/01). Two additional metabolites (D3 and D4) were detected at negligible amounts (0.2%) in the reflux extract of Day 30 irradiated soil sample. D3 was tentatively characterised as M-04 (called RPA 407288 in the report) based on its retention time in two normal phase TLC systems.

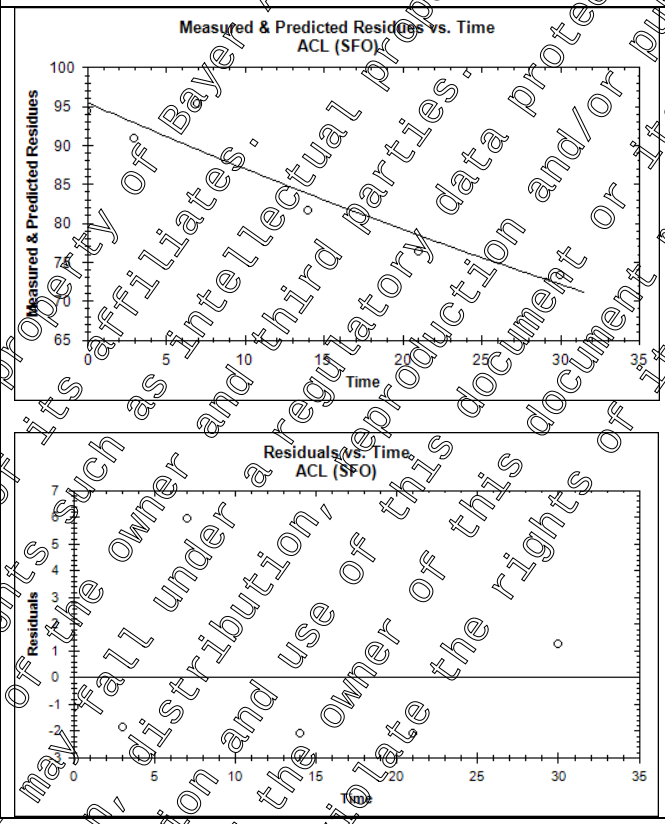
F. Degradation kinetics: Aclonifen slowly degraded under irradiated conditions. No degradation of [14C]-aclonifen occurred during incubation in the dark. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit DT_{50} value of 74.02 days for the irradiated soil.

Table 7.1.1.3- 4: DT_{50} values for aclonifen in soil photolysis study

Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]

Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
SFO	94.3	k = 0.009365	2.7	0.00268	0.006022	0.013	74.02	245.9

Table 7.1.1.3- 5: Degradation of aclonifen under photolytic conditions in [redacted] soil with time

Trial / Best Fit Model	Graphical Representations
Irradiated / SFO	

III. CONCLUSION

The degradation rate of aclonifen was slightly enhanced in the presence of light leading to the formation of unidentified polar material and other minor photodegradation products. No degradation of aclonifen occurred during incubation in the dark.

Assessment and conclusion by applicant:

The study was conducted in accordance with US EPA Guidelines Subdivision N, Section 161-3 (1982). The study is considered valid to assess the photodegradation of [aniline-UL-^{14}C] aclonifen in soil.

Assessment and conclusion by RMS:

CA 7.1.2 Rate of degradation in soil

CA 7.1.2.1 Laboratory studies

CA 7.1.2.1.1 Aerobic degradation of the active substance

The aerobic degradation of aclonifen in soil has been investigated in four reliable studies at 20°C, one evaluated during the previous EU review and three new studies, which are summarised under Point KCA 7.1.1.1. An additional soil study was evaluated during the previous EU review and was considered acceptable to assess the aerobic degradation of aclonifen but not the anaerobic degradation. For reasons elaborated in the study summary is now considered only as supporting data to assess the aerobic degradation of aclonifen and not reliable for anaerobic degradation.

For procedural reasons the two previously submitted studies also have to be included under Point KCA 7.1.2.1.1 in the current dossier (KCA 7.1.2.1.1/04 and KCA 7.1.2.1.1/02) but the summaries are provided in full only in Point KCA 7.1.1.1. A final soil degradation study KCA 7.1.2.1.1/03 was evaluated during the previous EU review where it was criticized but finally was considered acceptable to assess the aerobic degradation of aclonifen. For reasons elaborated in the study summary is now considered only as supporting data to assess the aerobic degradation of aclonifen.

Three kinetic evaluation reports (KCA 7.1.2.1.1/03, KCA 7.1.2.1.1/05 and KCA 7.1.2.1.1/07) are listed. For procedural reasons the two previously submitted kinetic evaluation reports also have to be included under Point KCA 7.1.2.1.1 in the current dossier (KCA 7.1.2.1.1/04 and KCA 7.1.2.1.1/05) but these reports are fully superseded by the latest kinetic evaluation report (KCA 7.1.2.1.1/07).

Finally, the aerobic degradation of aclonifen in soil has been investigated in soil at 10°C (KCA 7.1.2.1.1/06). KCA 7.1.2.1.1/06 was evaluated during the previous EU review and considered acceptable to assess the aerobic degradation of aclonifen. For reasons elaborated in the study summary is now considered only as supporting data to assess the aerobic degradation of aclonifen.

Report reference	Author, Year	Aniline Label	Phenoxy Label	Comment
KCA 7.1.2.1.1/01 M-174177-02-1	[redacted], 1994	✓	✓	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable. Summary provided under 7.1.1.1/01.
KCA 7.1.2.1.1/02 M-165109-01-1	[redacted] H., 1983	✓	✓	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered as supporting data only. Summary provided under 7.1.1.1/01.
KCA 7.1.2.1.1/03 M-165114-01-1	Anonymous., 1982	-	-	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered as supporting data only.
KCA 7.1.2.1.1/04 M-266704-01-1	[redacted], 2006	-	-	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Superseded by M-300678-01-1.
KCA 7.1.2.1.1/05 M-300678-01-1	[redacted], [redacted], 2008	-	-	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Superseded by M-674934-01-1.
KCA 7.1.2.1.1/06 M-174228-01-1	[redacted], [redacted] & [redacted], 1988	-	-	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered as supporting data only.

KCA 7.1.2.1.1/07 M-674934-01-1	[redacted] & [redacted], 2019	-	-	New data not yet reviewed under UP.

Data Point:	KCA 7.1.2.1.1/01
Report Author:	[redacted]; [redacted]
Report Year:	1995
Report Title:	[14C]-Aclonifen: Soil degradation under various experimental conditions in accordance with the danish pesticide registration requirements
Report No:	R008566
Document No:	M-174177-02-1
Guideline(s) followed in study:	BBA: IV, 4-1
Deviations from current test guideline:	Current Guideline: OECD 307 (2002) Soil pH not reported
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The details of this study are fully summarised under point KCA 7.1.1.1/01.

Data Point:	KCA 7.1.2.1.1/02
Report Author:	[redacted]
Report Year:	1983
Report Title:	Aerobic and anaerobic degradation of CME 127 in the soil
Report No:	R003641
Document No:	M-165109-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: OECD 307 (2002) Degradation products not quantified in all samples. Recoveries not quantitative.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Supportive only

The details of this study are fully summarised under point KCA 7.1.1.1/02.

Data Point:	KCA 7.1.2.1.1/03
Report Author:	[redacted]
Report Year:	1982
Report Title:	Behaviour of pesticide active ingredient in the soil - CME 127
Report No:	R003643
Document No:	M-165114-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: OECD 307 (2002) Time zero values not measured, only nominal applied values reported. Analytical method not validated. Reporting standard unacceptable.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Supportive only

Executive Summary

The rate of degradation of non-radiolabelled aclonifen, applied at a rate equivalent to 20 mg/kg, was investigated in two soils for up to sixteen weeks. The soils used were German standard soils, [redacted] 2.2 loamy sand and [redacted] 2.3 sandy loam soils. Soil samples were incubated in the dark, at a moisture content equivalent to 40% of maximum water holding capacity (MWHC) under aerobic conditions at 22ºC.

Samples of soil were taken for analysis after 2, 4, 8 and 16 weeks. Soil samples were exhaustively extracted with acetone and acetone / water and aclonifen residues partitioned into dichloromethane with final analysis by gas chromatography (GC).

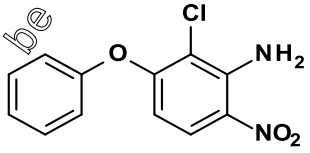
Aclonifen degraded at a moderate rate in standard [redacted] soils incubated under aerobic conditions. The degradation rate was similar to that measured in more recent, reliable studies.

The study deviates in a number of important aspects to the requirements of OECD 307 (2002). Time zero values are not measured, and only nominal applied values are reported. The analytical method does not meet the requirements of SANCO/3029/99 rev. 4 and cannot be considered as fit for purpose. Moreover, the report is extremely brief and not considered acceptable according to current requirements. Consequently the study must be considered as supportive only.

I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: Aclonifen



Chemical name (IUPAC) 2-chloro-6-nitro-3-phenoxy-aniline
CA registry number: 74070-46-5
Lot or batch number: Not recorded
Chemical purity: Not recorded

Stability of test compound: Shown to be stable under the conditions of the test

Application vehicle: Not recorded

2. Soil The soils used were German standard soils, [redacted] 2.2 and [redacted] 2.3, classified as loamy sand and sandy loam soils respectively. Specific details are shown below.

Table 7.1.2.1.1- 1: Physico-chemical characteristics of the soil used in aerobic soil study

Characteristic / Code	Units	[redacted] Standard soil 2.2	[redacted] Standard soil 2.3
Origin	Country	Germany	Germany
Location	City or Township	[redacted]	[redacted]
Textural Class	-	Loamy sand	Sandy loam
pH	-	6.0	7.0
Organic Carbon	%	2.64	1.06
Particle Size Analysis Clay (<20 µm)	%	14.5	24.7

B. STUDY DESIGN AND METHODS

Experimental design

Parameter		Description
Duration of test		112 days
Soil condition		Before treatment, relative soil humidity was adjusted to 40% and the samples stored at 22°C in the dark for 4 weeks.
Concentration in test system		20 mg/kg (nominal)
Number of replications		Not stated
Test apparatus		100 g dry weight equivalent of soil
Test material application	Identity of solvent	Not stated
	Volume of application solution	Not stated
	Application method	Not stated
Traps for CO_2 and organic volatiles		Not applicable
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	22°C
	Moisture content	40% of maximum water holding capacity (MWHC)
	Lighting	Dark

Sampling

Parameter		Description
Sampling intervals	Aerobic, non-sterile	Duplicate samples of soil were taken for analysis after 14, 28, 56 and 112 DAT. No time zero samples were taken
Soil sampling procedures		Complete treated samples were removed at each sampling time and extracted as detailed below.
Collection of CO_2 and volatile organics		Not applicable

Analytical procedures

Soil samples were exhaustively extracted with acetone and acetone / water and aclonifen residues partitioned into dichloromethane with final analysis by gas chromatography (GC).

Additional analysis of soil extracts was undertaken to detect potential soil metabolites. Soil samples incubated for 112 days were extracted with methanol, methanol / dilute hydrochloric acid (1/1 at pH 2.5) and methanol / caustic soda (2/1). The extracts were partitioned with dichloromethane or ether and concentrated. Concentrated extracts were analysed by TLC or methylated with diazomethane and analysed by GC. No soil metabolites representing greater than 1% were detected.

Finally four potential soil metabolites M-01 (called RPA 407074 in the report), M-02 (called RPA 508285 in the report), M-03 (called RPA 407291 in the report) and M-04 (called RPA 407288 in the report) were applied to [redacted] 2.3 soil at a rate equivalent to 5 mg/kg. The soil samples were incubated in the dark at 40% maximum water holding capacity at 22 °C for 28 days. The soils were extracted as described previously for aclonifen and concentrated extracts analysed by TLC. After 28 days the % of applied material detectable for M-01, M-02, M-03 and M-04 were 65%, 14%, barely detectable and 7% respectively.

Degradation kinetics

Half-life of the active ingredient stated in the report approximate values and are no longer considered valid. The rate of degradation of aclonifen was re-evaluated using KinGUI assuming simple first order kinetics for the first approval of aclonifen (EFSA (2008) 149).

II. RESULTS AND DISCUSSION

The decline of aclonifen with time in each soil is presented in Table 7.1.2.1.1-2.

Table 7.1.2.1.1- 2: Decline of aclonifen

Time (days)	mg aclonifen/ kg soil (dry matter)		
	[redacted] 2.2 (A)	[redacted] 2.2 (B)	[redacted] 2.3
14	14.7	14.8	12.7
28	13.0	12.9	10.6
56	10.6	9.4	7.7
112	7.1	6.4	3.3

Aclonifen degraded at a moderate rate in soil treated at a nominal application rate of 20 mg/kg (ca. 30 kg/ha). The reported half-life values were approximately 12 weeks for [redacted] 2.2 soil and approximately 7 weeks for [redacted] 2.3 soil. The rate of degradation of aclonifen was re-evaluated using KinGUI assuming simple first order kinetics for the first approval of aclonifen (EFSA (2008) 149). The degradation rate was similar to that measured in more recent, reliable studies.

Table 7.1.2.1.1- 3: Degradation rate of aclonifen under aerobic conditions at 22 °C

Soil	M0 (mg/kg)[A]	DT_{50} (days)	DT_{90} (days)	chi^2	t-test
[redacted] 2.2 (A)	14.54	93.1	309.3	0.7	4.3E-4, >99%
[redacted] 2.2 (B)	14.60	76.4	253.7	2.7	0.0044, >99%
[redacted] 2.3	12.77	53.2	176.6	2.0	0.0015, >99%

[A] Initial residue at Day 14

Recoveries to demonstrate the extraction efficiency and verify the analytical method in both soils were reported as 91% and 93% in [redacted] 2.2 and [redacted] 2.3 soil, respectively. The limit of detection in soil matrix was reported as 0.05 mg/kg. However, compared to requirements laid down by SANCO/3029/99 rev. 4, there are a number of deviations such as method description, linearity and calibration, and precision data not reported in full and the number of determinations per fortification

level. Thus, the analytical method used in the study cannot be considered as fit for purpose. A full summary of the analytical method is provided in Document MCA-4, Chapter 4.1.2.

Additionally, no extraction and analysis of soil samples immediately after application of the test substance was conducted. The first sampling after application was on Day 14. The report is extremely brief with only a few details of the experiment, and not considered acceptable according to current requirements. Consequently, the study must be considered as supportive only.

III. CONCLUSION

The rate of degradation of aclonifen was investigated in two standard [redacted] soils, [redacted] 2.2 and 2.3 under laboratory aerobic conditions at 22 °C. The study deviates in a number of important aspects to the requirements of OECD 307 (2002) and consequently is now considered as supportive data only.

Assessment and conclusion by applicant:

This study is non-GLP and was conducted according to BBA Merkblatt 36 (1973). The study deviates in a number of important aspects to the requirements of OECD 307 (2002) in particular the analytical method is not considered fit for purpose, time zero samples were not taken and the report is too brief to be considered acceptable. The study is not considered reliable to assess the aerobic degradation of aclonifen in soil and is included as supporting data only.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.2.1.1/04
Report Author:	[redacted] A. J.
Report Year:	2006
Report Title:	Aclonifen- Kinetic evaluation of soil laboratory studies
Report No:	M-266704-01-1
Document No:	M-266704-01-1
Guideline(s) followed in study:	not specified
Deviations from current test guideline:	Current Guideline: FOCUS Degradation Kinetics (2006, 2014) Does Current Guideline: Not meet guideline recommendations.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon: December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

In the previous submission (Addendum to DAR, 2008), this modelling report was evaluated but not accepted as valid for risk assessment purposes. The report was superseded by KCA 7.1.2.1.1/05 ([redacted] & [redacted] 2008, M-300678-01-1) and hence a summary is not presented in this dossier.

Data Point:	KCA 7.1.2.1.1/05
Report Author:	[redacted]; [redacted]
Report Year:	2008
Report Title:	Kinetic modelling analysis of aclonifen from aerobic soil degradation studies, normalised to 20ºC and pF2
Report No:	VC/08/016A
Document No:	M-300678-01-1
Guideline(s) followed in study:	EU Council Directive 91/414/EEC, as amended by Commission Directive 95/36/EC of July 1995, Section 5, Point 7.1.1.
Deviations from current test guideline:	Current guideline: FOCUS Degradation Kinetics (2006, 2014) Does not meet guideline recommendation - impact: the study is superseded
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

In the previous submission (Addendum to DAR, 2008), this modelling report was evaluated and accepted as valid for risk assessment purposes. However additional studies have been conducted and the requirements of kinetic evaluations according to FOCUS kinetics have changed, thus the report is no longer considered as valid. It has been superseded by KCA 7.1.2.1.1/07 ([redacted] & [redacted], 2019, M-674934-01-1) and hence a summary is not presented in this dossier.

Data Point:	KCA 7.1.2.1.1/06
Report Author:	[redacted]
Report Year:	1988
Report Title:	HERBICIDES: ACLONIFEN Rate of degradation in two soils under aerobic conditions at 10°C
Report No:	R007107
Document No:	M-174228-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: OECD 307 (2002) Analytical method not fully validated.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Supportive only

Executive Summary

The rate of degradation of non-radiolabelled aclonifen, applied at a rate equivalent to 10 mg/kg, was investigated in two soils for up to one year. The soils used were classified as a loamy sand soil

([redacted]) and a clay loam soil ([redacted]). The soils were collected from cultivated fields which had received no pesticide applications since autumn 1985 and were sieved to 2 mm prior to use. Soil samples were incubated in the dark, at a moisture content equivalent to 50% of maximum water holding capacity (MWHC) under aerobic conditions at 10 ± 2 ºC. The purity of the non radiolabelled aclonifen was 97.3 %.

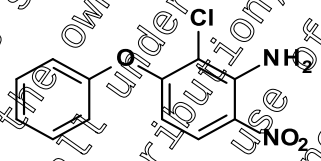
Duplicate samples of each soil were taken for analysis after 0, 1, 7, 14, 28, 42, 70, 126, 231, 315 and 364 days. Soil samples were extracted twice at ambient temperature with dichloromethane / acetone (40/60, v/v). The extracted soil was then washed with acetone. All extracts were combined and a sub-sample partitioned with dichloromethane and water. The organic layer was removed, dried by passing through anhydrous sodium sulphate and evaporated to dryness. The residue was dissolved in toluene and analysed by gas chromatography (GC). Concurrent recoveries to demonstrate the extraction efficiency and verify the analytical method from both soils were measured routinely in control samples fortified over the concentration range 0.1 to 10 mg/kg and mean recoveries of 97.1% and 96.0% were obtained for the loamy sand and clay loam soil, respectively. The limit of detection in soil matrix was determined as 0.007 mg/kg).

Aclonifen slowly degraded in [redacted] and [redacted] soils incubated under aerobic conditions at 10 ºC. Approximately 30% of the applied aclonifen remained in the soil extracts at the end of one year.

I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: Aclonifen



Chemical name (IUPAC)	2-chloro-6-nitro-3-phenoxy-aniline
CA registry number:	74070-46-5
Lot or batch number:	EA 664 T1
Chemical purity:	97.3%
Stability of test compound:	Shown to be stable under the conditions of the test
Application vehicle:	Acetone

2. Soil Two agricultural soils collected from two sites in the United Kingdom [redacted], Suffolk, UK (loamy sand), [redacted], Essex, UK (clay loam). The soils were collected from the field and sieved to 2 mm prior to use. Soils were collected from specific locations with no application of plant protection products since autumn 1985. Specific details are shown below.

Table 7.1.2.1.1- 4: Physico-chemical characteristics of the soil used in aerobic soil study

Characteristic / Code	Units	[redacted]	[redacted]
Origin	Country	Suffolk, UK	Essex, UK
Location	City or Township	Ogilvie's Farm, [redacted] [1]	[redacted] Farm, [redacted]
Particle Size Analysis			
Total Sand (0.06 - 2.0 mm)	%	87	48
Silt (0.002 - 0.06 mm)	%	4	29
Clay (<0.002 mm)	%	9	23
Textural Class	USDA	Loamy sand	Clay loam
pH		6.8	7.2
Organic Matter	%	2.31	3.09
Organic Carbon[1]	%	1.16	1.55
Cation Exchange Capacity	meq/100g	7.91	21.43
Bulk Density	g/cm³	1.48	1.43
Maximum Water Holding Capacity	%	22.34	28.51
Water Holding Capacity	% at 1/3 bar	12.36	24.58
Soil Moisture During Incubation [2]	%	11.17	14.26

[1] Same location as [redacted] on [redacted] Road, [redacted]
[2] Calculated from data in the report

B. STUDY DESIGN AND METHODS

1. In-life dates:

January 1987 – January 1988

2. Experimental design

Parameter		Description
Duration of test		354 days
Soil condition		Soil sieved to 2 mm and stored for a minimum of 14 days at 10°C
Concentration in test system		10 mg/kg
Number of replications		Duplicate
Test apparatus		Conical flasks containing 100 g dry weight equivalent of soil
Test material application	Identity of solvent	Acetone
	Volume of application solution	250 µL per 100 g soil dry weight
	Application method	Not stated but the soil was mixed thoroughly after the application.
Traps for CO_2 and organic volatiles		Not applicable
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	10 ± 2°C
	Moisture content	50% of maximum water holding capacity (MWHC)
	Lighting	Dark

Sampling

Parameter	Description

Parameter		Description
Sampling intervals	Aerobic, non-sterile	Duplicate samples of soil were taken for analysis after 0, 1, 7, 14, 28, 42, 70, 126, 231, 315 and 364 DAT
	Untreated soils for biomass	No biomass measurements were undertaken
Soil sampling procedures		Complete treated samples were removed at each sampling time and extracted as detailed below.
Collection of CO_2 and volatile organics		Not applicable

Analytical procedures

Soil samples were extracted twice at ambient temperature with dichloromethane / acetone (40/60, v/v). The extracted soil was then washed with acetone. All extracts were combined and a sub-sample partitioned with dichloromethane and water. The organic layer was removed, dried by passing through anhydrous sodium sulphate and evaporated to dryness. The residue was dissolved in toluene and analysed by gas chromatography (GC). The limit of detection in soil matrix was determined as 0.007 mg/kg.

Degradation kinetics

Aclonifen slowly degraded in soil treated at 10 mg/kg (ca. 15 kg/ha) and incubated at 10 ºC. The reported DT_{50} values were approximately 31 weeks for both soils. The rate of degradation of aclonifen was re-evaluated using KinGUI assuming simple first order kinetics for the first approval of aclonifen (EFSA (2008) 149).

II. RESULTS AND DISCUSSION

Method Validation

A full summary of the analytical method is provided in Document MCA-4, Chapter 4.1.2. Concurrent recoveries to demonstrate the extraction efficiency and verify the analytical method from both soils were measured routinely in control samples fortified over the concentration range 0.1 to 10 mg/kg and mean recoveries of 97.3% and 96.0% were obtained for the loamy sand and clay loam soil, respectively. Not all validation parameters according to SANCO 3029/99 rev. 4 are met but the analytical method can be regarded as fit for purpose with regard to this study.

The decline of aclonifen with time in each soil is presented in Table 7.1.2.1.1- 5. Aclonifen slowly degraded in soil treated at 10 mg/kg (ca. 15 kg/ha) and incubated at 10 ºC.

Table 7.1.2.1.1- 5: Decline of aclonifen (mean of duplicate values)

Time (days)	mg aclonifen/ kg soil (dry matter)	
	[redacted]	[redacted]
0	8.6883	8.5368
1	8.2805	8.3705
7	8.1978	8.3543
14	7.9850	7.4760
28	7.0965	7.1363
42	7.4670	7.3645
70	6.8105	6.6140
126	5.6393	5.8755
231	4.3655	3.8673
315	2.8965	2.9863
364	2.5183	2.6280

Aclonifen slowly degraded in soil treated at 10 mg/kg (ca. 15 kg/ha) and incubated at 10 °C. The reported DT_{50} values were approximately 31 weeks for both soils. The rate of degradation of aclonifen was re-evaluated using KinGUI assuming simple first order kinetics for the first approval of aclonifen (EFSA (2008) 149).

Table 7.1.2.1.1- 6: Degradation rate of aclonifen under aerobic conditions at 10 ºC

Soil	M_0 (mg/kg)	DT_{50} (days)	DT_{90} (days)	chi^2	t-test
[redacted]	8.36	222.6	739.5	3.4	1.6E-8, >99%
[redacted]	8.29	217.7	723.3	3.2	8.1E-9, >99%

III. CONCLUSION

Aclonifen slowly degraded in the [redacted] and [redacted] soils incubated under aerobic conditions at 10 ºC. No biomass measurements were undertaken so it is not possible to comment on the viability of the study. Approximately 30% of the applied aclonifen remained in the soil extracts at the end of one year.

Assessment and conclusion by applicant:

The study was not conducted to any stated guidance. No biomass measurements were undertaken so it is not possible to comment on the viability of the soil. The analytical method did not meet all of the parameters of SANCO 3029/99 rev. 4 but can be regarded as fit for purpose. The study is acceptable as supporting data.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.2.1.1/07
Report Author:	[redacted]; [redacted]
Report Year:	2019
Report Title:	Aclonifen: Kinetic evaluation of the degradation in soil under aerobic conditions
Report No:	VC/19/025C
Document No:	M-674934-01-1
Guideline(s) followed in study:	none
Deviations from current test guideline:	Current Guideline: FOCUS Degradation Kinetics (2006, 2014) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The aim of this report was to derive modelling and trigger endpoints for aclonifen using data collected from laboratory aerobic soil degradation studies following the FOCUS kinetics guidance (FOCUS, 2014) in 12 European soils.

The model fit as well as the statistical evaluation of the results were carried out with the in-house developed software KinGUI, version 2.1. The selection of the most appropriate kinetic model was based on a detailed statistical analysis including visual assessment, χ^2 test statistics, randomness of residuals, and t-test significance following the FOCUS guidance (2006, 2014a).

The resulting DT_{50} values (i.e., modelling and trigger endpoints) of aclonifen are given in the Tables below

Soil degradation not-normalised and normalised (to pF2 and 20°C, Q10: 2.58) DT_{50} values of aclonifen for modelling purposes (modelling endpoints)

Aclonifen	Dark aerobic conditions					
Soil type (origin)	**pH ($CaCl_2$)**	**t. °C / % MWHC**	**DT_{50} / DT_{90} (d)**	**DT_{50} (d) 20°C pF2/10 kPa***	**St. (χ^2err) (%)**	**Method of calculation**
[redacted] Farm Sandy loam[1]	n.a.	20/60	106.8/354.9	106.8	12.93	SFO
[redacted] Field Clay Loam [1]	n.a.	20/60	61.6/204.6	61.6	8.523	SFO
[redacted] Loamy Sand [1]	n.a.	20/60	82.2/273.0	82.2	5.7	SFO
Wurmwiese Sandy loam[2]	4.9	20/53.8	101.3/336.7	101.3	4.147	SFO
[redacted] Hof AXXa Sandy loam[3]	6.7	20/55	47.4/157.5	47.4	2.041	SFO
[redacted] am [redacted] Silt Loam[3]	6.1	20/55	59.1/196.2	59.1	2.731	SFO
[redacted] Hof Silt Loam[3]	5.5	20/55	95.4/317	95.4	4.145	SFO
[redacted] Clay Loam[3]	7.2	20/55	35.3/117.2	35.3	7.404	SFO
[redacted] Farm[4] Sandy loam	4.6	20/55	252.3/838.0	252.3	1.532	SFO
[redacted] Field[4] Loam	5.9	20/55	64.7/215.0	64.7	4.67	SFO
[redacted][4] Loam	7.0	20/55	64.2/213.1	62.7	2.501	SFO
[redacted][4] Loamy sand	5.3	20/55	114.0/378.6	114.0	3.33	SFO
Geometric mean				**79.1**		-

[1] [redacted] (1995) [2] [redacted] (2016) [3] [redacted] and [redacted] (2019) [4] [redacted] and [redacted] (2019)

* Normalised using a Q_{10} of 2.58 and Walker equation coefficient of 0.7

n.a. Not available

Soil degradation DT_{50} values of aclonifen to trigger additional studies (trigger endpoints)

Aclonifen	Dark aerobic conditions					
Soil type (origin)	**pH ($CaCl_2$)**	**t. °C / % MWHC**	**DT_{50} / DT_{90} (d)**	**DT_{50} (d) 20°C pF2/10 kPa***	**St. (χ^2err) (%)**	**Method of calculation**
[redacted] Farm Sandy loam[1]	n.a.	20/60	93.3/>1000	-	8.426	FOMC
[redacted] Field Clay Loam [1]	n.a.	20/60	54.6/396.3	-	7.979	FOMC
[redacted] Loamy Sand [1]	n.a.	20/60	82.2/273.0	-	5.7	SFO
Wurmwiese Sandy loam[2]	4.9	20/53.8	95.9/387.2	-	2.213	DFOP
[redacted] Hof AXXa Sandy loam[3]	6.7	20/55	47.4/157.5	-	2.041	SFO

Aclonifen	Dark aerobic conditions					
Soil type (origin)	**pH ($CaCl_2$)**	**t. °C / % MWHC**	**DT_{50} / DT_{90} (d)**	**DT_{50} (d) 20°C pF2/10 kPa***	**St. (χ^2err) (%)**	**Method of calculation**
[redacted] am [redacted] Silt Loam[3]	6.1	20/55	49.2/243.6	-	2.203	DFOP
[redacted] Hof Silt Loam[3]	5.5	20/55	85.8/331.0	-	1.812	DFOP
[redacted] Clay Loam[3]	7.2	20/55	26.4/146.5	-	2.318	DFOP
[redacted] Farm[4] Sandy loam	4.6	20/55	282.6/>1000	-	1.235	DFOP
[redacted] Field[4] Loam	5.9	20/55	55.0/515.4	-	2.934	FOMC
[redacted][4] Loam	7.0	20/55	59.5/237.4	-	0.4214	DFOP
[redacted][4] Loamy sand	5.3	20/55	121.4/580.2	-	1.104	DFOP

[1] [redacted] (1995) [2] [redacted] (2016) [3] [redacted] and [redacted] (2019) [4] [redacted] and [redacted] (2019)
n.a. Not available

I. MATERIALS AND METHODS

The experimental data generated in aerobic soil laboratory studies treated with aclonifen, were re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). The aim of this evaluation was to derive DT_{50} values for use as modelling endpoints and trigger endpoints. Modelling endpoints were normalised to a temperature of 20°C and a soil moisture content of pF 2, in compliance with the FOCUS kinetics guidance. A Q10 of 2.58 and measured reference moisture content at pF 2 were used when available in the normalisation.

The studies used in the re-evaluation are summarised below.

Study type	Number of soils	GLP	Data Point, Author &Year
Route and rate of degradation	3	Yes	CA 7.1.1.1/01, [redacted], (1995)
	1	Yes	CA 7.1.1.1/04, [redacted], (2016)
	4	Yes	CA 7.1.1.1/05, [redacted] and [redacted], (2019)
	4	Yes	CA 7.1.1.1/06, [redacted] and [redacted], (2019)

II. RESULTS AND DISCUSSION

The degradation of aclonifen was investigated in a total of 12 soils in four rate of degradation studies. The kinetic fits for aclonifen were performed using KinGUI (version 2.1).

Kinetic fits with SFO, FOMC and DFOP models were assessed for each soil. The modelling DT_{50} of aclonifen in each soil was selected from one of the three kinetic model fits, based on criteria following FOCUS (2014) guidance, and normalised to 20 °C and a moisture content of pF2. The persistence trigger DT_{90} of cymoxanil in each soil was selected from the best fit model.

For derivation of trigger endpoints, the kinetic evaluation for study CA 7.1.1.1/01, [redacted], (1995) was started by comparing SFO and FOMC fits. As FOMC fits were better for [redacted] Farm and [redacted] field, DFOP fits were also evaluated, with FOMC selected as best-fit. For [redacted], FOMC showed no improvement and SFO kinetics were selected as best-fit (Table 7.1.2.1.1- 7).

For derivation of modelling endpoints, the kinetic evaluation was started by assuming a single first-order (SFO) degradation. For residue data of all soils the SFO fit was deemed acceptable as the residuals were well distributed and χ2err test was < 15%.

Table 7.1.2.1.1- 7: Aclonifen ([redacted], 1995): kinetic and statistical results of the SFO, FOMC and DFOP curve fits (parent fit)

Kinetic model	DT_{50}	DT_{90}	VA	χ2err	k1 / α	k2 / β	tb / g	t-test k1 / k2	MS
	(d)			(%)	(1/d / -)	(1/d / -)	(d / -)	(-)	
Soil [redacted] Farm									
SFO	106.84	354.9	o	12.93	0.006488	-	-	0.00935	M
FOMC	93.33	> 1000	o	8.426	0.22910	4.76028	-	-	T
DFOP	> 1000	> 1000	-	7.984	5.007e-02	2.337e-14	4.908e-01	0.1807/0.5000	-
Soil [redacted] Field									
SFO	61.58	204.6	o	8.523	0.011256	-	-	0.000174	M
FOMC	54.60	396.3	o	7.979	1.2321	72.3053	-	-	T
DFOP	55.84	217.1	+	8.452	3.038e+00	9.981e-03	1.269e-01	2e-16/0.003103	-
Soil [redacted]									
SFO	82.19	273.0	o	5.7	8.434e-03	-	-	5.55e-05	M/T
FOMC	82.20	273.2	o	6.08	1.584e+03	1.877e+05	-	-	-
DFOP	78.78	281.4	o	6.125	485.988136	0.007942	0.065266	0.5/ 0.2338	-

MS: Model selected (T: for trigger evaluation; M: for modelling evaluation)

For derivation of trigger endpoint the kinetic evaluation for study CA 7.1.1.1/04, [redacted], (2016) was started by comparing SFO and FOMC fits, the lower χ2err test of FOMC makes consider the opportunity to evaluate DFOP fit model, which resulted to give the most appropriate fit (Table 7.1.2.1.1- 8).

For derivation of modelling endpoints, the kinetic evaluation was started by assuming a single first-order (SFO) degradation. the SFO fit was deemed acceptable as the residuals were well distributed and the error value of the χ2err test was <15%.

Table 7.1.2.1.1- 8: Aclonifen ([redacted], 2016): kinetic and statistical results of the SFO, FOMC and DFOP curve fit (parent fit)

Kinetic model	DT_{50}	DT_{90}	VA	χ^2err	k_1 / α	k_2 / β	t_b / g	t-test k_1 / k_2	MS
	(d)			(%)	(1/d / -)	(1/d / -)	(d / -)	(-)	
Soil Wurmwiese									
SFO	101.3	336.2	o	4.147	6.839e-03	-	-	5.46e-11	M
FOMC	104.30	>1000	o	3.224	0.4479	28.2325	-	-	-
DFOP	95.86	387.2	+	2.213	1.729e-01	5.524e-03	1.509e-01	5.261e-04 /3.230e-02	T

MS: Model selected (T: for trigger evaluation; M: for modelling evaluation)

For derivation of trigger endpoints, the kinetic evaluation for study CA 7.1.1.1/05, [redacted] and [redacted], (2019 was started by comparing SFO, FOMC and DFOP fits. The DFOP model gave a lower χ2err compared to the SFO model (Table 7.1.2.1.1- 9) in three soils ([redacted] am [redacted], [redacted] Hof and [redacted]) with acceptable visual fittings, then it was selected a trigger model.

For derivation of modelling endpoints, the kinetic evaluation was started by assuming a single first-order (SFO) degradation. For residues data from all the soils the SFO fit was deemed acceptable as the residuals were well distributed and the error value of the χ2err test was low.

Table 7.1.2.1.1- 9: Aclonifen ([redacted] and [redacted], 2019): kinetic and statistical results of the SFO, FOMC and DFOP curve fits (parent fit)

Kinetic model	DT_{50}	DT_{90}	VA	χ^2err	k_1 / α	k_2 / β	t_b / g	t-test k_1 / k_2	MS
	(d)			(%)	(1/d / -)	(1/d / -)	(d / -)	(-)	
Soil [redacted] Hof AXXa									
SFO	47.42	157.5	o	2.041	0.014618	-	-	0.00117	M/T
FOMC	47.42	157.5	o	2.177	7.242e+03	4.954e+05	-	-	
DFOP	47.42	157.5	o	2.351	1.462e-02	1.462e-02	3.430e-02	7.808e-03/1.475e-03	-
Soil [redacted] am [redacted]									
SFO	59.09	196.3	o	5.731	1.173e-02	-		8.168e-04	M
FOMC	45.89	595.2	+	2.219	0.7689	31.3652			-
DFOP	49.22	243.6	+	3.203	8.847e-02	8.246e-03	2.546e-01	0.004786/5.44e-07	T
Soil [redacted] Hof									
SFO	95.44	317	o	4.145	7.263e-03	-	-	4.846e-04	M
FOMC	92.93	> 1000		2.843	0.5919	41.7559	-	-	-
DFOP	85.83	331	+	1.812	6.566e-03	9.068e-01	8.785e-01	3.034e-04/4.930e-01	T
Soil [redacted]									
SFO	35.29	117.2	o	7.404	0.019641	-	-	0.001636	M
FOMC	25.94	198.4	o	2.27	1.1648	31.9042	-	-	-
DFOP	26.35	146.5	o	2.318	1.920e-01	1.302e-02	3.268e-01	0.00954/6.41e-06	T

MS: Model selected (T: for trigger evaluation; M: for modelling evaluation)

For derivation of trigger endpoints, the kinetic evaluation for study CA 7.1.1.1/06, [redacted] and [redacted], (2019) was started by comparing SFO and FOMC fits. The FOMC model resulted in a better χ2err of the parent fit compared to the SFO model (Table 7.1.2.1.1- 10) for all soils. The DFOP model then was applied to fit the residue data from these trials giving good fitting curves. DFOP kinetics were determined as best-fit for [redacted] Farm, [redacted] and [redacted]. FOMC was selected as best-for for [redacted] Field.

For derivation of modelling endpoints, the kinetic evaluation was started by assuming a single first-order (SFO) degradation. For residue data of all soils the SFO fit was deemed acceptable as the residuals were well distributed and the error value of the χ2err test was lower than 15%.

Table 7.1.2.1.1- 10: Aclonifen ([redacted] and [redacted], 2019): kinetic and statistical results of the SFO, FOMC and DFOP curve fits (parent fit)

Kinetic model	DT_{50}	DT_{90}	VA	χ^2err	k_1 / α	k_2 / β	t_b / g	t-test k_1 / k_2	MS
	(d)			(%)	(1/d / -)	(1/d / -)	(d / -)	(- / -)	
Soil [redacted] Farm									
SFO	252.3	838	o	1.532	2.748e-03	-	-	4.08e-11	M
FOMC	560.9	> 1000	-	1.187	0.30756	65.81815	-	-	-
DFOP	282.6	> 1000	o	1.235	2.222e-03	6.939e-02	9.367e-01	6.92e-05/0.153	T
Soil [redacted] Field									
SFO	64.71	215	o	4.67	1.071e-02	-	-	8.344e-04	M
FOMC	54.97	515.4	+	2.934	0.9664	52.4101	-	-	T
DFOP	56.67	264.8	+	3.214	0.061159	0.007659	0.239836	0.15864/0.00789	-
Soil [redacted]									
SFO	64.15	213.1	+	2.501	1.081e-02	-	-	2.51e-14	M
FOMC	58.26	343.6	+	1.026	1.6146	108.6517	-	-	-
DFOP	59.52	237.4	+	0.4214	0.070432	0.009021	0.148474	0.00991/2.91e-09	T
Soil [redacted]									
SFO	114	378.6	o	3.33	0.006081	-	-	1.42e-10	M
FOMC	124.3	> 1000	o	1.132	0.4594	35.3013	-	-	-
DFOP	121.4	580.2	o	1.104	4.393e-02	3.502e-03	2.309e-01	0.03705/0.00744	T

MS: Model selected (T: for trigger evaluation; M: for modelling evaluation)

The modelling and trigger endpoints are presented in Table 7.1.2.1.1- 11 and Table 7.1.2.1.1- 12 for aclonifen. The standard EFSA template can be seen in Table 7.1.2.1.1- 13 and graphical representations in Table 7.1.2.1.1- 14.

Table 7.1.2.1.1- 11: Soil degradation not-normalised and normalised (to pF2 and 20°C, Q10: 2.58) DT_{50} values of aclonifen for modelling purposes (modelling endpoints)

Study	Soil	Model fitted	DT_{50} not normalised (d)	DT_{50} normalised (d)
[redacted] (1995)	[redacted] Farm Sandy loam	SFO	106.8	106.8
[redacted] (1995)	[redacted] Field Clay Loam	SFO	61.6	61.6
[redacted] (1995)	[redacted] Loamy Sand	SFO	82.2	82.2
[redacted] (2016)	Wurmwiese Sandy loam	SFO	101.3	101.3
[redacted] and [redacted] (2019)	[redacted] Hof AXXa Sandy loam	SFO	47.4	47.4
[redacted] and [redacted] (2019)	[redacted] am [redacted] Silt Loam	SFO	59.1	59.1
[redacted] and [redacted] (2019)	[redacted] Hof Silt Loam	SFO	95.4	95.4
[redacted] and [redacted] (2019)	[redacted] Clay Loam	SFO	35.3	35.3
[redacted] and [redacted] (2019)	[redacted] Farm	SFO	252.3	252.3
[redacted] and [redacted] (2019)	[redacted] Field	SFO	64.7	64.7
[redacted] and [redacted] (2019)	[redacted]	SFO	64.2	62.7
[redacted] and [redacted] (2019)	[redacted]	SFO	114.0	114.0
Geomean				***79.1***

Table 7.1.2.1.1- 12: Soil degradation not-normalised DT_{50} values of aclonifen to trigger additional studies (trigger endpoints)

Study	Soil	Model fitted	DT_{50} not normalised (d)
[redacted] (1995)	[redacted] Farm Sandy loam	FOMC	93.3
[redacted] (1995)	[redacted] Field Clay Loam	FOMC	54.6
[redacted] (1995)	[redacted] Loamy Sand	SFO	82.2
[redacted] (2016)	Wurmwiese Sandy loam	DFOP	95.9
[redacted] and [redacted] (2019)	[redacted] Hof AXXa Sandy loam	SFO	47.4
[redacted] and [redacted] (2019)	[redacted] am [redacted] Silt Loam	DFOP	49.2
[redacted] and [redacted] (2019)	[redacted] Hof Silt Loam	DFOP	85.8
[redacted] and [redacted] (2019)	[redacted] Clay Loam	DFOP	26.4
[redacted] and [redacted] (2019)	[redacted] Farm	DFOP	282.6
[redacted] and [redacted] (2019)	[redacted] Field	FOMC	55.0
[redacted] and [redacted] (2019)	[redacted]	DFOP	59.5
[redacted] and [redacted] (2019)	[redacted]	DFOP	121.4

Table 7.1.2.1.1- 13: Standard EFSA template for kinetic fitting

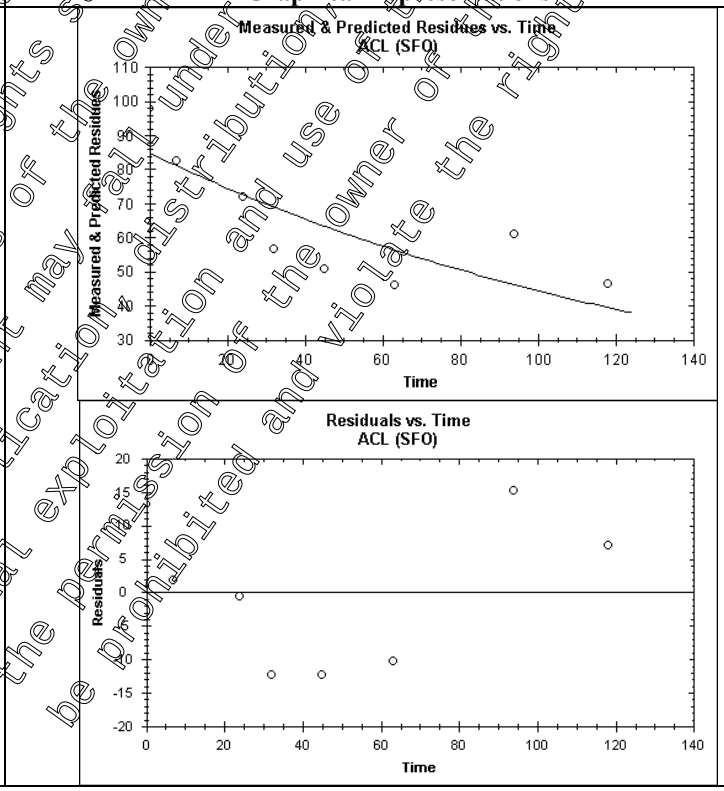
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]

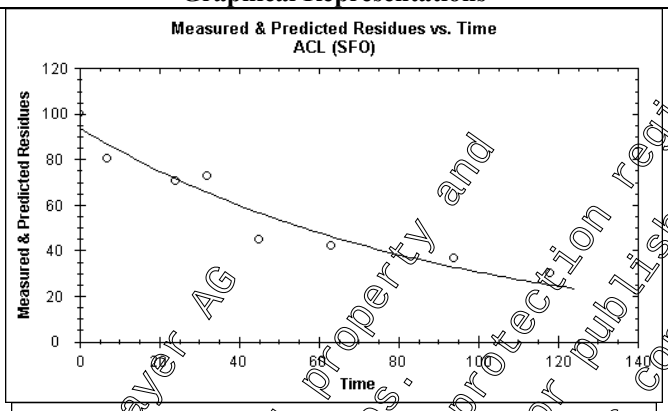
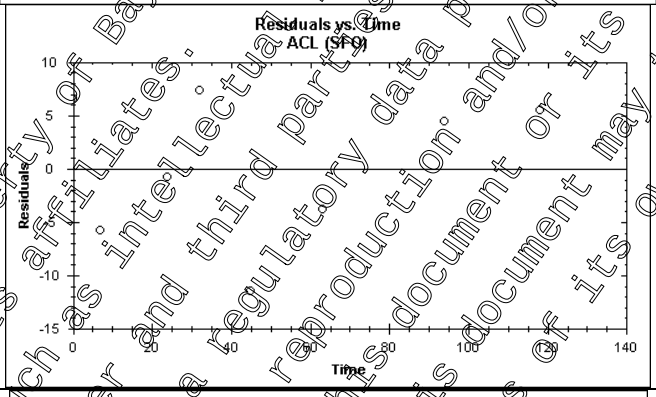
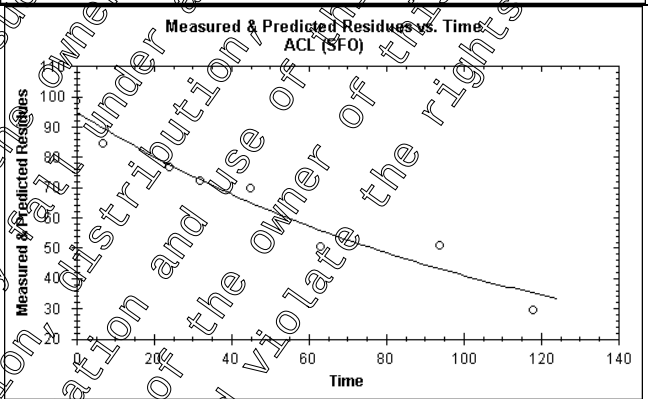
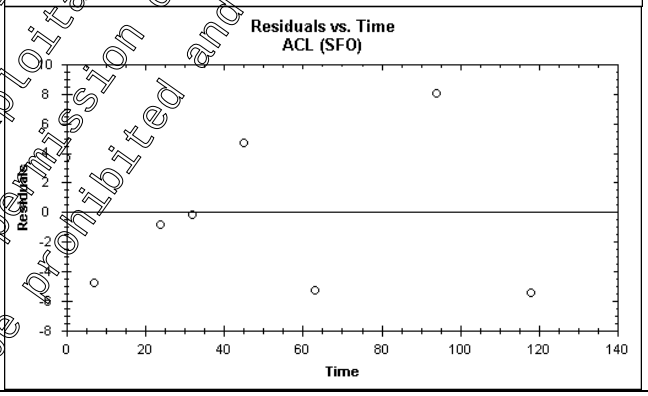
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted] Farm, [redacted], 1995	SFO	97.9	k = 0.006	12.93	0.009	69.57	99.72	106.8	354.9
	FOMC	97.9	α 0.23 β 4.76	8.42	n.r. n.r.	0.11 -8.40	0.45 17.9	93.3	>1000
	DFOP	97.9	k1 5.00e-02 k2 2.34e-14 g 4.91e-01	7.98	0.181 0.500 n.r.	n.r n.r -5.6e-02	n.r n.r 1.04	>1000	>1000
[redacted] Field, [redacted], 1995	SFO	100.2	k = 0.011	8.52	0.0002	0.008	0.014	61.6	204.6
	FOMC	100.2	α 1.23 β 72.31	7.98	0.162. 0.236	-0.98 -100.3	3.44 255.0	54.6	246.3
	DFOP	97.9	k1 3.04e+00 k2 9.98e-03 g 1.27e-01	8.45	<2e-16 0.003 0.134	3.04e+00 6.27e-03 -6.67e-02	3.04 0.01 0.32	55.8	217
[redacted], [redacted], 1995	SFO	98.4	k = 8.43e-03	5.7	5.55e-05	6.58e-03	0.01	82.2	273
	FOMC	98.4	α 1.58e+03 β 1.88e+05	6.08	0.0002 <2e-16	1.20e+03 1.88e+05	1965.1 187736	82.2	273.2
	DFOP	98.4	k1 485.488 k2 0.0079 g 0.0653	6.125	* 0.0016 0.2338	+Inf* 0.0055 -0.0943	+Inf* 0.01 0.23	78.8	281.4
Wurmwiese, [redacted], 2016	SFO	104.5	k = 6.839e-03	4.13	5.46e-11	9.26e+01	98.82	101.3	336.7
	FOMC	104.5	α 0.4479 β 28.23	3.224	n.r n.r	0.2125 0.5068	0.683 58.96	104.5	>1000
	DFOP	104.5	k1 1.73e-01 k2 5.52e-03 g 1.509e-01	2.313	0.0205 2.54e-08 -	- - 8.76e-02	- - 0.214	95.86	387.2
[redacted] Hof AXXa, [redacted] and [redacted], 2019	SFO	98.2	k = 0.0146	2.04	2.83e-09	n.r.	n.r.	47.4	157.5
	FOMC	98.2	α 1.724e+03 β 4.95e+05	2.177	n.r n.r	6.062e+03 4.95e+05	8423 495425	47.4	157.5
	DFOP	98.2	k 1.46e-002 k2 1.46e-02 g 3.43e-02	2.351	<2e-16 0.003 0.134	- - 3.42e-02	- - 0.034	47.4	157.5
[redacted] Farm, [redacted], [redacted] and [redacted], 2019	SFO	103.5	k = 1.17e-02	5.73	4.52e-10	9.04e+01	98.618	59.09	196.3
	FOMC	103.5	α 0.7689 β 31.3652	2.219	n.r n.r	0.5637 17.0302	0.974 45.70	45.89	595.2
	DFOP	103.5	k1 8.874e-02 k2 8.246e-03 g 2.546e-01	2.203	0.00479 5.44e-07 -	- - 1.504e-01	- - 0.359	49.22	243.6
Hof, [redacted] and [redacted], 2019	SFO	104.6	k = 2.263e-03	4.143	2.59e-10	9.205e+01	98.579	95.44	317
	FOMC	104.6	α 0.5919 β 41.7559	2.843	0.2871 5.8478	0.2871 5.8478	0.897 77.664	92.93	>1000
	DFOP	104.6	k1 6.566e-03 k2 9.068e-01 g 8.785e-01	1.812	2.77e-11 0.0454 -	- - 8.76e-01	- - 0.916	85.83	331
[redacted], [redacted] and [redacted], 2019	SFO	102.0	k = 0.0196	7.404	4.66e-09	-	-	35.29	117.2
	FOMC	102.0	α 1.1648 β 31.9042	3.27	- -	0.7202 12.6762	1.609 51.132	25.94	196.5
	DFOP	102.0	k1 1.020e-01 k2 1.302e-02 g 3.268e-01	2.318	0.00954 6.41e-06 -	- - 1.633e-01	- - 0.490	26.35	146.5
[redacted] Farm, [redacted], 2019	SFO	100.8	k = .748e-03	1.532	4.08e-11	-	-	252.3	838
	FOMC	100.8	α 0.30756 β 65.81815	1.187	- -	0.07565 -11.21610	0.539 142.852	560.9	>1000
	DFOP	100.8	k1 2.222e-03 k2 6.939e-02 g 9.367e-01	1.235	6.92e-05 0.153 -	- - 8.701e-01	- - 1.003	282.6	>1000

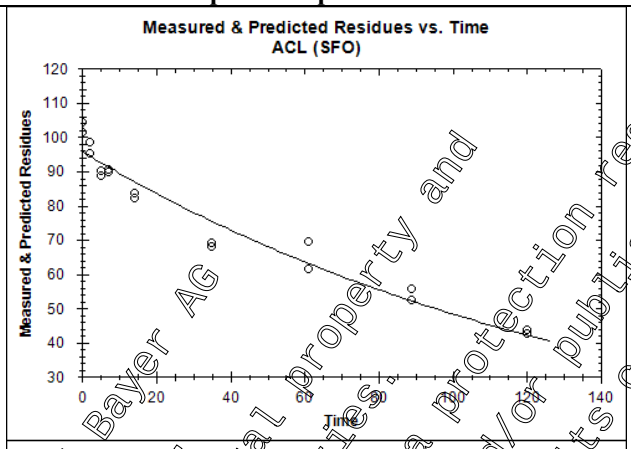
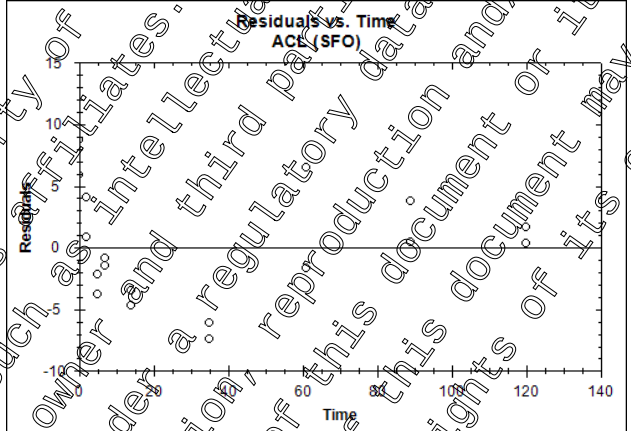
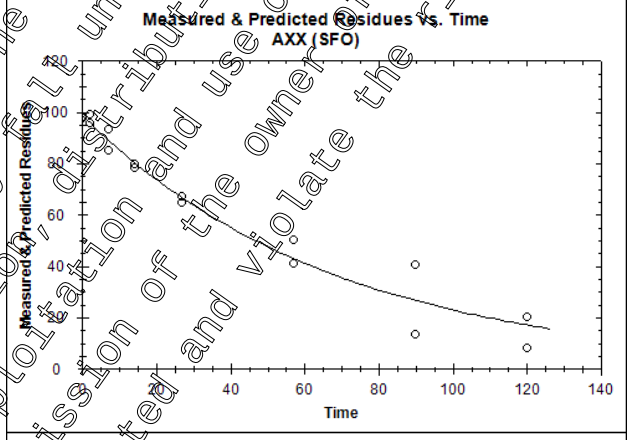
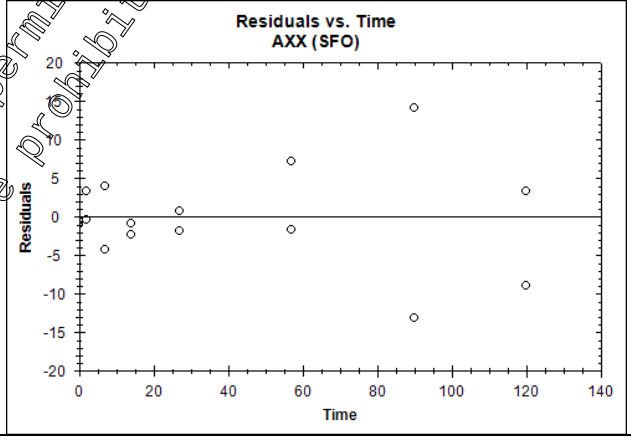
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted] Field, [redacted], 2019	SFO	101.4	k =1.071e-02	4.67	1.96e-09	-	-	64.71	215
	FOMC	101.4	α 0.9664 β 52.4101	2.934	- -	0.2028 -11.5297	1.73 116.35	54.97	515.4
	DFOP	101.4	k1 0.061159 k2 0.007659 g 0.239836	3.214	0.15864 0.00789 -	- - -0.124525	- - 0.604	5667	264.8
[redacted], [redacted], 2019	SFO	98.8	k =1.081e-02	2.501	2.51e-14	-	-	64.15	213.1
	FOMC	98.8	α 1.6146 β 108.6517	1.026	- -	0.9825 52.0262	2.247 164.677	58.26	343.6
	DFOP	98.8	k1 0.070432 k2 0.09021 g 00.148474	0.424	0.01091 2.91e-09 -	- - 0.062343	- - 0.235	59.52	2374
[redacted], [redacted], 2019	SFO	100.3	0.006081	3.33	142e-10	-	-	114	378.6
	FOMC	100.3	α 0.4594 β 35.3013	1.132		0.2853 11.4177	0.633 59.185	124.3	>1000
	DFOP	100.3	k1 4.393e-02 k2 3.502e-03 g 2.269e-01	1.104	0.03705 0.00744	- - 4.123e-02	- - 0.432	121.4	580.2

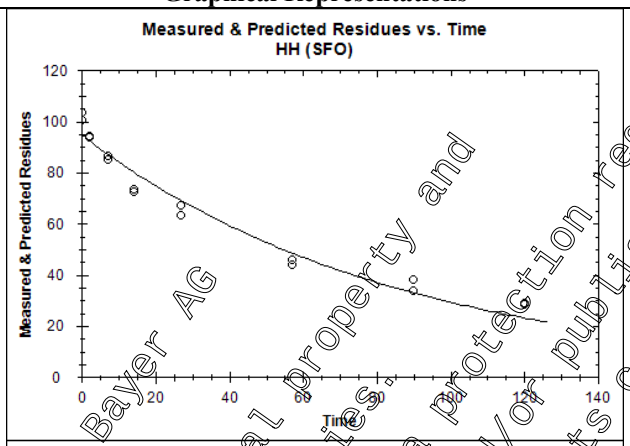
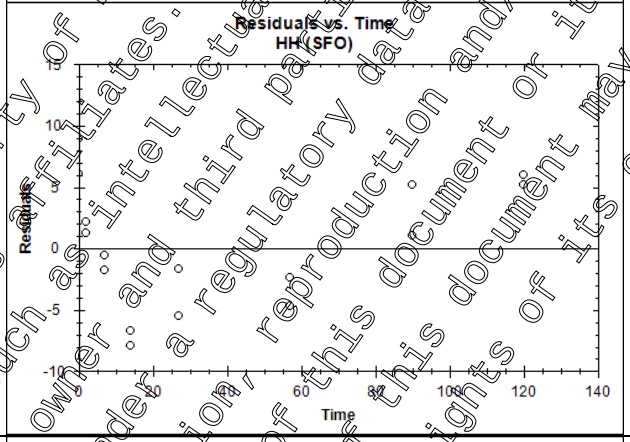
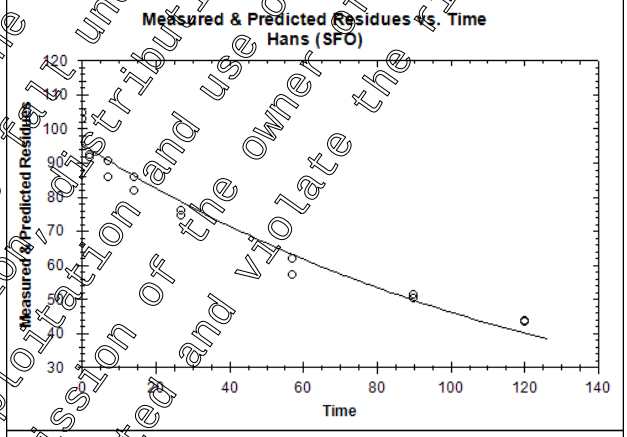
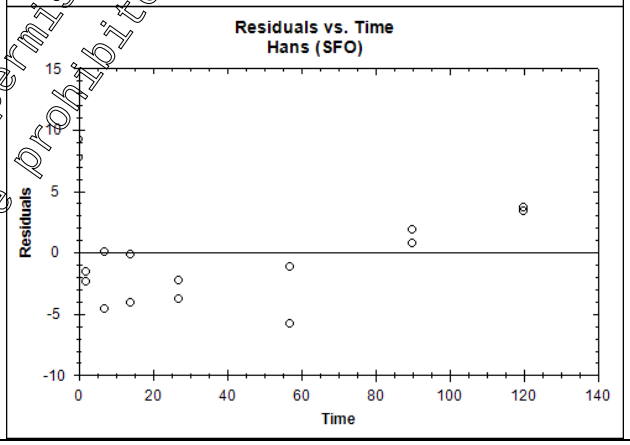
n.r. not relevant, * Hessian not invertible – NA was calculated for standard deviation, confidence interval and t-test

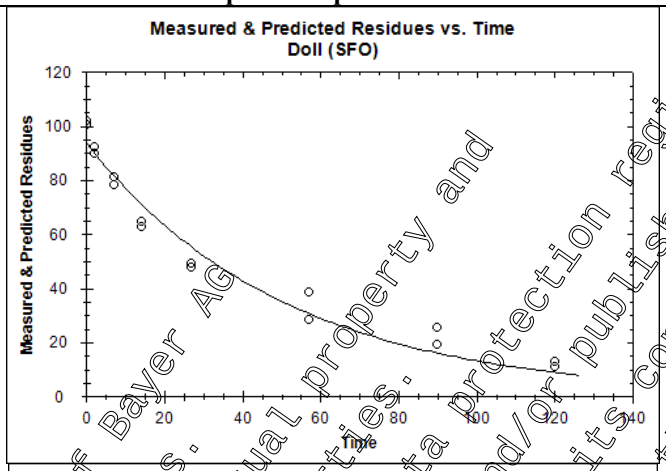
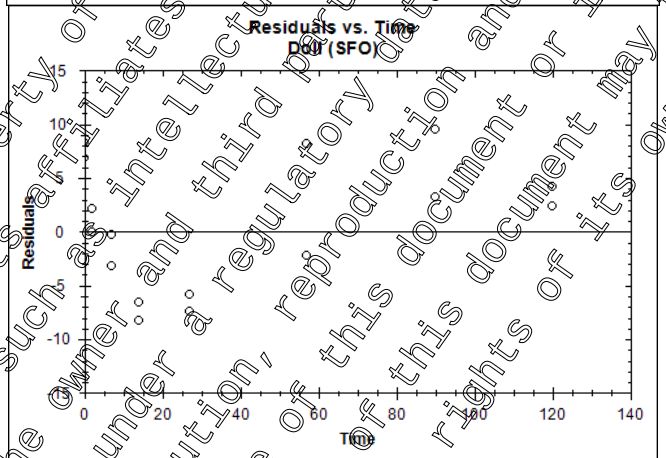
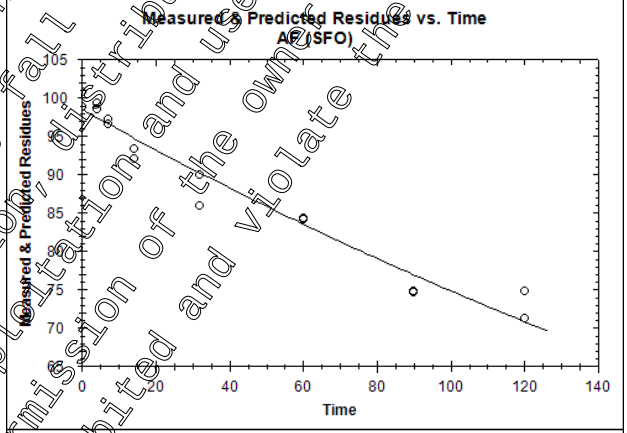
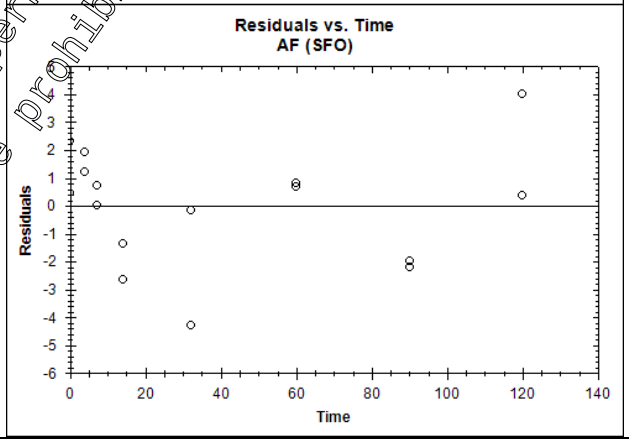
Table 7.1.2.1.1- 14: Graphical representations of best fit models

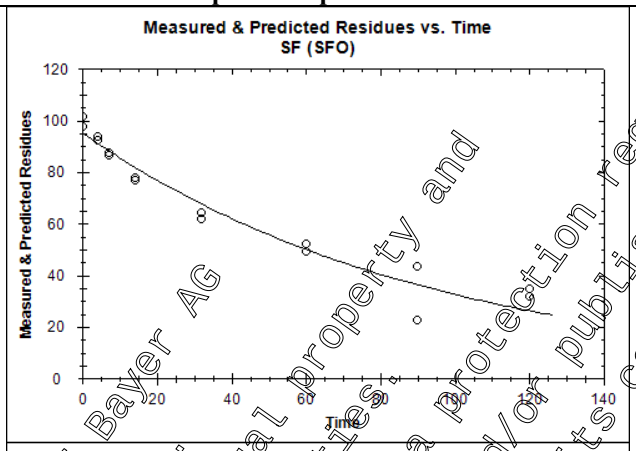
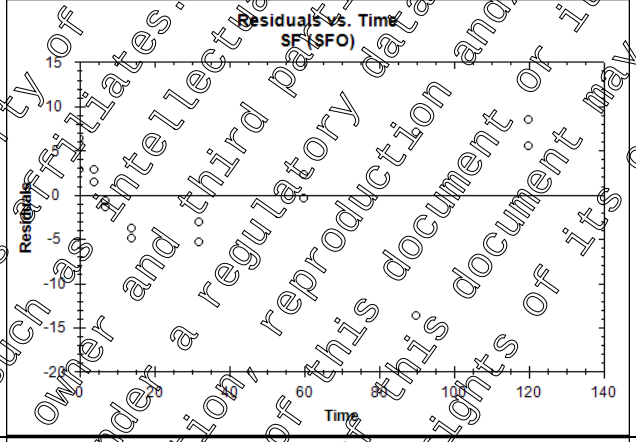
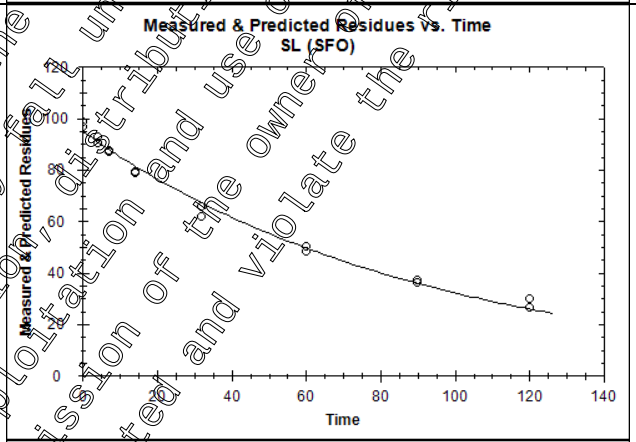
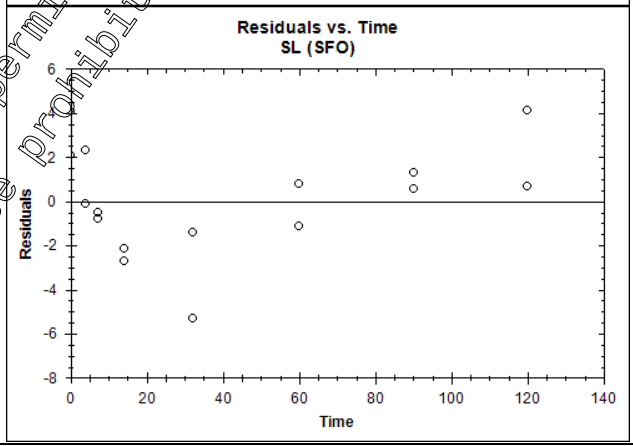
Trial / Best Fit Model	Graphical Representations
[redacted] Farm / SFO	

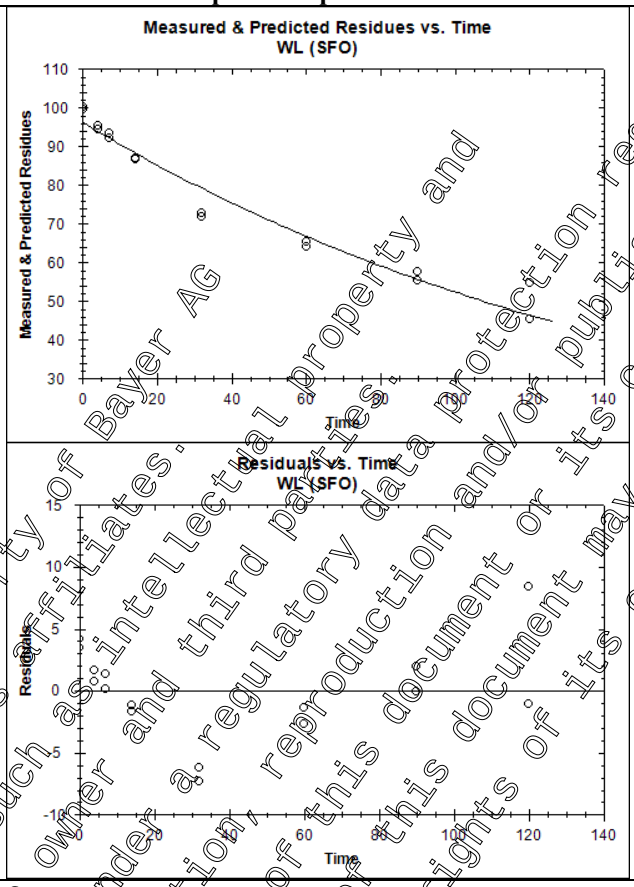
Trial / Best Fit Model	Graphical Representations
[redacted] Field / SFO	 
[redacted] SFO	 

Trial / Best Fit Model	Graphical Representations
Wurmwiese / SFO	 
[redacted] Hof AXXa / SFO	 

Trial / Best Fit Model	Graphical Representations
[redacted] am [redacted] / SFO	 
[redacted] Hof / SFO	 

Trial / Best Fit Model	Graphical Representations
[redacted], / SFO	 
[redacted] Farm, [redacted], 2019, / SFO	 

Trial / Best Fit Model	Graphical Representations
[redacted] Field, [redacted], 2019, / SFO	 
[redacted], [redacted], 2019 / SFO	 

Trial / Best Fit Model	Graphical Representations
[redacted], [redacted], 2019 / SFO	

III. CONCLUSION

Modelling and trigger endpoints for aclonifen were calculated with kinetic models according to guidance provided by FOCUS (2006, 2014) in 12 European soils.

Modelling endpoints for aclonifen ranged from 35.3 days to 252.3 days with a geomean of 79.1 days. Trigger endpoints ranged from 26.4 days to 282.6 days.

Assessment and conclusion by applicant:

The modelling report was conducted according to FOCUS Degradation Kinetics (2006, 2014) and is considered valid to assess best fit and modelling DT50 values for aclonifen in aerobic soil laboratory studies.

Assessment and conclusion by RMS:

CA 7.1.2.1.2 Aerobic degradation of metabolites, breakdown and reaction products

The major elimination route of aclonifen in soil is the formation of soil bound residues which are ultimately mineralised to carbon dioxide. Although a number of minor metabolites were observed sporadically, individual components did not exceed 5% of applied radioactivity. The levels of

metabolites were too low to determine rates of formation and decline. No significant metabolites of aclonifen are formed in the soil compartment from aerobic, anaerobic or photolytic mechanisms.

CA 7.1.2.1.3 Anaerobic degradation of the active substance

The rate of degradation of aclonifen under anaerobic conditions is summarised under point CA 7.1.1.2.

CA 7.1.2.1.4 Anaerobic degradation of metabolites, breakdown and reaction products

Cleavage of aclonifen or its minor soil metabolite M-01 could lead to the formation of phenol or hydroquinone from cleavage of M-01). A review of the published information on phenol and hydroquinone under anaerobic conditions is summarised under point 7.1.1.2.

CA 7.1.2.2 Field studies

CA 7.1.2.2.1 Soil dissipation studies

A terrestrial field dissipation study with aclonifen, formulated as BANDUR®, a suspension concentrate containing 600 g/l aclonifen, was conducted at four trial sites in Germany, Northern Europe. The field and analytical phases of the trials were reported separately in KCA 7.1.2.2.1/01 and KCA 7.1.2.2.1/02. Both reports have been summarised here to provide a single overview of the trials.

In addition, a second terrestrial field dissipation study (KCA 7.1.2.2.1/03) with aclonifen, formulated as BANDUR®, was conducted at two trial sites in Southern Europe; [redacted] in Spain and [redacted], Southern France.

These studies were evaluated during the previous EU review and are still considered as reliable to assess the rate of degradation of aclonifen under field conditions.

Three kinetic evaluation reports (KCA 7.1.2.2.1/04, KCA 7.1.2.2.1/05 and KCA 7.1.2.2.1/06) are listed. For procedural reasons the two previously submitted kinetic evaluation reports also have to be included under Point KCA 7.1.2.2.1 in the current dossier (KCA 7.1.2.2.1/04 and KCA 7.1.2.2.1/05) but these reports are fully superseded by the latest kinetic evaluation report (KCA 7.1.2.2.1/06).

Report reference	Author, Year	Comment
KCA 7.1.2.2.1/01 M-174746-01-1	[redacted] & [redacted], 1994	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.1.2.2.1/02 M-174743-01-1	[redacted], 1994	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.1.2.2.1/03 M-232115-01-1	[redacted] & [redacted], 2003	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.1.2.2.1/04 M-266704-01-1	[redacted], 2006	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Superseded by M-266725-01-1 in part and now fully superseded by M-675285-01-1.
KCA 7.1.2.2.1/05 M-266725-01-1	[redacted], 2006	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Superseded by M-675285-01-1.

KCA 7.1.2.2.1/06 M-675285-01-1	[redacted] & [redacted], 2019	New data not yet reviewed under UP.

Data Point:	KCA 7.1.2.2.1/01
Report Author:	[redacted]; [redacted]
Report Year:	1994
Report Title:	Final report - on testing the fate of residues of SAG 127 01 H (Bandur) in soil under field conditions (field report)
Report No:	R007356
Document No:	M-174746-01-1
Guideline(s) followed in study:	BBA: IV / 3-3, IV / 4-1
Deviations from current test guideline:	Current Guideline: ENV/JM/MONO(2016)6; EFSA (2014) A number of minor deviations
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Data Point:	KCA 7.1.2.2.1/02
Report Author:	[redacted]
Report Year:	1994
Report Title:	ACLONIFEN - Determination of residues in soil samples of the study-no. 92-327
Report No:	R007355
Document No:	M-174743-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: ENV/JM/MOCurrent Guideline: No(2016)6; EFSA (2014) A number of minor deviations
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

A terrestrial field dissipation study with aclonifen, formulated as BANDUR®, a suspension concentrate containing 600 g/L aclonifen, was conducted at four trial sites in Germany. The formulated material was applied once to bare soil plots, at the rate required to achieve an application of 3.3 kg/ha of aclonifen in July 1992. Samples of soil were taken at intervals up to 14 months at three sites and up to 26 months at the fourth site. Soil samples were analysed by a GC method to determine levels of aclonifen present. Results of aclonifen (expressed as mg/kg) in soil are listed in the table

below. None of the control samples exhibited any residue of aclonifen. Samples from 10-20 cm and 20-30 cm depths for [redacted] regularly exhibited detectable amounts of aclonifen. The levels represented approximately 1% of the initial residue detected in the 0-10 cm horizon and were most probably the result of problems with sampling the high organic matter soil.

Aclonifen mg/kg (dry weight)		Days after treatment								
Field Site	**Depth [cm]**	0	*ca.* 20	*ca.* 70	*ca.* 120	*ca.* 240	*ca.* 325	*ca.* 430	687	791
[redacted]	**0-10**	2.166	1.616	0.635	0.729	0.391	0.133	0.076		
	10-20	0.012	nd	nd	nd	nd	nd	nd	-	-
	20-30	<LOQ	nd	nd	nd	nd	nd	nd	-	-
[redacted]	**0-10**	1.447	1.403	1.491	0.331	0.566	0.151	0.092		
	10-20	0.032	nd	nd	<LOQ	nd	nd	nd	-	-
	20-30	nd	nd	nd	nd	nd	nd	nd	-	-
[redacted]	**0-10**	2.02	1.963	1.284	1.023	0.942	0.556	0.486	0.225	0.215
	10-20	0.028	0.915	nd	0.022	0.021	0.034	nd	0.023	nd
	20-30	0.022	nd	nd	0.026	nd	0.043	nd	nd	nd
[redacted]	**0-10**	2.058	0.823	0.417	0.251	0.272	0.101	0.035		-
	10-20	nd	nd	nd	0.017	nd	nd	nd	-	-
	20-30	0.058	0.017	nd	nd	nd	nd	nd	-	-

nd = not detected, < LOQ = 0.01 mg/kg , - = not sampled

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit not normalised DT_{50} values of 46.4, 117.6, 144.5 and 13.8 days and DT_{90} values of 310.1, 390.6, 757.7 and 210.9 days for [redacted], [redacted], [redacted] and [redacted] sites, respectively.

I. MATERIALS AND METHODS

1. Test Substance

Formulation Name:	BANDUR
Test Material	Aclonifen
Formulation Type:	SC
Actual content of Active Ingredient	600 g/L
Lot or batch number	9001 V 10

2. Trial Locations & Soils

The soil dissipation of aclonifen was studied at four different locations in Germany. The characteristics of the trial locations and soils types are summarised below.

Locations:	D-[redacted], (Germany) D-[redacted], (Germany) D-[redacted] (Germany) D-[redacted] (Germany)
Pre-treatment history	Not stated
Crop history	[redacted]: 1989 (bare), 1990 (bare), 1991 (bare) [redacted]: 1989 (bare), 1990 (bare), 1991 (bare) [redacted]: 1989 (bare), 1990 (bare), 1991 (bare)

	[redacted] : 1989 (bare), 1990 (winter rye), 1991 (winter wheat)
Pesticides used in preceding 3 years	Not stated
Distance of weather station from test site, for rainfall and air temperature	SKG-9268-01 [redacted] – [redacted], 7 km from trial SKG-9268-02 [redacted], [redacted], [redacted], 35, 10, & 28 km from trial SKG-9268-03 [redacted] (Airport), [redacted], 50 km from trial SKG-9268-04 [redacted] / [redacted], 9 km from trial

Table 7.1.2.2.1- 1: Physico-chemical characteristics of the soil sites

Soil Characteristic		Units	[redacted]	[redacted]	[redacted]	[redacted]
Location			Germany Nordrhein Westfalen	Germany Hesse	Germany Lower Saxony	Germany Rhineland-Palatinate
Post Code			D-[redacted]	D-[redacted]	D-[redacted]	D-[redacted]
Trial Number			SKG-9268-01	SKG-9268-02	SKG-9268-03	SKG-9268-04
Sampling depth		cm	30	25	30	30
pH	1992	KCl	5.9	6.4	6.3	7.3
	1993	KCl	5.5	5.4	5.3	not reported
	1994	KCl	5.6	5.1	5.5	7.4
Organic carbon content	1992	%	2.0	1.2	3.2	0.9
	1993	%	2.3	1.4	4.0	not reported
	1993	%	1.8	1.5	3.5	0.9
Textural class	1992	DIN	silty loam (uL)	loam (L)	sandy loam (sL)	sandy loam (sL)
	1993	DIN	silty loam (uL)	silty loam (uL)	sandy loam (sL)	not reported
	1994	DIN	silty loam (uL)	silty loam (uL)	loamy sand (lS)	sandy loam (sL)
Preceding crop	1989		Fallow	Fallow	Fallow	Winter Wheat
	1990		Fallow	Fallow	Fallow	Winter Rye
	1991		Fallow	Fallow	Fallow	Fallow

Table 7.1.2.2.1- 2: Summary of weather data

Location	Period	Temperature (°C) Mean	Precipitation (mm) actual
[redacted] SKG-9268-01 [redacted], 7 km from trial site	July 1992	19.6	61.3
	Aug 1992	18.9	147.2
	Sep 1992	15.0	29.7
	Oct 1992	8.1	61.7
	Nov 1992	8.1	100.9
	Dec 1992	4.1	57.0
	Jan 1993	5.4	68.8
	Feb 1993	2.1	24.9
	March 1993	6.4	4.8
	April 1993	12.0	53.0

Location	Period	Temperature (°C) Mean	Precipitation (mm) actual
	May 1993	15.4	82.6
	June 1993	17.4	39.2
	July 1993	17.7	124.6
	Aug 1993	16.2	35.3
	Sep 1993	13.6	164.0
[redacted] SKG-9268-02 [redacted] 35 km from trial site	July 1992	18.8	120.0 [a]
	Aug 1992	18.7	80.0 [a]
	Sep 1992	13.6	26.0 [b]
	Oct 1992	6.6	49.0 [b]
	Nov 1992	5.7	72.0 [b]
	Dec 1992	1.6	59.0 [b]
	Jan 1993	2.2	114.0 [b]
	Feb 1993	4.4	22.0 [b]
	March 1993	4.6	18.0 [b]
	April 1993	11.6	28.0 [b]
	May 1993	15.1	68.0 [b]
	June 1993	16.4	67.0 [b]
	July 1993	16.7	89.0 [b]
	Aug 1993	15.9	33.0 [b]
	Sep 1993	12.3	58.0 [b]
[redacted] SKG-9268-03 [redacted] 50 km from trial site	July 1992	19.0	48
	Aug 1992	17.9	97
	Sep 1992	13.9	62
	Oct 1992	6.8	70
	Nov 1992	6.5	113
	Dec 1992	2.9	53
	Jan 1993	3.1	86
	Feb 1993	1.2	30
	March 1993	4.4	19
	April 1993	11.1	45
	May 1993	14.7	46
	June 1993	15.4	48
	July 1993	15.7	163
	Aug 1993	15.0	83
	Sep 1993	12.3	148
[redacted] SKG-9268-04 [redacted] 9 km from trial site	July 1992	20.6	50.7
	Aug 1992	21.5	36.7
	Sep 1992	15.3	22.7
	Oct 1992	8.1	62.5
	Nov 1992	7.3	70.4
	Dec 1992	2.3	39.6
	Jan 1993	4.8	27.2
	Feb 1993	0.5	7.7
	March 1993	6.3	3.8

Location	Period	Temperature (°C) Mean	Precipitation (mm) actual
	April 1993	12.7	24.5
	May 1993	16.4	57.4
	June 1993	18.5	23.2
	July 1993	19.1	23.7
	Aug 1993	19.4	8.9
	Sep 1993	14.7	91.8

[a] [redacted] weather station 10 km from trial site
[b] [redacted] weather station 28 km from trial site

B. STUDY DESIGN AND METHODS

1. Experimental dates:

Field Phase: July 1992 to 9 September 1994

2. Experimental design

At each trial site two plots were set up, each measuring 50 m² in total. One plot was treated with the test substance. The second plot was left untreated to provide control samples.

Rainfall and air temperature were obtained from the regional official weather stations throughout the trial period ([redacted], 7 km distance from [redacted] site, [redacted] and [redacted], 10 km and 28 km from [redacted] site, [redacted], 50 km from [redacted] site and [redacted] / [redacted], 9 km from [redacted] site). In addition air temperature, soil temperature, wind speed, relative air humidity, degree of cloudiness, soil structure and soil moisture were measured at each sampling occasion at the sites.

Soil cores were taken to a depth of 30 cm during the trials. At each sampling date 20 samples from each plot were taken. All samples were divided into 0-10 cm, 10-20 cm and 20-30 cm. Field samples were frozen immediately after sampling. The soil samples were dispatched to the analytical laboratory in Germany and stored at -18 °C until required for analysis.

Experimental design, plot set up and application details

Details	[redacted]	[redacted]	[redacted]	[redacted]
Trial Number	SKG-9268-01	SKG-9268-02	SKG-9268-03	SKG-9268-04
Duration of study	426 days	432 days	791 days	426 days
Uncropped (bare) or cropped	Bare	Bare	Bare	Bare
Controls used	Yes	Yes	Yes	Yes
Number of plots	1 treated 1 untreated control	1 treated 1untreated control	1 treated 1 untreated control	1 treated 1 untreated control
Size per treated plot	50 m²	50 m²	50 m²	50 m²
Size per untreated control plot:	50 m²	50 m²	50 m²	50 m²
Distance between each plot	8 m	8 m	8 m	8 m
Margin divide	8 m	8 m	8 m	8 m
Nominal application rate used (g a.s./ha)	3300 g a.s./ha	3300 g a.s./ha	3300 g a.s./ha	3300 g a.s./ha

Details	[redacted]	[redacted]	[redacted]	[redacted]
Trial Number	SKG-9268-01	SKG-9268-02	SKG-9268-03	SKG-9268-04
Actual application rate and target (%)	3018 g a.s./ha (109.3%)	3366 g a.s./ha (98%)	3186 g a.s./ha (103.6%)	3300 g a.s./ha (100%)
Application date	9th July 1992	7th July 1992	7th July 1992	29th July 1992
Application method	Double tank knapsack sprayer	Double tank knapsack sprayer	Double tank knapsack sprayer	Double tank knapsack sprayer
Type of spray equipment	AGR-SP-01-0788 knapsack sprayer with TEEjet XR 8002 VS nozzles, 5 Spray nozzles, 2.5 m swath width.			
Volume of spray solution applied	5.03 L/ha	5.61 L/ha	5.31 L/ha	5.5 L/ha
Identification and volume of carrier used	Water 366 L/ha	Water 408 L/ha	Water 386 L/ha	Water 400 L/ha
Pan evaporation data available?	No	No	No	No
Meteorological conditions during application				
Cloud cover (%)	15	10	5	7
Air temperature (°C)	23	24	23	26
Atmospheric moisture (%)	61	55	47	55
Wind speed (m/sec)	0	1	1	0
Rainfall (mm)	-	-	-	-
Irrigation (mm)	No	No	No	No
Verification of application available	No	No	No	No

Soil sampling details

Details	[redacted]	[redacted]	[redacted]	[redacted]
Trial Number	SKG-9268-01	SKG-9268-02	SKG-9268-03	SKG-9268-04
Sampling intervals (days)	0, 7, 21, 42, 70, 96, 124, 243, 328 and 426	0, 7, 23, 43, 67, 99, 127, 244, 339 and 432	0, 7, 22, 43, 67, 97, 125, 247, 329, 427 and 687	0, 7, 20, 40, 73, 99, 125, 231, 307 and 426
Method of soil collection	By soil core	By soil core	By soil core	By soil core
Sampling depth	30 cm depth	30 cm depth	30 cm depth	30 cm depth
Number of cores collected per plot	20 per timepoint	20 per timepoint	20 per timepoint	20 per timepoint
Depth of segments	0-10 cm 10-20 cm 20-30 cm	0-10 cm 10-20 cm 20-30 cm	0-10 cm 10-20 cm 20-30 cm	0-10 cm 10-20 cm 20-30 cm
Storage conditions	Frozen -20°C	Frozen -20°C	Frozen -20°C	Frozen -20°C
Maximum storage length	152 days	154 days	154 days	132 days

Analytical procedures

The analytical method used to analyse the soil samples is described in detail in the study report. The procedure involved initial extraction of the soil with acetone in the presence of hyflosupercel. The acetone extract was evaporated to dryness and the residue re-dissolved in hexane. The hexane extract was cleaned-up using a Florisil column and the aclonifen residues eluted with toluene : hexane (8:2). The solvent was evaporated and the residue re-dissolved in toluene. Final analysis was by gas chromatography with electron capture detection (GC/ECD). The limit of quantification was 0.010 mg/kg.

Degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.2.1/06. A brief summary of the approach for trigger endpoints is provided below.

The non-normalised data was best fit by the First Order Multiple Compartment (FOMC) model at the [redacted] site with a $\chi 2$ error of 13%, the Simple First Order (SFO) model at the [redacted] site with a $\chi 2$ error of 24% and the Double First Order in Parallel (DFOP) model at the [redacted] and [redacted] sites with $\chi 2$ errors of 9 and 7%.

II. RESULTS AND DISCUSSION

A. Application Verification

Application was targeted at a rate of 3300 g a.s./ha. The actual application rate ranged from 3018 to 3366 g a.s./ha (98-109.3% of the intended application rate, calculated from the sprayer output).

Method Validation

A full summary of the analytical method is provided in Document MCA-4, Chapter 4.1.2. Although not all validation parameters according to SANCO 3029/99 rev. 4 are met, the analytical method can be regarded as fit for purpose with regard to this study. The recoveries were performed in a concentration range, which is appropriate for the study and showed good results with an average recovery over all determinations of 87 % with a relative standard deviation (RSD) of 12 %.

B. Findings:

Residue Decline

The results for aclonifen are presented below as soil residue concentrations (on a mg/kg dry weight basis) for each of the treated plots.

[redacted] trial: The average initial concentration of aclonifen in the soil samples taken immediately after application was 2.166 mg/kg. This corresponds to an apparent application rate of 3.2 kg/ha which is in very good agreement with the nominal application rate of 3.3 kg/ha (assuming a soil bulk density of 1.5 g/cm³). The dissipation of aclonifen was moderately rapid with residue levels below 5% of the initial concentration by 14 months. Residues of aclonifen were detected only in the 0-10 cm soil horizon throughout the trial except for Day 0 sampling, where residues were also detected in the lower horizons, which was concluded to be an artefact of sampling.

[redacted] trial: The average initial concentration of aclonifen in the soil samples taken immediately after application was 1.447 mg/kg. This corresponds to an apparent application rate of 2.2 kg/ha which is lower than the nominal application rate of 3.3 kg/ha (assuming a soil bulk density of 1.5 g/cm³). The dissipation of aclonifen was moderately rapid with residue levels below 10% of the initial concentration by 14 months. Residues of aclonifen were detected only in the 0-10 cm soil horizon throughout the trial except for Day 0 sampling, where residues were also detected in the lower horizons, which was concluded to be an artefact of sampling.

[redacted] trial: The average initial concentration of aclonifen in the soil samples taken immediately after application was 2.02 mg/kg. This corresponds to an apparent application rate of 3.0 kg/ha which is in good agreement with the nominal application rate of 3.3 kg/ha (assuming a soil bulk density of 1.5 g/cm^3). The dissipation of aclonifen was slower at the [redacted] site although residue levels had declined to below 10% of the initial concentration by 26 months. Residues of aclonifen were detected mainly in the 0-10 cm soil horizon throughout the trial. Low residues were detected in the 10-20 cm horizon and occasionally in the 20-30 cm horizon from day 0 onwards. They represented approximately 1% of the initial residue detected in the 0-10 cm horizon and were most probably the result of problems with sampling the high organic matter soil.

[redacted] trial: The average initial concentration of aclonifen in the soil samples taken immediately after application was 2.058 mg/kg. This corresponds to an apparent application rate of 3.1 kg/ha which is in good agreement with the nominal application rate of 3.3 kg/ha (assuming a soil bulk density of 1.5 g/cm^3). The dissipation of aclonifen was rapid with residue levels below 5% of the initial concentration by 10 months. Residues of aclonifen were detected only in the 0-10 cm soil horizon throughout the trial except for occasional residues detected in the lower horizons which were concluded to be an artefact of sampling.

Table 7.1.2.2.1- 3: Residues of aclonifen (expressed as mg/kg) in soil after application at a nominal application rate of 3.3 kg as/ha

Depth [cm]	DAT	-1	0	21	70	124	243	328	426		
0 - 10	[redacted]	nd	2.166	1.616	0.635	0.729	0.391	0.133	0.076		
10 - 20		nd	0.012	nd	nd	nd	nd	nd	nd		
20 - 30		nd	<LOQ	nd	nd	nd	nd	nd	nd		
	DAT	-1	0	23	67	127	244	339	432		
0 – 10	[redacted]	nd	1.447	1.403	1.491	0.331	0.566	0.151	0.092		
10 - 20		nd	0.032	nd	nd	<LOQ	nd	nd	nd		
20 - 30		nd	nd	nd	nd	nd	nd	nd	nd		
	DAT	-1	0	22	67	125	247	329	427	687	791
0 – 10	[redacted]	nd	2.02	1.963	1.284	1.023	0.942	0.556	0.486	0.225	0.115
10 – 20		nd	0.028	0.015	nd	0.022	0.021	0.034	nd	0.023	nd
20 - 30		nd	0.022	nd	nd	0.026	nd	0.043	nd	nd	nd
	DAT	-1	0	20	73	125	231	307	426		
0 – 10	[redacted]	nd	2.058	0.823	0.417	0.251	0.272	0.101	0.037		
10 - 20		nd	nd	nd	nd	0.017	nd	nd	nd		
20 - 30		nd	0.058	0.017	nd	nd	nd	nd	nd		

nd = not detected, LOQ = 0.010 mg/kg
Note: residues detected 10-20cm or 20-30cm represents e.g. ~1 to 3 % of the residues in 0-10cm layer

C. Degradation Rate:

Aclonifen degraded at a moderate rate in soil residue trials conducted in Germany. The reported DT_{50} values were derived using Timme, Frehse and Laska model and ranged from 16 to 208 days. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.2.1/06. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.2.1- 4.

Table 7.1.2.2.1- 4: Field dissipation $DisT_{50}$ of aclonifen for trigger purpose, not normalised

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted]	FOMC	0.7	α 1.35600 β 69.46980	13.1	n.r. n.r.	-0.14633 -53.88370	2.858 192.823	46.35	310.9
[redacted]	SFO	0.5	k = 0.005895	24.36	0.013583	0.391697	0.674	117.6	390.6
[redacted]	DFOP	0.7	k1 0.0221731 k2 0.0025857 g 0.2905999	8.931	0.18782 0.00914 -	n.r n.r -0.0611277	n.r n.r. 0.642	144.5	737.7
[redacted]	DFOP	0.7	k1 0.079327 k2 0.004775 g 0.726187	6.515	0.007751 0.021672 -	- - 0.609800	- - 0.843	13.77	210.9

n.r. not relevant

III. CONCLUSION

Following a single application of aclonifen at a nominal application rate of 3.3 kg/ha to bare soil in summer 1992, the decline of aclonifen was followed for up to 791 days after application at four sites in Germany. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit not normalised DT_{50} values of 46.4, 117.6, 144.5 and 13.8 days at the [redacted], [redacted], [redacted], [redacted], [redacted] and [redacted] sites, respectively.

Assessment and conclusion by applicant:

The study was conducted in accordance with BBA guidance (1986, 1990). The study is considered valid to assess the dissipation of aclonifen under field conditions in soil.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.2.2.1/03
Report Author:	[redacted]
Report Year:	2003
Report Title:	Aclonifen: Field soil dissipation study in Southern Europe
Report No:	C032811
Document No:	M-232115-01-1
Guideline(s) followed in study:	-
Deviations from current test guideline:	Current Guideline: ENV/JM/MOCurrent Guideline: No(2016)6; EFSA (2014) A number of minor deviations
Previous evaluation:	Yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

A terrestrial field dissipation study with aclonifen, formulated as EXP04209E (BANDUR®), a suspension concentrate containing 600 g/L aclonifen, was conducted at two trial sites in Southern Europe, [redacted] in Spain and [redacted], Southern France. The formulated material was applied once to bare soil which had been sown with sunflowers, prior to crop emergence. The rate applied was equivalent to 2.7 kg/ha of aclonifen and was applied on the 26 April 2001 in the Spanish trial and on the 16 May 2001 in the French trial. Samples of soil were taken at intervals up to 18 months and analysed by a GC method to determine levels of aclonifen present in the samples. Results of aclonifen (expressed as mg/kg) in soil are listed in the tables below.

Trial 1: [redacted], [redacted], Spain

Depth [cm]	DAT										
	-1	0	7	15	29	62	124	183	278	369	533
0 - 10	<LOQ	1.008	1.942	1.425	1.122	0.509	0.453	0.361	0.318	0.214	0.103
10 - 20	<LOQ	ns	0.046	0.046	0.029	0.019	0.014	0.03	0.012	0.023	<LOQ
20 - 30	<LOQ	ns	0.019	<LOQ	<LOQ	<LOQ	0.011	<LOQ	<LOQ	<LOQ	<LOQ
30 - 60	<LOQ	ns	ns	ns	ns	ns	ns	ns	ns	<LOQ	<LOQ

ns= not sampled LOQ = 0.010 mg/kg

Trial 2: [redacted], [redacted] Alps, France

Depth [cm]	DAT							
	-1	0	7	14	29	62	128	184
0 - 10	<LOQ	1.76	1.328	1.293	1.222	0.213	0.063	0.015
10 - 20	<LOQ	ns	0.013	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
20 - 30	<LOQ	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
30 - 60	<LOQ	ns	ns	ns	ns	ns	ns	ns

ns= not sampled LOQ = 0.010 mg/kg

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit not normalised DT_{50} values of 21.7 and 31.8 days and DT_{90} values of 332.3 and 105.5 days for the [redacted] and [redacted] sites respectively.

I. MATERIALS AND METHODS

1. Test Substance

Formulation Name:	BANDUR®
Test Material	Aclonifen
Formulation Type:	Suspension concentrate
Actual content of Active Ingredient	600 g/L
Lot or batch number:	AE F068300

2. Trial Locations & Soils

The soil dissipation of aclonifen was studied at two different locations, one in Spain, one in France. The characteristics of the trial locations and soils types are summarised below.

Locations:	Trial 1 [redacted], [redacted], North West Spain Trial 2 [redacted], [redacted] Alps, Southern France
Pre-treatment history	Trial 1 1998 none, 1999 none, 2000 none

	Trial 2 1998 none, 1999 atrazine & rimsulfuron, 2000 atrazine
Crop history	Trial 1 1998 sunflower, 1999 hemp, 2000 winter wheat Trial 2 1998 spring barley, 1999 maize, 2000 maize
Chemicals applied to the plots during the study	Trial 2 12 May 2001 flurochloridone (500 g a.i. /ha)
Pesticides used in preceding 3 years	Trial 2 1999 atrazine (1000 g a.i./ha) & rimsulfuron (15 g a.i. /ha), 2000 atrazine (1000 g a.i./ha)
Distance of weather station from test site	Trial 1 [redacted] 32 km from trial site Trial 2 [redacted] 8 km from trial site

Table 7.1.2.2.1- 5: Physico-chemical characteristics of the soil sites

Soil Characteristics	Units	Trial 1		Trial 2	
Location		Spain [redacted] North West Spain		France [redacted] Southern France	
Sampling depth	cm	0-30 cm	30-60 cm	0-30 cm	30-60 cm
Particle Size Analysis					
Total Sand (0.063 - 2.0 mm)	%	36.25	29.38	44.87	42.04
Silt (0.002 - 0.063 mm)	%	36.98	41.04	43.36	45.56
Clay (<0.002 mm)	%	26.78	29.68	11.77	12.41
Textural Class	ADAS	Clay Loam	Clay Loam	Sandy silt loam	Sandy silt loam
pH	Water (1:5)	8.5	8.7	8.7	8.8
	1M KCl (1:5)	7.8	8.0	8.2	8.2
	0.01 M $CaCl_2$	7.6	7.8	7.6	7.7
Cation Exchange Capacity	mEq/100g	18.2	15.4	8.2	9.1
Organic Carbon Content	%	1.3	0.7	1.1	0.7
Maximum Water Holding Capacity	%	45.1		41.8	-
Soil Microbial Biomass Initial	µg microbial C / g soil	439.9	-	306.7	-
Final		212.7	-	100.4	-
Preceding crop 1998		Sunflower		Spring Barley	
1999		Hemp		Maize	
2000		Winter Wheat		Maize	

Table 7.1.2.2.1- 6: Summary of weather data

Location	Period	Precipitation (mm) actual	Irrigation (mm) actual
[redacted], Spain Trial 1 [redacted] 32 km from trial site	Apr-01	73.4	0.0
	May-01	32.63	16.0
	Jun-01	8.16	0.0
	Jul-01	32.44	12.0
	Aug-01	7.73	0.0
	Sep-01	22.03	20.0
	Oct-01	26.93	0.0
	Nov-01	48.76	0.0
	Dec-01	40.41	0.0
	Jan-02	8.89	0.0
	Feb-02	9.38	0.0
	Mar-02	26.72	30.0
	Apr-02	64.66	20.0
	May-02	49.15	29.0
	Jun-02	42.23	0.0
	Jul-02	27.94	12.0
	Aug-02	37.12	20.0
	Sep-02	54.12	0.0
	Oct-02	51.39	0.0
	Nov-02	27.92	0.0
	Dec-02	28.11	0.0
[redacted], France [redacted] 8 km from trial site	May-01	85.2	0.0
	Jun-01	48.2	29.0
	Jul-01	49.6	15.0
	Aug-01	18.0	0.0
	Sep-01	105.2	0.0
	Oct-01	162.8	0.0
	Nov-01	12.4	0.0
	Dec-01	3.4	0.0
	Jan-02	44.8	0.0
	Feb-02	56.4	0.0
	Mar-02	50.4	0.0
	Apr-02	28.0	0.0
	May-02	117.4	0.0

B. STUDY DESIGN AND METHODS

1. Experimental dates:

Field Phase: 22 February 2001 to 21 February 2003

2. Experimental design

At each trial site four experimental plots SP1, SP2, SP3 and SP4 were treated with the test substance. A fifth plot was left untreated to provide control samples. The total treated area measured 1020 m² in the Spanish trial (Trial 1) and 1110 m² in the French trial (Trial 2).

Weather data including rainfall, air temperature, soil temperature, wind speed and direction, relative air humidity and evapo-transpiration were obtained from the regional official weather stations throughout the trial period ([redacted] / [redacted], 32 km distance from Spanish trial (Trial 1) and Montélimar Ancône, 8 km distance from French trial (Trial 2). Rainfall was supplemented with additional irrigation to maintain the crops, in accordance with local practices.

Soil cores were taken to a depth of 10 cm at Day 0 in both trials. In the French trial, soil cores were taken to a depth of 30 cm during the remainder of the trial (6 months). In the Spanish trial, soil cores were taken to a depth of 30 cm at time-points up to nine months and to a depth of 60 cm during the remainder of the trial (18 months). Samples were divided into depths of 0-10 cm, 10-20 cm, 20-30 cm and 30-60 cm. Field samples were frozen within six to twelve hours after sampling and shipped frozen to Inveresk Research Analytical Laboratory, Scotland. The samples were then stored at -20 ºC until required for analysis.

Experimental design, plot set up and application details

Details	[redacted], Spain	[redacted], France
Trial Number	**Trial 1**	**Trial 2**
Duration of study	533 days	184 days
Uncropped (bare) or cropped	Cropped – sunflower	Cropped – sunflower
Controls used	Yes	Yes
Number of plots	1 treated plot subdivided into 4 subplots 1 untreated control	1 treated plot subdivided into 4 subplots 1 untreated control
Size per plot	68 m x 15 m (4 sub plots: 17 m x 15 m)	37 m x 30 m (4 sub plots: 18.5 m x 15 m)
Size per untreated control plot	5 m x 15 m	12 m x 18.5 m
Distance between each plot	n/a	n/a
Margin divide	10 m between treated and control	10 m between treated and control
Nominal application rate used (g a.s./ha)	2700 g a.s./ha	2700 g a.s./ha
Actual application rate and target (%)	2679.0 g a.s./ha (99.2%)	2675.7 g a.s./ha (99.1%)
Application date	26th April 2001	16th May 2001
Application method	Sprayer	Sprayer
Type of spray equipment	Azo sprayer with Lurmark F110.01 nozzles, 6 nozzles	Azo sprayer with Lurmark F110.01 nozzles, 6 nozzles
Volume of spray solution applied	253 L/ha	202 L/ha
Meteorological conditions during application		
Cloud cover (%)	0	100
Air temperature (°C)	6.6-9.2	20.3
Atmospheric moisture (%)	57.5	64
Wind speed (m/sec)	0.6-1.4	0.9
Rainfall (mm)	0	-
Irrigation (mm)	Not stated	Not stated
Verification of application available	Yes	Yes

Details	[redacted], Spain	[redacted], France
Trial Number	**Trial 1**	**Trial 2**
Filter paper spray check results	Sub-plot 1 3117 g a.i. /ha Sub-plot 2 2379 g a.i. /ha Sub-plot 3 2482 g a.i. /ha Sub-plot 4 2059 g a.i. /ha Mean 2509 g a.i. /ha	Sub-plot 1 4590 g a.i. /ha Sub-plot 2 3068 g a.i. /ha Sub-plot 3 2029 g a.i. /ha Sub-plot 4 2335 g a.i. /ha Mean 3006 g a.i. /ha

Soil sampling details

Details	[redacted], Spain	[redacted], France
Trial Number	**Trial 1**	**Trial 2**
Sampling intervals (days)	-1, 0, 7, 15, 29, 62, 124, 183, 278, 369 & 533	-1, 0, 7, 14, 29, 62, 128 & 184
Method of soil collection	By soil core	By soil core
Sampling depth	30 cm depth 60 cm depth	30 cm depth 60 cm depth
Number of cores collected per plot	20 per timepoint (5 in each sub-plot)	20 per timepoint (5 in each sub-plot)
Depth of segments	0-10 cm 7.6 cm diameter 10-20 cm 6.3 cm diameter 20-30 cm 5.0 cm diameter 30-60 cm 9.2 cm diameter (at final 2 timepoints)	0-10 cm 7.6 cm diameter 10-20 cm 6.3 cm diameter 20-30 cm 5.0 cm diameter
Storage conditions	Frozen -20°C	Frozen -20°C
Maximum storage length	Not stated	Not stated

Analytical procedures

The analytical method involved initial extraction of the soil with acetone in the presence of hyflosupercel. The acetone extract was evaporated to dryness and the residue re-dissolved in hexane. The hexane extract was cleaned-up using a Florisil column and the aclonifen residues eluted with toluene : hexane (8:2). The solvent was evaporated and the residue re-dissolved in toluene. Final analysis was by gas chromatography with electron capture detection (GC/ECD). The limit of quantification was 0.010 mg/kg.

Degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.1.2.2.1/06. A brief summary of the approach for trigger endpoints is provided below.

The non-normalised data was best fit by the Double First Order in Parallel (DFOP) model at the [redacted] site with a $\chi 2$ error of 6% and the Simple First Order (SFO) model at the [redacted] site with a $\chi 2$ errors of 14%.

II. RESULTS AND DISCUSSION

A. Application Verification

Application was targeted at a rate of 2700 g a.s./ha. The actual application rate ranged from 2679 to 2675 g a.s./ha (99% of the intended application rate, calculated from the sprayer output).

Analytical Methodology:

A full summary of the analytical method is provided in Document MCA-4, Chapter 4.1.2. The method used fulfils the validation criteria defined in the European Guidance Document SANCO/825/00 rev.8 on residue analytical methods. In this study the method was additionally validated concurrently with sample analyses and is suitable for the determination of aclonifen in soil samples by gas chromatography with electron capture detection (GC-ECD).

B. Findings:

Residue Decline

The results for aclonifen are presented below as soil residue concentrations (on a mg/kg dry weight basis) for each of the treated plots.

Spanish trial (Trial 1): The nominal application rate at the Spanish trial was 2.7 kg/ha. The measured initial concentration of aclonifen were significantly lower than both the calibrated spray rate (2.679 kg/ha) and filter paper results (2.509 kg/ha) would indicate. On the assumption of a soil bulk density of 1.5 g/cm3, the calibrated spray rate would correspond to a soil concentration of 1.8 mg/kg for the 10 cm soil depth. The average day 0 residue at 1.008 mg/kg (range 0.675-1.457) is therefore significantly below the calibrated rate. The kinetic evaluation contained in the study report therefore excluded the day 0 residues from the calculation. At the next time point, the soil residue is at 2.007 mg/kg and is in much better agreement with the calibrated application rate. The initial dissipation of aclonifen was rapid followed by a slower second dissipation phase. Residues of aclonifen were detected mainly in the 0-10 cm soil horizon throughout the trial. Low residues were detected in the 10-20 cm horizon and occasionally in the 20-30 cm horizon from day 7 onwards.

French trial (Trial 2): The average initial concentration of aclonifen in the soil samples taken immediately after application was 1.760 mg/kg. This corresponds to an apparent application rate of 2.64 kg/ha which is in good agreement with the nominal application rate of 2.7 kg/ha (assuming a soil bulk density of 1.5 g/cm3). The dissipation of aclonifen was rapid with residue levels less than 1% of the initial concentration by six months. Residues of aclonifen were detected mainly in the 0-10 cm soil horizon throughout the trial except for low residues occasionally detected in lower horizons.

Table 7.1.2.2.1- 7: Residues of aclonifen (expressed as mg/kg) in soil after application at a nominal application rate of 2.7 kg as/ha Trial 1: [redacted], [redacted], Spain

Depth [cm]	DAT										
	-1	0	7	15	29	62	124	183	278	369	533
0 - 10	<LOQ	1.008	1.942	1.425	1.122	0.509	0.453	0.361	0.318	0.214	0.103
10 - 20	<LOQ	ns	0.046	0.046	0.029	0.019	0.014	0.03	0.012	0.023	<LOQ
20 - 30	<LOQ	ns	0.019	<LOQ	<LOQ	<LOQ	0.011	<LOQ	<LOQ	<LOQ	<LOQ
30 - 60	<LOQ	ns	ns	ns	ns	ns	ns	ns	ns	<LOQ	<LOQ

ns= not sampled LOQ = 0.010 mg/kg

Table 7.1.2.2.1- 8: Residues of aclonifen (expressed as mg/kg) in soil after application at a nominal application rate of 2.7 kg as/ha Trial 2: [redacted], [redacted] Alps, France

Depth [cm]	DAT							
	-1	0	7	14	29	62	128	184
0 - 10	<LOQ	1.76	1.328	1.293	1.222	0.213	0.063	0.016
10 - 20	<LOQ	ns	0.013	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
20 - 30	<LOQ	ns	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
30 - 60	<LOQ	ns	ns	ns	ns	ns	ns	ns

ns= not sampled LOQ = 0.010 mg/kg

C. Degradation Rate:

Aclonifen was readily degraded in soil residue trials conducted in Spain and Southern France. The reported DT_{50} values were derived using using simple first order (SFO) and Hockey stick models and ranged from 27 to 46 days. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.1.2.2.1/06. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.1.2.2.1- 4.

Table 7.1.2.2.1- 9: Field dissipation $DisT_{50}$ of aclonifen for trigger purpose, not normalized

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted] – outlier excluded	DFOP	0.939	k 0.046475 k2 0.002547 g 0.706855	6.467	0.00968 6.27e-10	0.009489 -0.001075 0.588483	0.083 0.006 0.945	21.7	332.3
[redacted]	SFO	0.69	k 0.021827	14.49	2.00e-05	9.046e-01	98.618	31.8	105.5

III. CONCLUSION

Following a single application of aclonifen in summer 1991 at a nominal application rate of 2.7 kg/ha to bare soil, prior to emergence of sunflowers, the decline of aclonifen was followed for up to 18 months after application at two sites in Southern Europe ([redacted], Spain and [redacted], Southern France). A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit non-normalised DT_{50} values of 21.7 and 31.8 days at the [redacted] and [redacted] sites respectively.

Assessment and conclusion by applicant:

The study was conducted in accordance with the data requirements of EU (=EEC) 95/36/EC. The study is considered valid to assess the dissipation of aclonifen under field conditions in soil.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.2.2.1/04
Report Author:	[redacted] A. J.
Report Year:	2006
Report Title:	Aclonifen- Kinetic evaluation of soil laboratory studies
Report No:	M-266704-01-1
Document No:	M-266704-01-1
Guideline(s) followed in study:	not specified
Deviations from current test guideline:	Current Guideline: FOCUS Degradation Kinetics (2006, 2014) Does Current Guideline: Not meet guideline recommendations.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

In the previous submission (Addendum to DAR, 2008), this modelling report, which also addressed the kinetic evaluation of field dissipation studies, was evaluated and accepted in part as valid for risk assessment purposes. The report was superseded in part by KCA 7.1.2.1.1/05 ([redacted] & [redacted] 2008, M-266725-01-1). However, the requirements of kinetic evaluations according to FOCUS kinetics have changed and the report is no longer considered as valid at all. It has been fully superseded by KCA 7.1.2.1.1/07 ([redacted] & [redacted], 2019, M-675285-01-1) and hence a summary is not presented in this dossier.

Data Point:	KCA 7.1.2.2.1/05
Report Author:	[redacted] A. J.
Report Year:	2006
Report Title:	Aclonifen - Re-evaluation of aclonifen field dissipation studies
Report No:	M-266725-01-1
Document No:	M-266725-01-1
Guideline(s) followed in study:	not specified
Deviations from current test guideline:	Current Guideline: FOCUS Degradation Kinetics (2006, 2014) Does Current Guideline: Not meet guideline recommendations.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

In the previous submission (Addendum to DAR, 2008), this modelling report was evaluated and accepted as valid for risk assessment purposes. However the requirements of kinetic evaluations according to FOCUS kinetics have changed and the report is no longer considered as valid. It has been

superseded by KCA 7.1.2.1.1/07 ([redacted] 2019, M-675285-01-1) and hence a summary is not presented in this dossier.

Data Point:	KCA 7.1.2.2.1/06
Report Author:	[redacted]; [redacted]
Report Year:	2019
Report Title:	Aclonifen: Kinetic evaluation of the dissipation in soil under field conditions for trigger endpoints
Report No:	VC/19/025F
Document No:	M-675285-01-1
Guideline(s) followed in study:	none
Deviations from current test guideline:	Current Guideline: FOCUS Degradation Kinetics (2006, 2014) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

Un-normalised field dissipation DT_{50} values of aclonifen in the soil matrix under European field conditions were derived for trigger purposes according to FOCUS kinetics (FOCUS, 2006, 2014b) and EFSA guidance on field dissipation studies (EFSA, 2014).

In two separated studies aclonifen was applied to bare soil; the studies were carried out respectively in Germany (in year 1992 on four soils, application at rate between 3018 and 3366 g/ha) and in Spain and France (in year 2001 on two soils, application at rate of 2700 g/ha).

The residue data were kinetically and statistically evaluated, based on the procedure explained by FOCUS kinetics, using the software tool KinGUI 2.1.

Trigger endpoint DT_{50} values and modelling endpoints for aclonifen are summarised in the tables below:-

Field dissipation $DisT_{50}$ of aclonifen for trigger purpose, not normalised

Location (country)	Soil type	pH ($CaCl_2$)	Depth (cm)	DT_{50} (d) actual	DT_{90} (d) actual	St. (χ^2err) (%)	Method of calculation
[redacted] (Germany)	Silty loam	5.67 (KCl)	0-30	46.4	310.1	13.10	FOMC
[redacted] (Germany)	Loam	5.63 (KCl)	0-30	117.6	390.6	24.36	SFO
[redacted] (Germany)	Sandy loam	5.70 (KCl)	0-30	144.5	757.7	8.931	DFOP
[redacted] (Germany)	Sandy loam	7.35 (KCl)	0-30	13.8	210.9	6.515	DFOP
[redacted] (Spain)	Clay Loam	7.6	0-30	21.7	332.3	6.467	DFOP
[redacted] (France)	Sandy Silt Loam	7.8	0-30	31.8	105.5	14.49	SFO

Field dissipation $DisT_{50}$ of aclonifen for modelling purpose, not normalised

Site	Kinetic model	$DisT_{50}$ not normalised (d)
[redacted]	SFO	63.7
[redacted]	SFO	117.6
[redacted]	**SFO**	**196.8**
[redacted]	DFOP DT_{90}/3.32	63.5
[redacted]	DFOP DT_{90}/3.32	100.1
[redacted]	SFO	31.8

I. MATERIALS AND METHODS

The purpose of this study is to evaluate six legacy European field trials conducted at the field sites of [redacted], Meishner [redacted], located in Germany, [redacted] in Spain and [redacted] in France for derivation of persistence and modelling endpoints. The datasets collected were evaluated following FOCUS kinetics guidance (FOCUS, 2014) and EFSA guidance on evaluating laboratory and field studies to obtain $DegT_{50}$ values (EFSA, 2014).

Details of the terrestrial field dissipation studies used in the kinetic evaluation are summarised in CA 7.1.2.2.1/01 and CA 7.1.2.2.1/03. In all sites located in Germany the active substance was applied onto bare soil early summer. No cultivation or irrigation activities occurred during the trial periods. No fertilisers were applied to the trials during the field phase of the study. No herbicides other than aclonifen were applied to maintain bare soil conditions. In the [redacted] and [redacted] the active substance was applied onto bare soil in late spring, the application was made onto bare soil pre-emergence of a sunflower crop.

Soil samples have been collected to a depth of 30 cm at defined time intervals after the application. After sampling, the cores were cut into 0 – 10 cm, 10 – 20 cm and 20 – 30 cm increments, according to soil depth. These core samples were analysed for aclonifen, with a limit of quantification LOQ 1 µg/kg.

Table 7.1.2.2.1- 10: Summary of terrestrial field dissipation studies

Document	Location	Rate (g a.s./ha)	Soil Texture	pH (KCl)	Duration (days)
CA 7.1.2.2.1/01, [redacted] et al, 1994	[redacted] (Germany)	3018	Silty loam	5.67	426
	[redacted] (Germany)	3366	Loam	5.63	432
	[redacted] (Germany)	3186	Sandy loam	5.70	791
	[redacted] (Germany)	3300	Sandy loam	7.35	426
CA 7.1.2.2.1/03, [redacted] & [redacted], 2003	[redacted] (Spain)	2700	Clay Loam	8.0	534
	[redacted] (France)	2700	Sandy Silt Loam	8.2	184

Data was processed following FOCUS kinetics guidance. Concentration of < LOD was replaced with ½ LOD.

Kinetic calculations for the degradation of aclonifen in field soils were performed using KinGUI 2.1 with three kinetic models – SFO, FOMC and DFOP. The goodness of fit with each model was evaluated based on visual assessment and chi-square test, and the degradation rate was then also evaluated via the t-test. The degradation rates for persistence trigger and exposure modelling were then determined from an acceptable kinetic fit following FOCUS and EFSA guidance.

II. RESULTS AND DISCUSSION

Non-normalised field datasets were used to derive persistence endpoints for aclonifen from 6 field dissipation trials in Europe applying the active substance aclonifen. Kinetic parameters were derived for trigger purpose according to FOCUS kinetics (FOCUS 2006, 2014) based on dissipation of the parent compound only. Dissipation rates are evaluated for the corresponding study conditions at the field site (not temperature or moisture normalised).

In all 6 soils, the best fit for dissipation of aclonifen for trigger purpose could be described assuming SFO, FOMC or DFOP model Table 7.1.2.2.1- 11. The statistical assessment shows acceptable results with relative errors ε of the χ2err test from 6.36 % to 24.7 %.

Table 7.1.2.2.1- 11: Data for aclonifen: kinetic and statistical results of the SFO, FOMC, and DFOP curve fits (not normalised)

Kinetic model	$DisT_{50}$	$DisT_{90}$	VA	χ^2err	k_1 / α	k_2 / β	t_b / g	t-test k_1 / k_2
	(d)			(%)	(1/d / -)	(1/d / -)	(d / -)	(-)
[redacted]								
SFO	63.68	211.5	o	17.36	0.010885		-	0.00253
FOMC	**46.35**	**310.1**	**o**	**13.1**	**1.35600**	**69.46980**	-	-
DFOP	43.47	314.7	o	13.2	0.032897	0.004824	0.543795	0.157376/ 0.116100
[redacted]								
SFO	**117.6**	**390.6**	**o**	**24.36**	**0.005895**	-	-	**0.013583**
FOMC	117.6	390.6	o	26.32	8.87e+03	1.506e+06	-	-
[redacted]								
SFO	196.8	653.9	+	11.25	0.0035212	-	-	5.29e-05
FOMC	150.4	905.4	o	10.00	1.56464	269.75723	-	-
DFOP	**144.5**	**757.7**	**+**	**8.931**	**0.0221731**	**0.0025857**	**0.2905999**	**0.18782/ 0.00914**
[redacted]								
SFO	20.67	68.66	-	26.12	0.03354		-	0.010439
FOMC	12.82	188.5	+	7.411	0.71474	7.83144	-	-
DFOP	**13.77**	**210.9**	**+**	**6.515**	**0.079327**	**0.004775**	**0.726187**	**0.007751/ 0.021672**
[redacted]								
SFO	*87.62*	*291.1*	-	*29.92*	*0.007911*		-	*8.17e-5*
FOMC	*24.7*	*407.1*	-	*30.41*	*1.84256*	*163.55295*	-	-
DFOP	*68.35*	*587.4*		*31.67*	*0.014023*	*0.00135*	*0.779486*	*0.1424/ 0.4124*
[redacted] **– outlier excluded**								
SFO	22.05	73.26		26.76	0.031432	-	-	2.79e-10
FOMC	10.95	183		7.838	0.66589	5.97497	-	-
DFOP	**21.72**	**332.3**	**+**	**6.467**	**0.046475**	**0.002547**	**0.766855**	**0.00968/ 6.27e-10**
[redacted]								
SFO	**31.77**	**105.5**	**+**	**14.49**	**0.021817**	**-**	**-**	**2.00e-05**
FOMC	31.77	105.5	+	15.66	3.004e+04	1.377e+06	-	-

For the trial site in [redacted] (Germany), FOMC was selected as best-ft kinetics for trigger endpoint determination based on χ^2 error and visual fit. For modelling purposes, SFO kinetics are acceptable.

For the trial site in [redacted] (Germany), SFO was selected as best-ft kinetics for trigger endpoint determination based on χ2 error and visual fit. For modelling purposes, SFO kinetics are acceptable.

For the trial site in Schichteler (Germany), DFOP was selected as best-ft kinetics for trigger endpoint determination based on χ2 error and visual fit. For modelling purposes, SFO kinetics are acceptable.

For the trial site in [redacted] (Germany), DFOP was selected as best-ft kinetics for trigger endpoint determination based on $\chi 2$ error and visual fit. For modelling purposes, SFO kinetics were not acceptable and a pseudo-SFO DT_{50} was derived from the best-fit DT_{90}/3.32 (DT90 within experimental period).

For the trial site in [redacted] (Spain), initial evaluations with the full datsets resulted in poor kinetic fits due to an apparent outlier of time zero residues – average M0 residues (0.336 mg/kg) are 50% of the day 8 residues and below day 16 and day 30 values. Additional evaluations were conducted excluding time zero residues as an outlier, resulting in a significant improvement both visually and statistically. Time zero residues appear to be too low and the significant improvement of the fit to the remainder of the dataset justifies their exclusion as an outlier. Following exclusion of time zero residues as an outlier, DFOP was selected as best-ft kinetics for trigger endpoint determination based on $\chi 2$ error and visual fit. For modelling purposes, SFO kinetics were not acceptable and a pseudo-SFO DT_{50} was derived from the best-fit DT_{90}/3.32 (DT_{90} within experimental period).

For the trial site in [redacted] (France), SFO was selected as best-ft kinetics for trigger endpoint determination based on $\chi 2$ error and visual fit. For modelling purposes, SFO kinetics are acceptable.

In the following, the kinetic parameters which were obtained with the selected models are compiled. Model selection was described in the preceding section. Trigger endpoints are presented in Table 7.1.2.2.1- 12 with modelling endpoints in Table 7.1.2.2.1- 13 .

Table 7.1.2.2.1- 12: Data for aclonifen: kinetic and statistical results of the SFO, FOMC, and DFOP curve fits (not normalised Field dissipation $DisT_{50}$ of aclonifen for trigger purpose, not normalised

Site	Kinetic model	$DisT_{50}$ not normalised (d)	$DisT_{90}$ not normalised (d)
[redacted]	FOMC	46.4	310.1
[redacted]	SFO	117.6	390.6
[redacted]	DFOP	144.5	757.7
[redacted]	DFOP	13.8	210.9
[redacted]	DFOP	21.7	332.3
[redacted]	SFO	31.8	105.5

Table 7.1.2.2.1- 13: Data for aclonifen: kinetic and statistical results of the SFO, FOMC, and DFOP curve fits (not normalised Field dissipation $DisT_{50}$ of aclonifen for modelling purpose, not normalised

Site	Kinetic model	$DisT_{50}$ not normalised (d)
[redacted]	SFO	63.7
[redacted]	SFO	117.6
[redacted]	SFO	196.8
[redacted]	DFOP DT_{90}/3.32	63.5
[redacted]	DFOP DT_{90}/3.32	100.1
[redacted]	SFO	31.8

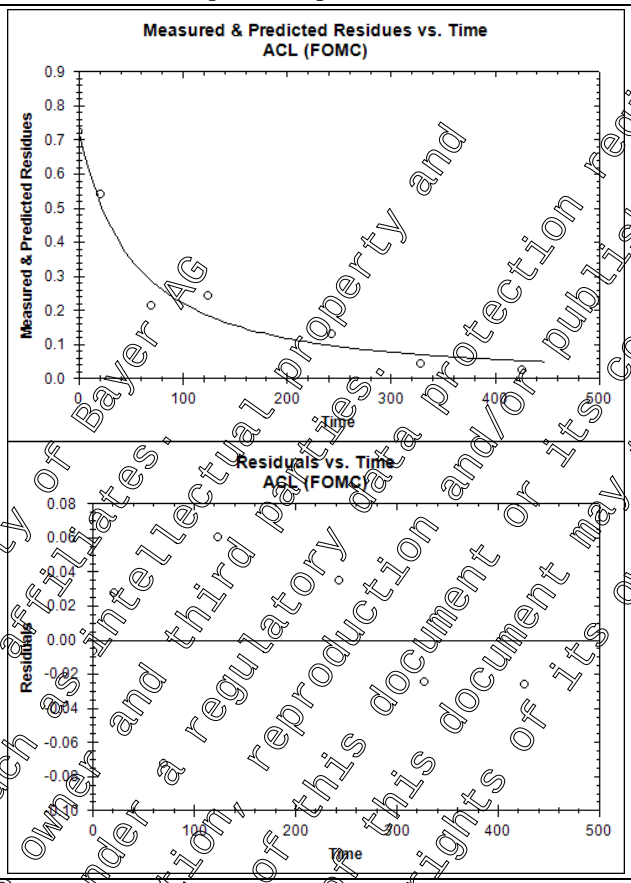
The standard EFSA template can be seen in Table 7.1.2.2.1- 14 and graphical representations in Table 7.1.2.2.1- 15.

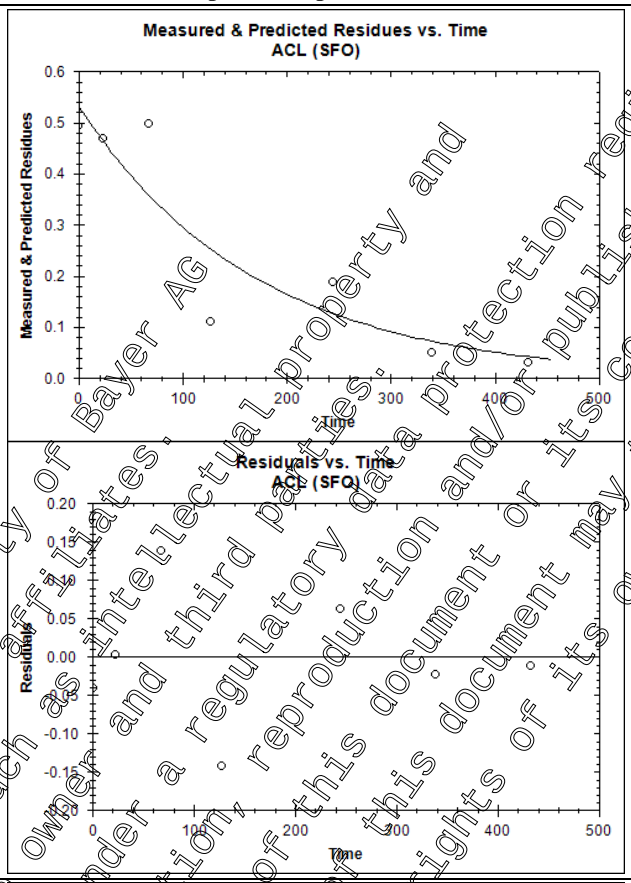
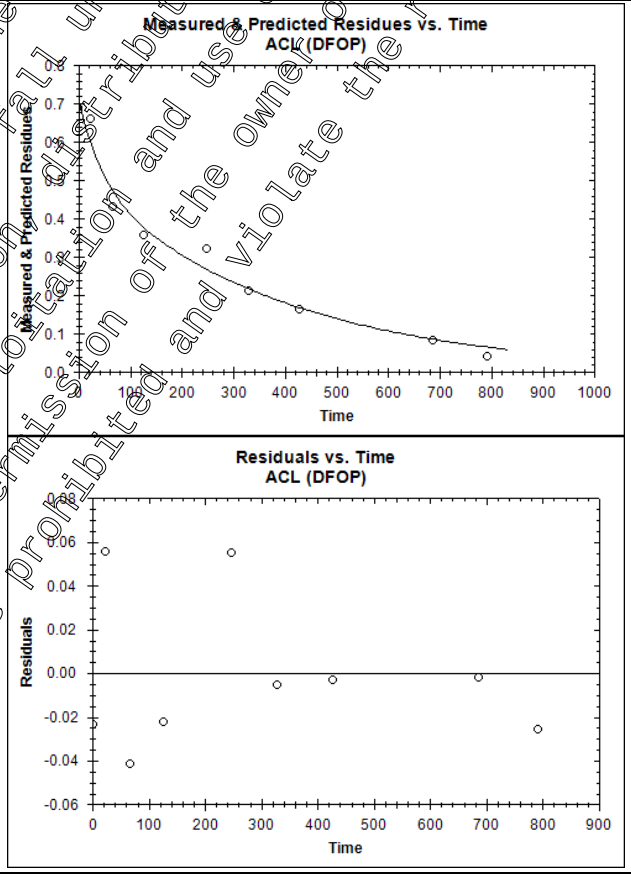
Table 7.1.2.2.1- 14: Standard EFSA template for kinetic fitting

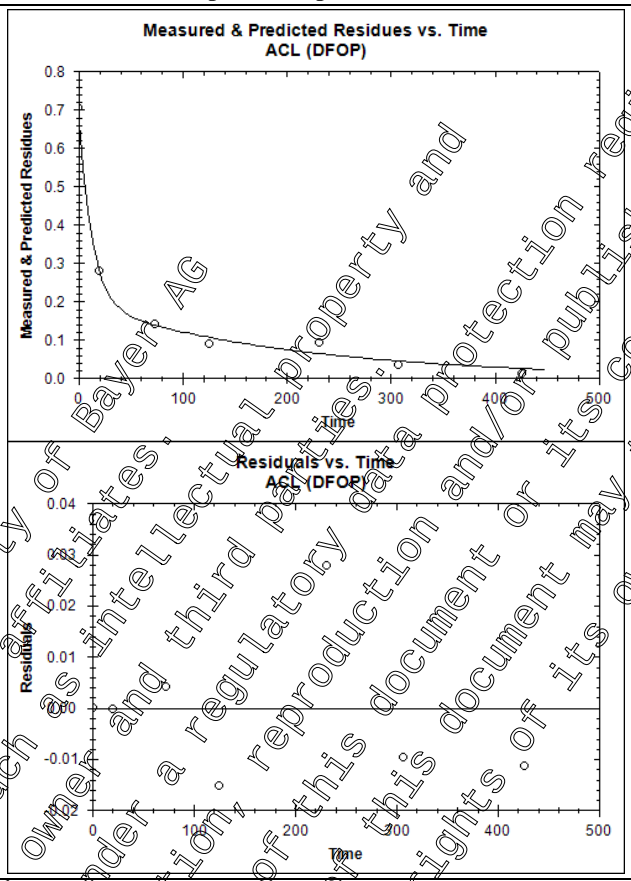
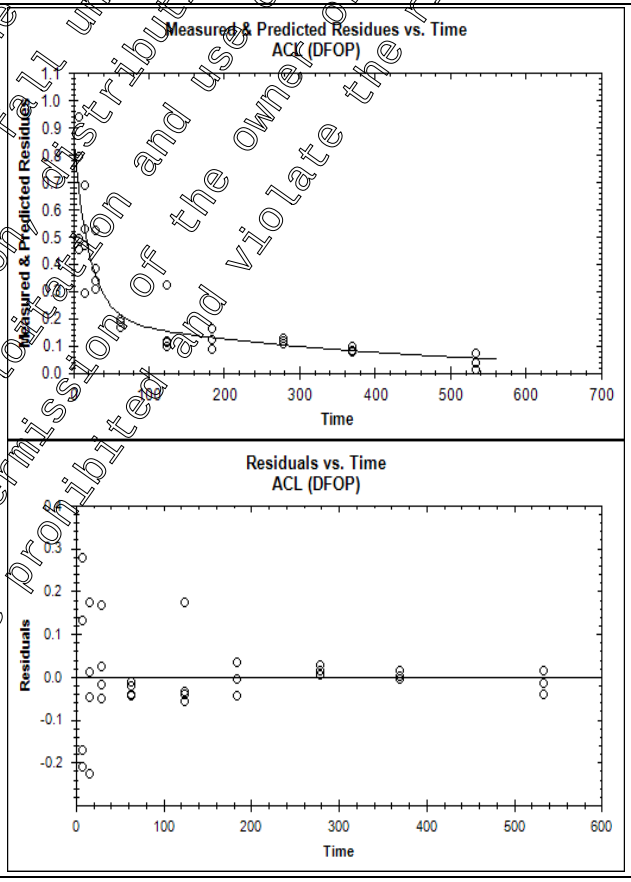
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted], Stratmann et al, 1994	SFO	0.7	k = 0.010885	17.36	0.00253	0.372734	0.808	63.68	211.5
	FOMC	0.7	α 1.35600 β 69.46980	13.1	n.r. n.r.	-0.14633 -53.88370	2.858 192.823	46.35	310.1
	DFOP	0.7	k1 0.032897 k2 0.004824 g 0.543795	13.2	0.157376 0.116100 n.r.	n.r n.r -0.056712	n.r n.r 1.144	43.47	314.7
[redacted], Stratmann et al, 1994	SFO	0.5	k = 0.005895	24.36	0.013583	0.391697	0.674	117.6	390.6
	FOMC	0.5	α 8.87e+03 β 1.506e+06	26.32	n.r. n.r.	8.878e+03 1.306e+06	8.878e+03 1.306e+06	117.6	390.6
[redacted], Stratmann et al, 1994	SFO	0.7	k = 0.0035212	11.25	5.29e-05	0.3870400	0.726	196.8	653.9
	FOMC	0.7	α 1.56464 β 269.75723	10.00	n.r. n.r	-0.26483 -195.19772	3.394 734.712	150.4	905.4
	DFOP	0.7	k1 0.0221731 k2 0.0025857 g 0.2985999	8.931	0.18782 0.00914	n.r n.r -0.0611273	n.r n.r 0.642	144.5	357.7
[redacted], Stratmann et al, 1994	SFO	0.7	k = 0.03354	26.12	0.010439	0.53414	0.824	20.67	68.66
	FOMC	0.7	α 0.71474 β 7.83144	7.411	n.r n.r	0.43363 1.47505	0.956 14.188	12.82	188.5
	DFOP	0.7	k1 0.079327 k2 0.004775 g 0.726187	6.515	0.007751 0.021672 -	- - 0.609800	- - 0.843	13.77	210.9
[redacted], [redacted] & [redacted], 2003	SFO	*0.8*	*k = 0.007911*	*29.92*	*817e-5*	*n.r.*	*n.r.*	*87.62*	*291.1*
	FOMC	*0.8*	*α 1.84256* *β 163.55295*	*30.41*	*n.r* *n.r*	*-2.59937* *-394.83383*	*6.284* *721.940*	*74.7*	*407.1*
	DFOP	*0.8*	*k1 0.014023* *k2 0.00135* *g 0.779486*	*31.67*	*0.142* *0.424* -	- - *-0.243096*	- - *1.802*	*68.35*	*587.4*
[redacted] outlier excluded, [redacted] & [redacted], 2003	SFO	0.939	k = 0.031432	26.76	2.79e-10	0.024149	0.039	22.05	73.26
	FOMC	0.939	α 0.66589 β 5.97497	7.838	n.r n.r	0.028030 -12.61934	1.051 24.369	10.95	183.7
	DFOP	0.939	k 0.046475 k2 0.002547 g 0.766855	6.467	0.00968 6.27e-10 -	0.009489 -0.001075 0.588483	0.083 0.006 0.945	21.72	332.3
[redacted], [redacted] & [redacted], 2003	SFO	0.69	k = 0.021817	14.49	2.00e-05	9.04e+01	98.618	31.77	105.5
	FOMC	0.69	α 3.004e+04 β 1.377e+06	15.66	n.r n.r	3.004e+04 1.377e+06	3.004e+04 1.377e+06	31.77	105.5

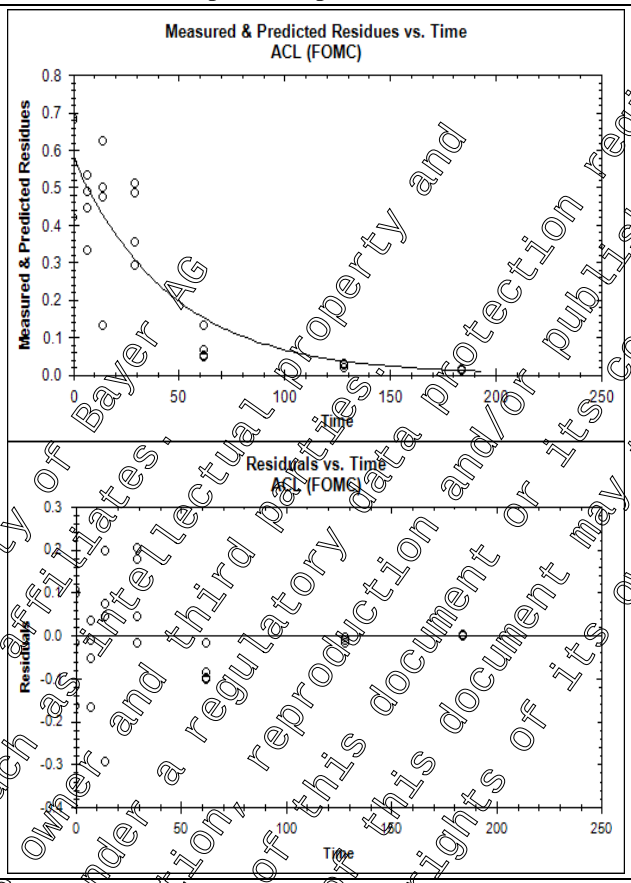
Table 7.1.2.2.1- 15: Graphical representations of best fit models

Trial / Best Fit Model	Graphical Representations

Trial / Best Fit Model	Graphical Representations
[redacted] / FOMC	

Trial / Best Fit Model	Graphical Representations
[redacted] [redacted] / SFO	
[redacted] / DFOP	

Trial / Best Fit Model	Graphical Representations
[redacted] / DFOP	
[redacted] / DFOP	

Trial / Best Fit Model	Graphical Representations
[redacted] / SFO	

III. CONCLUSION

Un-normalised field dissipation DT_{50} values of aclonifen in the soil matrix under European field conditions were derived for trigger purposes according to FOCUS kinetics (FOCUS, 2006, 2014) and EFSA guidance on field dissipation studies (EFSA, 2014).

A worst case SFO DT_{50} of 196.8 days was calculated for use in PEC_{soil} calculations.

Assessment and conclusion by applicant:

The modelling report was conducted according to FOCUS Degradation Kinetics (2006, 2014) and is considered valid to assess best fit and modelling DT_{50} values for aclonifen in field dissipation studies.

Assessment and conclusion by RMS:

CA 7.1.2.2.2 Soil accumulation studies

Soil accumulation studies were carried out with aclonifen as field DT_{90} values indicated some persistence leading to residual residue levels remaining one year after application under Northern European climates. Consequently, accumulation studies were conducted to determine aclonifen levels in soil following annual applications over a three year period at sites near [redacted], Nordrhein Westfalen in Northern Germany and [redacted], Bavaria in Southern Germany. No accumulation of aclonifen residues was observed at either location. The data from these trials has been summarised as separate field and analytical reports (KCA 7.1.2.2.2/01 and KCA 7.1.2.2.2/02). A single summary incorporating all the pertinent details from these documents is given below.

The studies were evaluated during the previous EU review and were accepted as plausible but were not considered sufficient to address the potential accumulation of aclonifen in soil. An assessment of accumulated PEC_{soil} for aclonifen is provided in Document M-CP, Section 9.1.3.

Report reference	Author, Year	Comment
KCA 7.1.2.2.2/01 M-198726-01-2	[redacted], 2000	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.1.2.2.2/02 M-198724-01-1	[redacted], 2000	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.

Data Point:	KCA 7.1.2.2.2/01
Report Author:	[redacted]
Report Year:	2000
Report Title:	Long term trial with regard to investigating the degradation behaviour in soil of the pesticide active ingredient Aclonifen contained in the herbicide Bandur (R)
Report No:	C009517
Document No:	M-198726-01-2
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: ENV/JM/MOCurrent Guideline: No(2016)6; EFSA (2014) Insufficient timepoints for current study design.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Data Point:	KCA 7.1.2.2.2/02
Report Author:	[redacted]
Report Year:	2000
Report Title:	Determination of its residues in soil from an accumulation study in Germany Aclonifen
Report No:	C009516
Document No:	M-198724-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: ENV/JM/MONO(2016)6; EFSA (2014) Insufficient timepoints for current study design
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

A three year terrestrial field dissipation and accumulation study with aclonifen, formulated as BANDUR, a suspension concentrate formulation containing 600 g/l aclonifen, has been conducted at two sites in Germany at [redacted], Nordrhein Westfalen and [redacted], Bavaria. The formulated material was applied each year at the rate required to achieve an annual application of 2.7 kg/ha of aclonifen.

The soil residues were monitored over three years following a single application of aclonifen in spring each year. BANDUR was applied to bare soil prior to crop emergence within a three year crop rotation of potatoes (1997), sunflowers (1998) and field peas (1999). The initial application to the trial located at [redacted], Nordrhein Westfalen was on 7 May 1997 with subsequent applications made on 22 April 1998 and 24 March 1999. The application dates for the trial located at [redacted], Bavaria were 12 May 1997, 4 May 1998 and 6 April 1999.

Samples of soil were taken at intervals throughout the three year period and analysed by a GC-MS method to determine levels of aclonifen present in the samples (shown below).

Mean aclonifen mg/kg (dry weight) 0 – 30 cm										
Trial	**Year 1**				**Year 2**			**Year 3**		
DAT	-1	0	135	350	0	160	336	0	135	345
[redacted]	nd	0.7691	<LOQ	0.0153*	1.0118**	0.014	0.0077	0.7313	0.0177	0.0142
DAT	-1	0	130	337	0	113	337	0	122	351
[redacted]	nd	0.6725	0.0773	0.029*	0.6909	0.0285	0.0168	0.7392**	0.0178	<LOQ

nd = not detected, LOQ = 0.010 mg/kg, DAT = Days after treatment,
* low plateau concentration, ** high plateau concentration

I. MATERIALS AND METHODS

1. Test Substance

Formulation Name:	BANDUR
Test Material	Aclonifen
Formulation Type:	SC
Actual content of active ingredient:	600 g/L
Lot or batch number:	OP970018 (1997 & 1998); OP980353 (1999)

2. Trial Locations & Soils

The soil accumulation of aclonifen was studied at two different locations in Germany. The characteristics of the trial locations and soils types are summarised below.

Locations:	Germany D-[redacted], Nordrhein, Westfalen D-[redacted], Bavaria
Crop history	[redacted], winter wheat (1996) [redacted], winter barley (1996)
Pre-treatment history	[redacted], plowing (25 cm) and 2 x rototiller (15 cm) (1997) [redacted], plowing (25 cm) and 2 x rototiller (15 cm) (1997)
Pesticides used in preceding year	[redacted] 3.0 l/ha + 30 g IPU + Hoestar (April 1996) 0.8 l/ha + 2.0 l/ha Corbel + CCC 720 (April 1996) 0.2 l/ha Karate (June 1996) 1.25 l/ha Granit plus (July 1996) [redacted] 250 mL/dt Abavit – seed dressing (Sept 1995) 2.5 l/ha + 1.0 kg Stopm + IPU (Oct 1995) 1.0 l/ha Harvesan (May 1996) 1.25 l/ha Terpal C (May 1996)
Pesticides used in 1997 (see study report for further details for years 2 & 3)	[redacted] **Northrhine-Westfalia** 5.0 l/ha Basta (border), 5.0 l/ha Basta (under leaf spraying) 2.0 kg/ha Acrobat Plus, 2.0 kg/ha Ridomil MZ Super 2.0 kg/ha Acrobat Plus, 2.0 kg/ha + 0.2 1/ha Ridomil MZ Super + Brestan, 2.0 kg/ha Ridomil MZ Super, 2.0 kg/ha Acrobat Plus [redacted] **, Bavaria** 25 g/ha Cato, 2.5 1/ha Basta (border), 2.0 1/ha Ciluan, 2.0 kg/ha Acrobat Plus, 2.0 kg/ha Acrobat Plus, 0.4 1/ha Shirlan 0.4 1/ha Shirlan, 0.4 1/ha Shirlan
Distance of weather station from test site, for rainfall and air temperature	Bad [redacted] for the trial located in Nordrhein Westfalen (approx 80 km from trial site) [redacted] for the trial located in Bavaria

(approx. 18 km from trial site)

Soils Characteristics

The soils at both test sites were silty loam soils classified as lessivated brown forest soils. The characteristics of the soils are summarised below.

Table 7.1.2.2.2- 1: Physico-chemical characteristics of the soil sites

Characteristic	Units	Area 30	Area 70
Location		[redacted], Nordrhein Westfalen, D-[redacted], Northern Germany	[redacted], Bavaria, D-[redacted], Southern Germany
Textural Class	DIN	Silty Loam	Silty Loam
pH		5.9	6.1
Organic Matter Content	%	2.33	3.3

Table 7.1.2.2.2- 2: Summary of weather data

Location: [redacted], Area 30 Weather Station: Bad [redacted], Approx. 80 km from trial site			**Location:** [redacted], Area 70 Weather Station: [redacted], Approx. 18 km from trial site		
Period	**Temperature (°C)**	**Precipitation (mm)**	**Period**	**Temperature (°C)**	**Precipitation (mm)**
	Mean	**actual**		**Mean**	**actual**
May 06, 1997	9	11	May 11, 1997	18	0
May 07, 1997* [1, 2]	6	2	**May 12, 1997* [1, 2]**	19	0
May 08, 1997	8	0	May 13, 1997	19	0
May 09, 1997	7	4	May 14, 1997	16	5
May 10, 1997	11	2	May 15, 1997	19	0
May 11, 1997	15	0	May 16, 1997	21	0
Sep. 19, 1997 [3]	11	0	Sep. 19, 1997 [3]	15	1
Jan. 01-31, 1998	3.4	77	Jan. 01-31, 1998	0.7	36
Feb. 01-28, 1998	5.5	19	Feb. 01-28, 1998	3.2	15
Mar. 01-31, 1998	5.9	121	Mar. 01-31, 1998	4.7	35
Apr. 01-30, 1998	8.9	119	Apr. 01-30, 1998	8.5	51
May 01 - 31,1998	13.8	84	May 01-31, 1998	14.2	40
June 01-30, 1998	16.0	103	June 01-30, 1998	16.9	48
July 01-31, 1998	15.6	72	July 01-31, 1998	17.4	81
Aug. 01-31, 1998	16.2	82	Aug. 01-31, 1998	18	60
Sep. 01-30, 1998	14.5	141	Sep. 01-30, 1998	12.8	122
Oct. 01-31, 1998	8.7	220	Oct. 01-31, 1998	8.9	142
Nov. 01-30, 1998	2.8	86	Nov. 01-30, 1998	0.7	70
Dec. 01-31, 1998	2.3	70	Dec. 01-31, 1998	-0.3	36
Apr. 21, 1998	8	0	May 03, 1998	11	4
Apr. 22, 1998 [4, 5]**	13	0	**May 04, 1998** [4, 5]**	8	0
Apr. 23, 1998	16	1	May 05, 1998	9	0
Apr. 24, 1998	11	6	May 06, 1998	10	0
Apr. 25, 1998	14	0	May 07, 1998	14	0

Location: [redacted], Area 30 Weather Station: Bad [redacted], Approx. 80 km from trial site			**Location:** [redacted], Area 70 Weather Station: [redacted], Approx. 18 km from trial site		
Period	**Temperature (°C)**	**Precipitation (mm)**	**Period**	**Temperature (°C)**	**Precipitation (mm)**
	Mean	**actual**		**Mean**	**actual**
Apr. 26, 1998	12	8	May 08, 1998	18	0
Sep. 19, 1998 [6]	14	0	Aug. 25, 1998 [6]	13	0
Jan. 01-31, 1999	4.4	77	Jan. 01-31, 1999	0.6	35
Feb. 01-28, 1999	1.0	94	Feb. 01-28, 1999	-1.7	77
Mar. 01-31, 1999	6.4	80	Mar. 01-31, 1999	5	42
Apr. 01-30, 1999	9.6	87	Apr. 01-30, 1999	8.5	55
May 01-31, 1999	13.7	86	May 01-31, 1999	14.3	102
June 01 -30, 1999	15.1	71	June 01-30, 1999	14.9	81
July 01-3 1,1999	19.1	85	July 01-31, 1999	18.4	107
Aug. 01-31, 1999	17.1	99	Aug. 01-31, 1999	17.3	63
Sep. 01-30, 1999	18.3	67	Sep. 01-30, 1999	16.4	76
Oct. 01-31, 1999	9.8	45	Oct. 01-31, 1999	8.5	53
Nov. 01-30, 1999	4.6	81	Nov. 01-30, 1999	1.4	77
Dec. 01-31, 1999	3.3	120	Dec. 01-31, 1999	0.2	82
Apr. 23, 1999	5.8	4	Apr. 05, 1999	10	10
Apr. 24, 1999* [7, 8]**	9.6	0	**Apr. 06, 1999*** **	14	18
Apr. 25, 1999	12.9	0	Apr. 07, 1999	9	0
Apr. 26, 1999	12.2	0	Apr. 08, 1999	8	1
Apr. 27, 1999	6.8	1	Apr. 09, 1999	7	0
Apr. 28, 1999	4.4	0	Apr. 10, 1999	9	0
Aug. 06, 1999 [9]	20.4	0	Apr. 24, 1999 [7, 8]	14	18
Jan. 01-31,2000	2.5	110	Aug. 06, 1999 [9]	13	4
Feb. 01-28,2000	4.9	113	Jan. 01-31,2000	-1.3	37
Mar. 01-31,2000	5.8	138	Feb. 01-28,2000	3.1	58
Mar. 03, 2000 [10]	4.8	8.9	Mar. 01-31,2000	5	83
			Mar. 22, 2000 [10]	8.6	0

Note: * Treatment year 1, ** Treatment year 2 & *** Treatment year 3, numbers in superscript relate to sampling days

B. STUDY DESIGN AND METHODS

1. Experimental dates:

Analytical Phase: 25 April 1997 to 12 May 2000

2. Experimental design

A three year terrestrial field dissipation and accumulation study with aclonifen, formulated as BANDUR, a suspension concentrate formulation containing 600 g/L aclonifen, has been conducted at two sites in Germany at [redacted], Nordrhein Westfalen and [redacted], Bavaria. The formulated material was applied each year, at the rate required to achieve an annual application of 2.7 kg/ha of aclonifen.

The soil residues were monitored over three years following a single application of aclonifen in spring each year. BANDUR was applied to bare soil prior to crop emergence within a three year crop rotation of potatoes (1997), sunflowers (1998) and field peas (1999). The initial application to the trial located at [redacted], Nordrhein Westfalen was on 7 May 1997 with subsequent applications made on 22 April 1998 and 24 March 1999. The application dates for the trial located at [redacted], Bavaria were 12 May 1997, 4 May 1998 and 6 April 1999.

At each trial site, an experimental plot measuring 1600 m² in total was treated with the test substance. Rainfall, air temperature and hours of sunshine were taken from the regional official weather service (Bad [redacted] for the trial located in Nordrhein Westfalen and [redacted] for the trial located in Bavaria).

Soil samples were taken immediately after each treatment and at selected timepoints thereafter. A total of three time points were taken at each trial site per year. Soil cores were taken to a depth of 30 cm throughout the trial period. At each sampling date 20 core samples were combined to give two replicate samples from each trial.

Storage stability samples were prepared at the start of the study by spiking untreated soil from each trial site with aclonifen (at concentrations of 0.05 mg/kg and 0.5 mg/kg). The samples were stored under similar conditions to soil samples from the accumulation trials. The stability of aclonifen residues in both soil matrices was confirmed for up to 33 months at <–18 °C. Field samples were frozen immediately after sampling, stored at <–18 °C and shipped frozen to the analytical laboratory. The samples were then stored at -18 ºC until required for analysis. Samples were stored for a maximum period of *ca.* one year prior to analysis.

Experimental design, plot set up and application details

Details	[redacted]			[redacted]		
Trial Number	Area 30			Area 70		
Duration of study	345 days			351 days		
Uncropped (bare) or cropped	Cropped			Cropped		
Controls used	No			No		
Number of plots	1 treated			1 treated		
Size per treated plot	1600 m²			1600 m²		
Size per untreated control plot:	n/a			n/a		
Distance between each plot	n/a			n/a		
Margin divide	n/a			n/a		
Nominal application rate used (g a.s./ha)	2700			2700		
Actual application rates and target (%)	2730 (101.1%)	2730 (101.1%)	2648 (98.1%)	2730 (101.1%)	2760 (102.25)	2675 (99.15)
Application dates	7 Mar 1997	22 April 1998	24 Mar 1999	12 Mar 1997	4 May 1998	6 Apr 1999
Application method	Knapsack sprayer			Knapsack sprayer		
Type of spray equipment	BASF-GLORIA knapsack sprayer equipped with 6 nozzles on a 250 cm boom			BASF-GLORIA knapsack sprayer equipped with 6 nozzles on a 250 cm boom		
Volume of spray solution applied	4.5 L/ha			4.5 L/ha		
Identification and volume of carrier used	Water 300 L/ha			Water 300 L/ha		
Pan evaporation data available	No			No		
Meteorological conditions during application	1997	1998	1999	1997	1998	1999
Cloud cover (%)	50	0	90	50	100	50
Air temperature (°C)	13	15	11	19	7	10
Atmospheric mositure (%)	81	61	72	52	79	68
Wind speed (m/sec)	0-2	0	0-2	0	2	2
Rainfall (mm)	0	1	0	0	0	18
Irrigation (mm)	0	0	0	0	0	0

Details	[redacted]			[redacted]		
Trial Number	Area 30			Area 70		
Verification of application available	Yes	Yes	Yes	Yes	Yes	Yes

Soil sampling details

Details	[redacted]	[redacted]
Trial Number	Area 30	Area 70
Method of sampling	Random	Random
Sampling intervals (days)	Year 1 0, 135, 350 Year 2 0, 160, 336 Year 3 0, 135, 345	Year 1 0, 130, 357 Year 2 0, 113, 337 Year 3 0, 122, 351
Method of soil collection	50 mm soil core	50 mm soil core
Sampling depth	30 cm depth	30 cm depth
Number of cores collected per plot	20 per timepoint	20 per timepoint
Depth of segments	0-30 cm	0-30 cm
Storage conditions	Frozen -18°C	Frozen -18°C

Analytical procedures

The analytical method involved initial extraction of the soil with acetone in the presence of hyflosupercel, followed by a clean-up procedure with solid phase extraction cartridges (Florisil). Final analysis was by gas chromatography with mass spectroscopic detection (GC/MS) in the selective ion monitoring (SIM) mode. The limit of quantification was 0.010 mg/kg. The stability data confirm that aclonifen in soil samples was stable during storage for at least 33 months at < -18 °C.

II. RESULTS AND DISCUSSION

A. Application Verification

Application was targeted at a rate of 2700 g a.s./ha. The actual application rate ranged from 2648 to 2760 g a.s./ha (98.1-102.2% of the intended application rate, calculated from the sprayer output onto petri dishes at the trial sites).

Analytical Methodology:

A full summary of the analytical method is provided in Document MCA-4, Chapter 4.1.2. Although not all validation parameters according to SANCO/3029/99 rev. 4 are met, the analytical method can be regarded as fit for purpose with regard to this study.

B. Findings:

Residue Decline

The results for aclonifen are presented below as soil residue concentrations (on a mg/kg dry weight basis) for each of the treated plots.

[redacted] **trial:** The measured initial concentration of aclonifen was 0.7691 mg/kg (mean value) immediately after application, equivalent to 3.46 kg/ha which is higher than the nominal application rate of 2.7 kg/ha (assuming a soil bulk density of 1.5 g/cm^3). The dissipation of aclonifen was very rapid with residue levels ≤ 2% of the initial concentration by four months. This rapid dissipation was also observed after the second and third applications.

[redacted] **trial:** The average initial concentration of aclonifen in the soil samples taken immediately after application was 0.6725 mg/kg (mean value). This corresponds to an apparent application rate of 3.03 kg/ha which is in good agreement with the nominal application rate of 2.7 kg/ha (assuming a soil bulk density of 1.5 g/cm^3). The dissipation of aclonifen was rapid with residue levels *ca.* 10% of the initial concentration by four months and < 5% by one year. This rapid dissipation was also observed after the second and third applications.

Table 7.1.2.2.2- 3: Residues of aclonifen (expressed as mg/kg) in soil after applications at a nominal application rate of 2.7 kg as/ha

Depth 0-30 cm	**Year 1**				**Year 2**			**Year 3**		
[redacted] **Trial**										
DAT	**-1**	**0**	**135**	**350**	**0**	**160**	**336**	**0**	**135**	**345**
Replicate 1	nd	0.7732	<LOQ	0.0154	1.018	0.0137	nd	0.6032	0.0149	0.0154
Replicate 2	nd	0.765	nd	0.0151	1.005	0.0143	0.0153	0.8594	0.0204	0.013
Mean	nd	0.7691	<LOQ	0.0153	1.0115	0.014	0.0077	0.7313	0.0177	0.0142
[redacted] **Trial**										
DAT	**-1**	**0**	**130**	**357**	**0**	**113**	**337**	**0**	**122**	**351**
Replicate 1	nd	0.6505	0.0859	0.0254	0.704	0.0244	0.0181	0.8369	0.0355	<LOQ
Replicate 2	nd	0.6945	0.0687	0.0326	0.6777	0.0325	0.0155	0.6415	<LOQ	nd
Mean	nd	0.6725	0.0773	0.029	0.6909	0.0285	0.0168	0.7392	0.0178	<LOQ

nd = not detected; LOQ = 0.010 mg/kg, DAT = Days after treatment

III. CONCLUSION

The accumulation potential of aclonifen was assessed for up to three years after application to cropped soil at two sites in Northern Europe at [redacted], Nordrhein Westfalen and [redacted], Bavaria in Germany. No accumulation of aclonifen residues was observed at either location.

Assessment and conclusion by applicant:

The fact that no accumulation of aclonifen residues was observed at either test site was considered as plausible in the previous EIs submission (DAR_2006). However this study was not conducted to any stated guidance and insufficient timepoints were taken for it to be considered as fully sufficient to assess the accumulation of aclonifen residues in soil following repeated annual applications according to current requirements, and thus the possibility of accumulation of residues in soil has been addressed in the Document MCP-9 by calculation.

Assessment and conclusion by RMS:

CA 7.1.3 Adsorption and desorption in soil

CA 7.1.3.1 Adsorption and desorption

CA 7.1.3.1.1 Adsorption and desorption of the active substance

The adsorption and desorption of aclonifen has been investigated in two studies (KCA 7.1.3.1.1/01 and KCA 7.1.3.1.1/02).

Report reference	Author, Year	Comment
KCA 7.1.3.1.1/01 M-174332-01-1	[redacted], P., 1991	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.1.3.1.1/02 M-562667-02-1	[redacted], E. & [redacted], 2019	New data not yet reviewed under UP

Aclonifen OECD 106 K_{oc} values have been measured in a total of 8 soils. Study KCA 7.1.3.1.1/01 was evaluated during the previous EU review. Study KCA 7.1.3.1.1/02 is a new study for Annex I renewal submission.

The geometric mean K_{oc} value of 5727 and arithmetic mean 1/n value of 0.88 were selected for PEC_{gw} and PEC_{sw} modelling.

Report reference	Soil	Texture	pH	OC [%]	K_f	K_{oc}	1/n
KCA 7.1.3.1.1/01 M-174332-01-1	[redacted] (90/8)	Loam	6.4	1.1	58.5	5318	0.878
	[redacted] (90/10)	Sandy loam	7.3	1.7	92.6	5447	0.885
	[redacted] 2.2 (90/9)	Loamy sand	5.7	2.5	265.3	10612	1.003
KCA 7.1.3.1.1/02 M-562667-02-1	[redacted] Hof AXXa	Sandy loam	5.8	1.7	87.7	5156.9	0.8358
	[redacted] am [redacted]	Silt loam	6.2	2.0	111.9	5594.8	0.8522
	[redacted] Hof	Loam	5.6	2.8	181.5	6480.4	0.8778
	[redacted] Hof Wurmwiese	Sandy loam	5.0	1.9	92.4	4863.8	0.8400
	[redacted] II	Loam	7.0	6.1	252.5	4139.9	0.8615
Arithmetic mean					142.8	5951.6	**0.8792**
Geometric mean					125.0	**5727.0**	0.8778

Data Point:	KCA 7.1.3.1.1/01
Report Author:	[redacted]
Report Year:	1991
Report Title:	Herbicides: Aclonifen-14C: Adsorption/desorption on three soils
Report No:	R007158
Document No:	M-174332-01-1
Guideline(s) followed in study:	OECD: 106
Deviations from current test guideline:	Current Guideline: OECD 106 (2000) Minor deviations only
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

This study has a number of deviations from the current version of OECD 106 (2002) including: Four test concentrations were used instead of five, soil was not pre-equilibrated with 0.01M $CaCl_2$ before adsorption measured, interstitial water in soil was

not accounted for in measurements. However aclonifen is so strongly adsorbed to soil these issues do not impact significantly on the K_{foc} and 1/n values measured in the study

Executive Summary

The adsorption/desorption characteristics of [U-^{14}C-aniline] labelled aclonifen were determined by the batch equilibrium method in three soils at a concentration range covering two orders of magnitude. The soils were obtained from agricultural areas representing different graphical origin and different soil properties. The soil characteristics were as follows:

Soil ID	Texture	pH	OC [%]
[redacted] (90/8)	Loam	6.4	1.1
[redacted] (90/10)	Sandy loam	7.3	1.7
[redacted] 2.2 (90/9)	Loamy sand	5.7	2.5

In an initial screening experiment the necessity of performing the advanced tests was determined. The results showed that strong adsorption had taken place. A mass balance was also determined. In the first preliminary study, the times required for equilibrium were determined. A shaking time between 16 and 24 hours was deemed acceptable for the desorption cycles. A second preliminary study was carried out to determine satisfactory equilibrium conditions for the adsorption cycle. An agitation period of 48 hours was deemed acceptable for the adsorption cycle.

The definitive adsorption and desorption studies were conducted in glass Soviref tubes (20 mL), at 20°C. Soil samples were treated with solutions of [U-^{14}C-aniline] labelled aclonifen in calcium chloride to produce duplicate samples per soil, with initial concentrations in the aqueous phase of 0.01, 0.05, 0.2 and 1.0 µg/L. The adsorption phase was followed by five desorption cycles.

In the definitive test individual recoveries ranged from 95.6 to 97.3% AR for all soils and test concentrations. Values for the Freundlich adsorption coefficients (K_f) are summarised in the table below. They ranged from 58.5 to 265.3 mL/g with corresponding values referenced to organic carbon (K_{foc} varying from 5318 to 10612 mL/g. Values for the Freundlich coefficient of adsorption 1/n ranged from 0.885 to 1.003. The Freundlich adsorption and desorption constants for aclonifen in soil are summarised below.

Soil name	Adsorption		Desorption (1st cycle)	
	K_f	K_{foc}	K_f	K_{foc}
[redacted] (90/8) Loam	58.5	5318	69.8	6345
[redacted] (90/10) Sandy Loam	92.6	5447	99.9	5876
[redacted] 2.2 (90/9) Loamy Sand	265.3	10612	304.1	12164

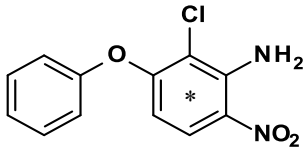
The desorption $K_{F(des)}$ and the normalized $K_{Foc(des)}$ values were 1.1 to 1.2 times higher than those obtained for adsorption, indicating that aclonifen once adsorbed to soil is not readily desorbed.

There is no significant correlation between pH and adsorption for the investigated soils.

In all soils investigated aclonifen was shown to be very strongly adsorbed with K_{foc} values which predict that it is immobile in soil.

I. MATERIALS AND METHODS

1. Test Material: [U-^{14}C-aniline]-aclonifen



* indicates position of ^{14}C radiolabel

Chemical name (IUPAC): 2-chloro-6-nitro-3-phenoxyaniline

Lot or batch number: KWC 1747

Specific radioactivity: 18.55 mCi / mmol or 2.59 MBq / mg

Radiochemical purity: 99.6%

CA registry number: 74070-46-5

Stability of test compound: Stable, determined within study

Application vehicle: Calcium chloride

2. Soils Sorption tests were performed with three soils covering a range of pH, organic carbon content and texture. The characteristics of the European soils are summarised below.

Table 7.1.3- 1: Physico-chemical characteristics of the soils

Soil	[redacted]	[redacted]	[redacted] **2.2**
Batch ID	**90/8**	**90/10**	**90/9**
Geographic Location	[redacted] France	nr [redacted], West Sussex, England UK	[redacted] Germany
GPS coordinates	Not reported	Not reported	Not reported
Soil preparation	Air-dried and sieved to 2 mm		
Textural Class (USDA)	Loam	Sandy loam	Loamy sand
Sand (%) Silt (%) [A] Clay (%) [A]	40.5 40.7 17.0	66.4 15.6 17.9	76.5 19.7 3.8
pH (media not reported)	6.4	7.3	5.7
Org. Matter [B] (%)	1.8	3.0	4.2
Org. Carbon (%)	1.1	1.7	2.5
CEC (meq/100 g)	8.0	10.52	8.0
Bulk Density (g/cm^3)	1.1	1.23	1.1

[A] According to USDA classification; [B] % Organic matter = % organic carbon x 1.724
CEC: Cation exchange capacity

B. STUDY DESIGN AND METHODS

1. In-life dates:

13 Sept 1990 – 21 March 1991

2. Experimental design

The adsorption to test vessels, equilibration time and parental mass balance were determined in a screening test. Preliminary studies were carried out in order to determine the time to reach adsorption equilibrium at a soil-to-solution ratio of 1:5.

For the definitive test, sample aliquots of 2 g dry weight of soil were weighed into glass centrifuge tubes. Following pre-equilibration, a solution of the test item in 0.01 M aqueous calcium chloride (10 mL) was added to each sample. Initial nominal concentrations of the ^{14}C-test item in the aqueous phase were 1.0, 0.2, 0.05 and 0.01 µg/L, thus covering two orders of magnitude. Each determination was performed in duplicate by shaking with a wrist-action shaker in the dark at 20 ± 2 °C.

For the definitive test an adsorption step of 48 hours was performed for all soils followed by 5 desorption steps of 18 hours each. For work-up the aqueous supernatant was separated from soil by decantation and centrifugation. Radioactivity in water and soil extracts was determined by liquid scintillation counting (LSC). An assessment of the stability of the test item in methanol extracts was undertaken. The stability of aclonifen in the extraction procedure was conclusively established. Finally, the adsorption and desorption parameters were calculated using the Freundlich adsorption isotherm.

Adsorption phase

Parameter		Description
Soil condition		Soils were air-dried and sieved to 2 mm
Soil sample weight		2 g (dry weight) per replicate
Equilibration solution		0.01M $CaCl_2$ for 48 hours
Control (screening test)		No soil (test item in 0.01M $CaCl_2$ only)
Test item concentration	Nominal application rates	Nominal concentrations in test solution: 0.01, 0.05, 0.2 and 1.0 µg/L
	Analytically (LSC) measured concentrations	Concentrations in test solution: 0.009805, 0.05031, 0.2049 and 0.9668 µg/L
Identity and concentration of co-solvent		Calcium chloride
Soil: Solution ratio		1:5 i.e. 2 g soil dry weight equivalent to 10 mL solution
Number of replicates	Control	Not stated
	Treatments	Duplicate
Equilibration conditions	Time	48 hours
	Temperature	19-22°C
	Dark	Wrapped in foil
	Shaking method	Mechanical wrist shaker
Method of separation of supernatant		Centrifugation
Centrifugation	Speed (g)	500 g
	Duration	10 minutes
	Method of separating supernatant	Supernatant was carefully decanted.

Desorption phase

Parameter	Description
Soil samples from adsorption phase used	Yes
Amount of test item present in the adsorbed state/adsorbed amount (mg a.i./kg soil)	The amounts of test item adsorbed to soil after adsorption were > 90% AR (reported for screening test).
Number of desorption cycles	5
Equilibrium solution and quantity used per treatment for desorption	The decanted solution was replaced by fresh aqueous 0.01 M $CaCl_2$ solution. A total volume of 10 mL was used as equilibration solution.
Soil: Solution ratio	1:5 i.e. 2 g soil dry weight equivalent to 10 mL solution

Parameter		Description
Number of replicates	Control	Not stated
	Treatments	Duplicate
Desorption Equilibration conditions	Time	18 hours
	Temperature	19-22°C
	Dark	Not stated
	Shaking method	Mechanical wrist shaker
Method of separation of supernatant		Centrifugation
Centrifugation	Speed (g)	500 g
	Duration	10 minutes
	Method of separating supernatant	Supernatant was carefully decanted.
Desorption cycles 2 to 5	Method	Same as above

Analytical procedures

The purity of the test item was investigated by radio-TLC analysis using 3 different mobile phases on pre-coated silica plates with its identity confirmed by co-chromatography with an analytical standard.

The parental mass balance was established in the pre-test and advanced test. Supernatants were analysed by LSC. Soil samples were extracted three times with methanol at ambient temperature. The radioactive content in the combined soil extracts was determined by LSC. Radio-TLC, radio-HPLC and mass spectrometry were used to confirm the presence of aclonifen in the pooled methanol extracts from the 1.0 µg/L treatments for each soil.

II. RESULTS AND DISCUSSION

A. Mass balance results of preliminary tests

For the screening tests the overall mean mass balance of each soil investigated ranged from 104.4 to 109.1% AR (see table below).

Table 7.1.3- 2: Screening test: Total recovery (%) of [U-^{14}C-aniline]-aclonifen in samples dosed at 1.0 µg/L after adsorption and desorption phases

Soil			Adsorbate	1st Desorbate	2nd Desorbate	1st Methanol Extract	2nd Methanol Extract	Mean Recovery
[redacted]	90/8	Loam	1.97	4.65	3.58	79.55	8.62	104.37
[redacted]	90/10	Sandy loam	4.87	2.89	2.53	87.38	10.32	107.99
[redacted] 2.2	90/9	Loamy sand	2.42	1.7	1.38	94.79	8.8	109.09

B. Transformation of test substance:

The stability of the test item in contact with soil under the conditions of the definitive test was confirmed by radio-TLC, radio-HPLC and mass spectrometry to be > 90% AR for all soils over the duration of the test.

C. Findings:

Within definitive tests, the amount of [U-^{14}C-aniline]-aclonifen recovered from the test system (adsorbate, desorbate & methanol extract) ranged from 93.3 to 101.0% AR for [redacted] soil, 94.2 to 104.4% AR for [redacted] soil and 90.4 to 100.9% AR for [redacted] 2.2 soil (see table below).

Table 7.1.3- 3: Definitive test: Total recovery of [U-^{14}C-aniline]-aclonifen in samples after adsorption and desorption phases

Soil / Test concentration (µg/L)	[redacted] 90/8 Loam	[redacted] 90/10 Sandy loam	[redacted] 2.2 90/9 Loamy sand
1.0	101.0	104.4	100.9
0.20	94.5	94.2	94.5
0.05	93.7	95.0	94.7
0.01	93.3	95.4	90.4
Mean recovery (%)	95.6	*97.2	*95.1
SD	± 3.5	± *4.5	± *4.6

Values derived from mean values of duplicate samples in terms of percentages of AR, SD = standard deviation
*Values calculated to be slightly different to reported values

The definitive test was performed at a soil-to-solution ratio of 1:5 for all three soils. Equilibrium of the test item was established after 48 hours shaking at this soil solution ratio, thus an adsorption time of 48 hours was chosen for the definitive test followed by a desorption step of 18 hours. The adsorption behaviour of [U-^{14}C-aniline]-aclonifen was accurately measured using a nominal concentration range of 0.01 µg/L to 1.0 µg/L by the Freundlich equation for all soils. Concentrations of [U-^{14}C-aniline]-aclonifen in aqueous and solid phase following 48 hours of adsorption are shown in the table below.

Table 7.1.3- 4: Definitive test: Concentration of [U-^{14}C-aniline]-aclonifen in aqueous and solid phase following 48 hours of adsorption.

Concentration (µg/mL)	Soil (µg/g)	Solution (µg/mL)
Soil ID	**[redacted] 90/8 Loam**	
0.01	0.047	0.00032
0.05	0.240	0.0018
0.20	0.985	0.009
1.0	4.58	0.058
Soil ID	**[redacted] (90/10) Sandy Loam**	
0.01	0.048	0.00015
0.05	0.245	0.0012
0.20	0.995	0.006
1.0	4.70	0.036
Soil ID	**[redacted] 2.2 (90/9) Loamy Sand**	
0.01	0.048	0.00015
0.05	0.248	0.0009
0.20	1.007	0.0036
1.0	4.77	0.019

The adsorption constants K of the Freundlich isotherms ranged from 58.5 to 265.3 mL/g with associated Freundlich exponents 1/n ranging from 0.878 to 1.003. The corresponding correlation coefficients for the adsorption isotherms ranged from 0.9985 to 0.9994 indicating a linear fit to the measured data. When normalized for the organic carbon content of soil K_{foc} values ranged from 5318 to 10612 mL/g. The Freundlich adsorption and desorption constants for aclonifen in soil are summarised below.

Table 7.1.3- 5: Adsorption constants of [U-^{14}C-aniline]-aclonifen in soil

Soil	Texture	pH *	OC [%]	Clay [%]	K_F	K_{Foc} **	1/n
[redacted] (90/8)	Loam	6.4	1.1	17.0	58.5	5318	0.878
[redacted] (90/10)	Sandy loam	7.3	1.7	17.9	92.6	5447	0.885
[redacted] 2.2 (90/9)	Loamy sand	5.7	2.5	3.8	265.3	10612	1.003
Arithmetic mean					138.8	7123	0.922
Geometric mean					112.8	6749	0.920
pH dependence						No	

* pH: Media not reported
**K_{foc}: Freundlich coefficients of adsorption
1/n: Slope of the Freundlich isotherms
K_{foc}: Adsorption coefficient per organic carbon (K_f x 100/% organic carbon)

The desorption K_{foc} values were in the same order of magnitude as those for the adsorption K_{foc} values and increased very slowly, if at all, with each desorption cycle.

Aclonifen was strongly adsorbed to soil with only 2 to 8% remaining in solution after the adsorption cycle. Each desorption cycle removed progressively smaller amounts (*ca.* 1 to 3%) with approximately 90% remaining on the soil after the final desorption cycle.

Table 7.1.3- 6: Summary of Freundlich desorption constants K_f and K_{oc} values

Desorption	Soil	[redacted] (90/8)	[redacted] (90/10)	[redacted] 2.2 (90/9)
	Textural class	Loam	Sandy Loam	Loamy Sand
1	K_{des}	69.8	99.9	304.1
	1/n	0.854	0.841	0.975
	$K_{oc\ des}$	6215	5876	12164
	Correlation	0.9985	0.9991	0.9992
2	K_{des}	87.9	111.3	293.0
	1/n	0.875	0.850	0.952
	$K_{oc\ des}$	7991	6547	11720
	Correlation	0.9999	0.9985	0.9980
3	K_{des}	85.1	106.5	276.9
	1/n	0.850	0.829	0.929
	$K_{oc\ des}$	7736	6265	11076
	Correlation	0.9999	0.9994	0.9990
4	K_{des}	90.5	110.5	296.2
	1/n	0.887	0.827	0.937
	$K_{oc\ des}$	8227	6500	11848
	Correlation	0.9999	0.9987	0.9984
5	K_{des}	100.0	107.1	245.5
	1/n	0.874	0.821	0.902
	$K_{oc\ des}$	9091	6300	9820
	Correlation	1.0000	0.9994	0.9998

D. OECD 106 evaluation checklist:

Relevant quality checks were performed to evaluate the acceptability of the study according to the EFSA Technical Report on the Outcome of Pesticides Peer Review Meeting on the OECD 106

Evaluators Checklist (2017). These checks confirmed that the mass balance of 95.1-97.2% (mass balances >90%) and % adsorption of 94.0-98.3% were all acceptable. The acceptability of the analytical method was not confirmed over the entire range of concentrations measured. The LSC LOQ required has since been calculated to be 0.52 Bq per 2 mL sample (equivalent to 0.0001 µg/L, 2 orders of magnitude lower than the lowest test concentration). The reported blank samples in the study are 9 to16 dpm (equivalent to a maximum of 0.27 Bq sample), which would be lower than the LOQ.

The use of the indirect method was appropriate based on a Kd * soil/solution ratio > 0.3 in all soils. The graphical fits of the Freundlich equation based on the standard linear regression form using log-log transformed data alongside the associated residual plots were evaluated and found to be acceptable. The R^2 of the standard linear regressions ranged from 0.999 to 1.000 and the visual fit of both the standard regression and the residual plots were acceptable.

The evaluation confirmed the study was fully acceptable according to all quality checks and thus it can be concluded the study is appropriate for use in regulatory modelling. The results of the evaluation are summarised in the tables below.

Table 7.1.3- 7: Results of OECD 106 evaluation checklist

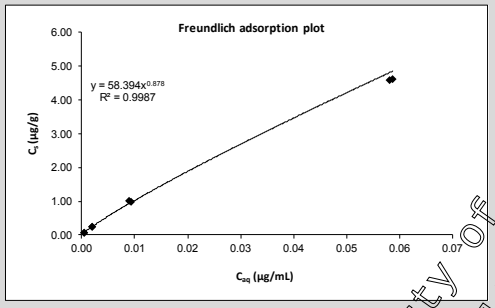
Soil	Units	Quality criteria	[redacted] (90/8) Loam	[redacted] (90/10) Sandy Loam	[redacted] 2.2 (90/9) Loamy Sand
Adsorption method (direct/indirect)	-	-	indirect	indirect	indirect
Soil : solution ratio	g/mL	-	1:5	1:5	1:5
Mass balance of ^{14}C (all concentrations)	%	>90%	95.6	97.2	95.1
Adsorbed percentage	%	>20%	93.95-96.87	96.14-97.98	97.92-98.30
K_d x (soil:solution ratio)		>0.3	15.69-31.26	24.96-48.33	47.33-57.20
$^{\#}K_{fE}$ / K_f	-	<1.2	1.05	1.05	1.05
$^{ads}K_f$ (95% conf. int)	L/kg	*	58.394 (48.50-70.31)	92.824 (79.53-107.65)	264.555 (200.03-349.89)
$^{ads}1/n$ (95% conf. int)	-	*	0.878 (0.85-0.91)	0.885 (0.86-0.91)	1.003 (0.96-1.05)
$^{ads}R^2$	-	-	0.999	0.999	0.998
$^{ads}K_{foc}$	L/kg	-	5308.6	5442.6	10582.2
Visual fit to Freundlich isotherm	-	-	Good	Good	Good
Residual plots randomly distributed	-	-	Acceptable	Good	Good

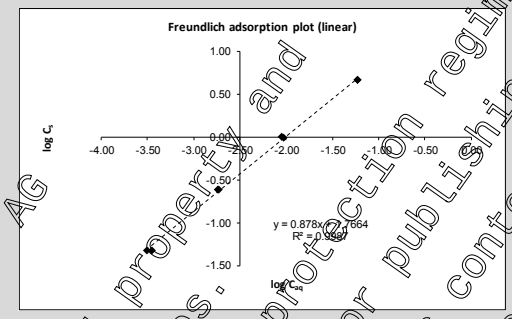
* Confidence intervals should be narrow (statistical analysis not performed by automated tool).
$^{\#}$F factor – for all soils, calculated using tier 3 data where F = 100 - % mass balance (^{14}C supernatant [%] + ^{14}C soil extract [%]). The F value was corrected for purity test material (99.6%).
OECD 106 Calculation Tool v2 used.
Note on assessing visual fits:
Poor - an unacceptable fit, the fitted curve does not represent the trend of the data points and residuals show strong deviations from random distribution. **Acceptable** – the fitted curve describes the trend of the data points, residuals may show some deviation from random distribution but it is not significant. **Good** – the fitted curve closely follows all the data points, residuals are randomly distributed.

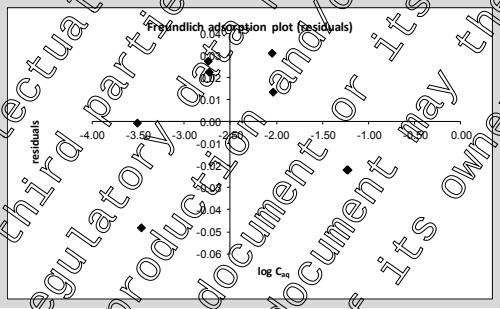
Freundlich plots from OECD 106 Checklist

[redacted] 90/8 Loam soil

Linear plot parameters and derived adsorption values			
Slope =	0.8780	$\log K_F$ =	1.7664
Intercept =	1.7664	K_F =	58.394
Correlation coefficient (r) =	0.9993	K_{Foc} =	5308.6
Coefficent of determination (r^2) =	0.9987	1/n =	0.8780

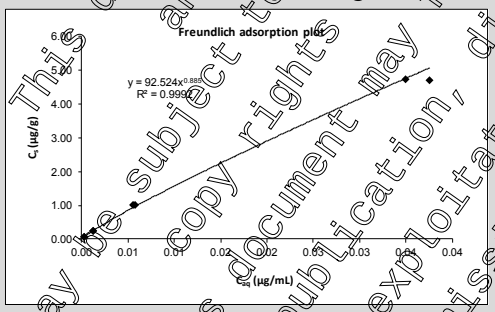


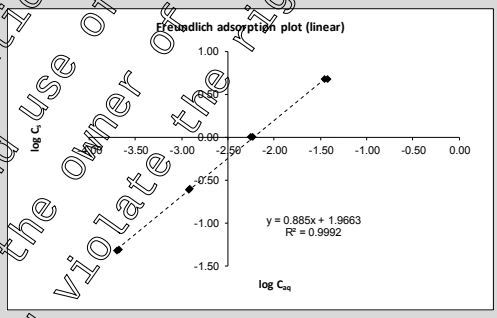


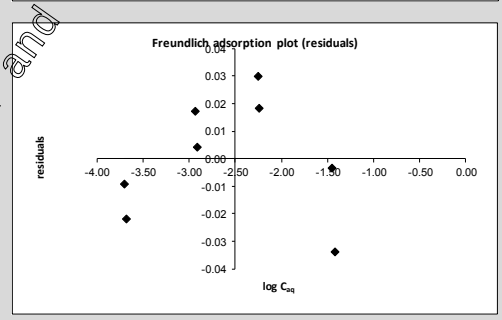


[redacted] 90/10 Sandy loam soil

Linear plot parameters and derived adsorption values			
Slope =	0.8850	$\log K_F$ =	1.9663
Intercept =	1.9663	K_F =	92.524
Correlation coefficient (r) =	0.9996	K_{Foc} =	5442.6
Coefficent of determination (r^2) =	0.9992	1/n =	0.8850

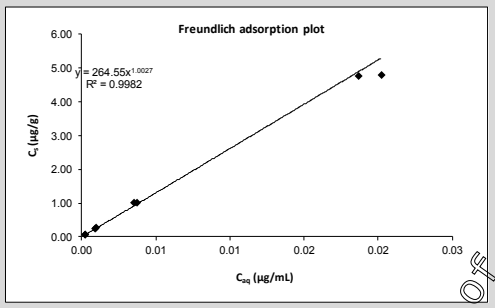


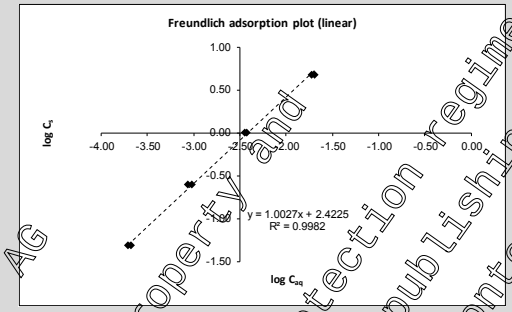




[redacted] 2.2 90/9 Loamy sand soil

Linear plot parameters and derived adsorption values			
Slope =	1.0027	$logK_F$ =	2.4225
Intercept =	2.4225	K_F =	264.555
Correlation coefficient (r) =	0.9991	K_{Foc} =	10582.2
Coefficent of determination (r^2) =	0.9982	1/n =	1.0027





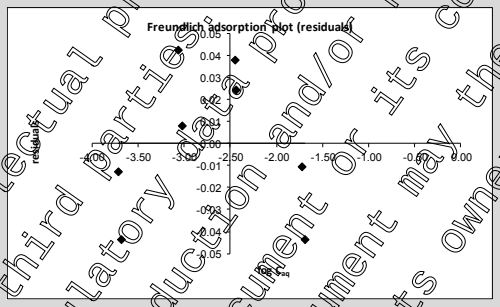


Table 7.1.3- 8: Summary of Quality Criteria and Regulatory Interpretation

Aclonifen		Quality Criteria			Overall Conclusion	Regulatory Interpretation
Soil Type	Soil Name	Met	Partially Met	Not Met		
Loam	[redacted] (90/8)	9	0	0	Met	Acceptable
Sandy Loam	[redacted] (90/10)	9	0	0	Met	Acceptable
Loamy Sand	[redacted] 2.2 (90/9)	9	0	0	Met	Acceptable

Table 7.1.3- 9: Impact on Endpoints

Soil Type	Soil Name	K_{foc} (Reported)	K_{foc} (OECD tool)	1/n (Reported)	1/n (OECD tool)
Loam	[redacted] (90/8)	5318	5309	0.878	0.878
Sandy Loam	[redacted] (90/10)	5447	5443	0.885	0.885
Loamy Sand	[redacted] 2.2 (90/9)	10612	10582	1.003	1.003
Arithmetic Mean			-	0.922	0.922
Geometric Mean		6749	6737	-	-
pH dependence		No	No	-	-

The small differences between the reported values and the OECD calculation tool (v2) are considered to be due to rounding in the calculations. The reported values have been used in modelling calculations.

III. CONCLUSION

The adsorption constants K_F of aclonifen ranged from 58.5 to 265.3 mL/g (geometric mean: 112.8 mL/g). The K_{Foc} values ranged from 5318 to 10612 mL/g (geometric mean: 6749 mL/g). Depending on the soil, the desorption constants $K_{F(des)}$ of aclonifen were about 1.1 to 1.2 times higher than the respective adsorption constants, indicating a strengthened binding of aclonifen once adsorbed to soils representing conditions relevant for the environment.

There was no significant correlation between pH and adsorption for the investigated soils.

Aclonifen was stable during the test. The parental mass balance was ≥ 95.1% AR. No major degradation product was observed.

These results indicate that aclonifen is strongly absorbed to soil and is predicted to be immobile in soil according to the Briggs classifications.

The OECD 106 Checklist (v2) was used to evaluate the study. All 3 soils can be considered 'met' with regard to the quality criteria and therefore acceptable for regulatory use.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 106 (1981).

It has a number of deviations from the current version of OECD 106 (2002). However aclonifen is so strongly adsorbed to soil these issues do not impact significantly on the K_{fec} and 1/n values measured in the study. The study meets all the quality criteria of the EFSA OECD 106 Checklist and is considered valid to assess the adsorption and desorption characteristics of the aclonifen in soil.

Assessment and conclusion by RMS:

Data Point:	KCA 7.1.3.1.1/02
Report Author:	[redacted]; [redacted]
Report Year:	2019
Report Title:	Amendment no. 1 to final report [aniline-UL-14C]aclonifen: Adsorption / desorption on five soils
Report No:	EnSa-16-0213
Document No:	M-562667-02-1
Guideline(s) followed in study:	OECD Test Guideline No. 106 Commission Regulation (EU) No 283/2013 in accordance with Regulation (EC) No 1107/2009 US EPA OCSPP Test Guideline No. 835.1230
Deviations from current test guideline:	Current guideline: OECD 106 (2000) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The adsorption / desorption behaviour of [aniline-UL-^{14}C] labelled aclonifen was studied in five soils in batch equilibrium experiments in the laboratory in the dark at 20 ± 2 °C. The soil characteristics were as follows:

Soil	Soil ID	Source	Texture (USDA)	pH *	OC [%]
[redacted] Hof AXXa	AX	[redacted], Germany	sandy loam	5.8	1.7

[redacted] am [redacted]	HH	[redacted], Germany	silt loam	6.2	2.0
[redacted] Hof	HN	[redacted], Germany	loam	5.6	2.8
[redacted] Hof Wurmwiese	WW	[redacted], Germany	sandy loam	5.0	1.9
[redacted] II	DD	[redacted], Germany	clay loam	7.0	6.1

* pH values were derived from aqueous 0.01 M $CaCl_2$ suspensions

The adsorption phase of the study was carried out using air-dried, sterilized soils equilibrated in aqueous 0.01 M $CaCl_2$ solution with soil-to-solution ratios of 1/80. Nominal concentrations of approx. 0.060, 0.028, 0.0055, 0.0026 and 0.00051 mg/L of aclonifen were applied in aqueous 0.01 M $CaCl_2$ solution. The desorption phase was performed by supplying pre-adsorbed soil samples with fresh aqueous 0.01 M $CaCl_2$ solution. Adsorption and desorption took place for 24 hours each for all soils. The test was performed in centrifuge tubes with screw caps.

The aqueous supernatant after adsorption and desorption was separated by centrifugation and the amount of test item in the supernatants was analysed by liquid scintillation counting (LSC). After the desorption step, the soil was dried and the residues were determined by combustion/LSC. The sorption parameters were calculated using Freundlich isotherms.

The test item was sufficient stable throughout the study. Mean parental mass balances were 94.9, 96.5, 97.8, 100.0 and 98.8% AR for soil AX, HH, HN, WW and DD, respectively.

Mean material balances were 104.9% AR for soil AX (range from 95.7 to 110.0% AR), 106.4% AR for soil HH (range from 104.0 to 110.7% AR), 107.4% AR for soil HN (range from 104.5 to 110.2% AR), 106.5% AR for soil WW (range from 103.4 to 109.2% AR) and 109.2% AR for soil DD (range from 106.4 to 113.0% AR).

In the definitive adsorption test 67.0 – 82.9% AR, 71.8 – 84.4% AR, 80.3 – 88.0% AR, 70.3 – 84.1% AR and 86.5 – 93.0% AR were adsorbed in soil AX, HH, HN, WW and DD, respectively.

The calculated adsorption constants K_F of the Freundlich isotherms ranged from 87.7 to 252.5 (mean: 145.2) for the tested soils. The Freundlich exponents 1/n ranged from 0.8358 to 0.8778 (mean: 0.8534), indicating that the concentration of the test item affected the adsorption behavior in the examined concentration range.

In general, the organic matter in soil determined as organic carbon content is the most important component responsible for binding organic chemicals. Therefore, the adsorption coefficients K_F were correlated with the organic carbon content of the soil to get a comparability of the adsorption behavior in different soils. For aclonifen, the K_{Foc} values ranged from 4139.9 to 6480.4 mL/g (geometric mean 5189.8 mL/g).

At the end of the adsorption and desorption 11.7 to 23.5%, 10.3 to 20.8%, 8.3 to 14.4%, 12.2 to 22.5% and 5.9 to 10.1% of the initially adsorbed amount were desorbed in soil AX, HH, HN, WW and DD, respectively.

The desorption $K_{F(des)}$ and the normalized $K_{Foc(des)}$ values were 1.3 to 1.5 times higher than those obtained for adsorption, indicating that aclonifen once adsorbed to soil is not readily desorbed.

There is no significant correlation between pH and adsorption for the investigated soils.

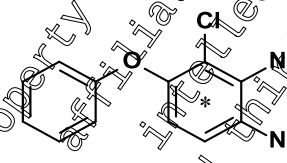
According to Briggs, aclonifen can be classified as immobile.

Soil	Texture (USDA)	pH *	OC [%]	Clay [%]	K_F [mL/g]	K_{Foc} [mL/g]	1/n
[redacted] Hof AXXa	Sandy loam	5.8	1.7	8	87.7	5156.9	0.8358
[redacted] am [redacted]	Silt loam	6.2	2.0	16	111.9	5594.8	0.8522
[redacted] Hof	Loam	5.6	2.8	13	181.5	6480.4	0.8778
[redacted] Hof Wurmwiese	Sandy loam	5.0	1.9	15	92.4	4863.8	0.8400
[redacted] II	Loam	7.0	6.1	26	252.5	4139.9	0.8615
Arithmetic mean					**145.2**	**5247.2**	**0.8535**
Geometric mean					**133.0**	**5189.8**	**0.8533**

* pH values were derived from aqueous 0.01 M $CaCl_2$ suspensions (soil characterization data)

I. MATERIALS AND METHODS

1. Test Material: [aniline-UL-^{14}C]-aclonifen



* indicates position of ^{14}C radiolabel

Chemical name (IUPAC): 2-chloro-6-nitro-3-phenoxy-aniline

Lot or batch number: KML 10004

Specific radioactivity: 6.59 MBq/mg (178.11 µCi/mg)

Radiochemical purity: >99%

CA registry number: Not stated

Stability of test compound: Not specified. The radiochemical purity of the test item was determined by HPLC/radio-detection prior to applications.

Application vehicle: Calcium chloride

2. Soils Sorption tests were performed with five soils covering a range of pH, organic carbon content and texture. The characteristics of the European soils are summarised below.

Table 7.1.3- 10: Physico-chemical characteristics of the soils

Parameter	Results/Units				
Soil Designation	[redacted] Hof AXXa	[redacted] am [redacted]	[redacted] Hof	[redacted] Hof Wurmwiese	[redacted] II
Code	AX	HH	HN	WW	DD
Soil Taxonomic Classification (USDA)	Sandy, mixed, mesic Typic Cambudoll	Loamy, mixed, mesic Typic Argudalf	Loamy-skeletal, mixed, semiactive, mesic Dystric Eutrudept	loamy, mixed, mesic Typic Argudalf	Fine-loamy, mixed, active, frigid Typic Eutrudept
Textural Class (USDA)	sandy loam	silt loam	loam	Sandy loam	loam
Sand [50 µm – 2 mm]	75%	18%	42%	55%	31%

Parameter	Results/Units				
Soil Designation	[redacted] Hof AXXa	[redacted] am [redacted]	[redacted] Hof	[redacted] Hof Wurmwiese	[redacted] II
Silt [2 µm – 50 µm]	17%	66%	45%	30%	43%
Clay [< 2 µm]	8%	16%	13%	18%	28%
pH (M $CaCl_2$)	5.8	6.2	5.6	5.0	7.0
pH (soil/water 1/1)	6.1	6.4	5.8	5.3	7.2
pH (saturated paste)	6.0	6.4	5.8	5.3	7.1
pH (soil/1 N KCl 1/1)	5.6	5.8	5.3	4.6	6.8
Organic Carbon (combustion)	1.7%	2.0%	2.8%	1.9%	6.1%
Organic Matter [a]	2.9%	3.4%	4.8%	3.3%	10.5%
Cation Exchange Capacity (meq/100 g)	8.4	1011.4	10.8	10.7	20.6
Water Holding Capacity maximum (MWHC)	50.3 g H_2O *ad* 100 g DW	51.9 g H_2O *ad* 100 g DW	64.4 g H_2O *ad* 100 g DW	60.1 g H_2O *ad* 100 g DW	98.0 g H_2O *ad* 100 g DW
at 1/10 bar (pF 2.0)	14.5%	31.9%	30.1%	25.5%	39.3%
Bulk Density (disturbed)	1.2 g/cm³	1.1 g/cm³	1.04 g/cm³	1.14 g/cm³	0.89 g/cm³

[a] % organic matter = % organic carbon x 1.724

B. STUDY DESIGN AND METHODS

1. Experimental dates:

7 Sept 2015 – 28 April 2016

2. Experimental design

The stability in aqueous solution, adsorption to test vessels, equilibration time and parental mass were determined in preliminary testing. The stability of aclonifen was confirmed in 0.01M calcium chloride solution over 96 hours. The potential for adsorption of aclonifen to the test vessels was tested over 96 hours and was found not to absorb to the test vessels. Teflon® centrifuge tubes (e.g. 42 mL) with Teflon® screw caps were used for all tests in this study.

The stability of aclonifen in calcium chloride was assessed over 96 hours at 20°C. After incubation for 96 hours the test item was detected with ≥ 91.0% of the applied radioactivity. Soil to solution ratios of 1:80 were regarded as adequate. The radioactivity adsorbed to soil was between 74.4 and 88% AR.

In the definitive test, with an equilibration time of 24 hours, the percentage adsorbed to soil ranged from 67.0 – 93.0%. The aqueous supernatant after adsorption and desorption was separated by centrifugation and the amount of test item in the supernatants was analysed by liquid scintillation counting (LSC). Generally, radio-HPLC was used to analyse the supernatants and soil extracts immediately after sampling. However, supernatants of the adsorption and desorption phase (definite test) as well as the application controls were remeasured by LSC after defrosting 142 to 146 days after the first measurement. This was done since LSC results for the lowest test concentration E were between the instrumental limit of quantitation (LOQ) and the instrumental limit of detection (LOD).

HPLC hyphenated to electrospray ionization mass spectrometry in single or multistage mode (ESI-MS(/MS)) and radio-detection was used for confirmation of the test item identity. After analysis, supernatants and soil extracts were stored at ≤ -18°C in the dark. The soils were air-dried and residues were determined by combustion/LSC to establish the material balance. The sorption parameters were calculated using Freundlich isotherms.

Adsorption phase

Parameter	Description

Soil Condition		Soils were gently air-dried, sieved to ≤ 2 mm, sterilized by gamma-irradiation and equilibrated to study conditions for at least over night with 38 mL aqueous 0.01 M $CaCl^2$ solution (corrected for soil moisture).
Have these soils been used for other laboratory studies?		The soils have been used in several degradation and sorption studies.
Soil sample weight		0.5 g dry weight equivalents per replicate.
Equilibrium solution used		aqueous 0.01 M $CaCl_2$ solution.
Control used		N/A
Test item concentrations	Nominal application rates	Nominal concentrations in test solution: 0.00051, 0.0026, 0.0055, 0.028 and 0.060 mg/L
	Analytically measured concentrations	Concentrations in test solution: 0.00049, 0.0025, 0.0050, 0.026 and 0.051 mg/L
Identity and concentration of co-solvent		N/A
Soil-to-solution ratio		1/80, i.e. 0.5 g soil dry weight equivalents to 40 mL solution (corrected for soil moisture).
pH of the equilibration solution	Initial	pH of aqueous 0.01 M CaCl2 solution without soil: 6.1
	Final	pH with soil and test item after adsorption equilibrium: range 6.4 – 7.3
Number of replications	Controls	N/A
	Treatments	Duplicate
Equilibration	Time	Equilibration with untreated 0.01 M $CaCl_2$ for 1 day
	Temperature	20.3°C
	Dark	Yes
	Shaking method	Mechanical overhead shaker, 30 ± 2 rpm
	Shaking time	24 hours
Method of separation of supernatant		Centrifugation
Centrifugation	Speed	10000 x g
	Duration	10 min
	Method of separation of soil and solution	Supernatant was carefully decanted.

Desorption phase

Parameter		Description
Were the soil residues from the adsorption phase used?		Yes
Amount of test item present in the adsorbed state / adsorbed amount		The amounts of test item adsorbed to soil after adsorption ranged from 67.0 to 93.0% AR.
Number of desorption cycles		One desorption cycle was performed for each concentration.
Equilibrium solution and quantity used per treatment for desorption		The decanted solution was replaced by fresh aqueous 0.01 M $CaCl_2$ solution. A total volume of 40 mL was used as equilibration solution.
Soil-to-solution ratio		1/80, i.e. 0.5 g soil dry weight equivalents to 40 mL solution (corrected for soil moisture).
Number of replications	Controls	N/A
	Treatments	Duplicate
Desorption equilibration	Temperature	20.3 °C
	Dark	Yes
	Shaking method	Mechanical overhead shaker, 30 ± 2 rpm
	Shaking time	24 hours
Method of separation of supernatant		Centrifugation
Centrifugation	Speed	10000 x *g*
	Duration	10 min
	Method of separation of soil and solution	Supernatant was carefully decanted.

Analytical procedures

The purity of the test item in the application solution was determined by radio-HPLC in the preliminary tests. The parental mass balance was established in the preliminary test. Supernatants were analysed by LSC. The soil was extracted three times at ambient temperature by shaking for 30 minutes each and once by microwave-assisted extraction at 70 °C for 10 minutes using 16 mL acetonitrile / water 4/1 (v/v). Afterwards, the soil was microwave-extracted a second time at 50°C using 16 mL methanol / water 1/1 (v/v) for 10 minutes. After each extraction step, extract and soil were separated by centrifugation and decantation. The supernatants (adsorption and desorption) and soil extracts were combined and the volumes determined. The radioactivity contents were determined by LSC and the stability of the test item was determined by HPLC/radio-detection to establish the parental mass balance.

The material balance was determined in the definitive test. Adsorption and desorption solutions were analysed by LSC, soils were air-dried and residues were determined by combustion/LSC to establish the material balance.

II. RESULTS AND DISCUSSION

A. Recovery of test item in preliminary test

Distribution of test item in combined solution and soil extracts, expressed as percentage of applied radioactivity (% AR) (mean) are presented in the table below.

Table 7.1.3- 11: Preliminary test: Total recovery of [U-^{14}C-aniline]-aclonifen in solution samples and soil extracts

Compartment	Soil ID					Mean
	AX	HH	HN	WW	DD	
Radioactivity in solution [% AR]	26.2	22.5	15.8	23.6	10.9	19.8
Test item in solution [% ROI] *	100.0	100.0	100.0	100.0	100.0	100.0
Test item in solution [% AR]	**26.2**	**22.5**	**15.8**	**23.6**	**10.9**	**19.8**
Radioactivity in soil extracts [% AR]	68.7	73.9	82.0	76.4	87.8	77.8
Test item in soil extracts [% ROI] *	100.0	100.0	100.0	100.0	100.0	100.0
Test item in soil extracts [% AR]	**68.7**	**73.9**	**82.0**	**76.4**	**87.8**	**77.8**
Non-extractable residues [% AR]	N/A	N/A	N/A	N/A	N/A	N/A
Total recovery of radioactivity [% AR]	94.9	96.5	97.8	100.0	98.8	97.6
Total recovery of test item [% AR]	**94.9**	**96.5**	**97.8**	**100.0**	**98.8**	**97.6**

*: percentage of regions of interest [% ROI] from primary chromatographic method
N/A: not applicable

B. Transformation of test substance:

The stability of test item and adsorption of test item to test vessel surface were determined prior to the definitive test in preliminary tests. Supernatants, controls as well as supernatants and soil extracts were analysed by LSC and HPLC-radio-detection immediately after sampling. Therefore, no storage stability investigations were necessary. However, supernatants of the adsorption and desorption phase (definite test) as well as the application controls were remeasured by LSC after defrosting 142 to 146 days after the first measurement. This was done since LSC results for the lowest test concentration E were between the instrumental limit of quantitation (LOQ) and the instrumental limit of detection (LOD). The stability of the test item (control with no soil) was confirmed by radio-HPLC to be ≥ 91.1% AR over the duration of the test.

C. Findings:

Within definitive tests, the amount of [^{14}C]aclonifen recovered from the test system (adsorption and desorption supernatants and soil residue combustions) ranged from 95.7 to 110.0% AR for soil AX, 104.4 to 110.7% AR for soil HH, 104.5 to 110.2% AR for soil HN, 103.9 to 109.2% for soil WW and 106.4 to 113.0% AR for soil DD (see table below).

Table 7.1.3- 12: Definitive test: Total recovery of [U-^{14}C-aniline]-aclonifen in solution samples and soil extracts

Soil ID	AX	HH	HN	WW	DD
* Conc. ID	[% AR]	[% AR]	[% AR]	[% AR]	[% AR]
A	104.5	105.8	104.5	106.1	108.5
A	102.8	105.9	105.6	106.7	107.4
B	95.7	109.6	110.2	108.4	113.0
B	109.2	110.7	109.8	109.2	110.6
C	104.9	105.7	107.0	103.9	109.3
C	105.2	104.0	108.0	105.6	109.3
D	105.7	104.3	106.9	104.8	106.8
D	105.0	106.0	106.1	106.1	106.4
E	105.8	107.3	107.7	108.3	110.2
E	110.0	105.0	108.3	106.1	111.1
Mean	104.9	106.4	107.4	106.5	109.2
SD	± 3.7	± 2.1	± 1.7	± 1.6	± 2.0

*A - 0.051, B – 0.026, C - 0.005, D - 0.0025 & E – 0.00049 mg/L
SD = standard deviation

The definitive test was performed at a soil-to-solution ratio of 1:80 for all soils. Equilibrium of the test item was established after 24 hours shaking for all soils. All five soils had a desorption step of 24 hours. The adsorption behaviour of [^{14}C]-aclonifen was accurately measured using a nominal concentration range of 0.0005 to 0.05 mg/L by the Freundlich equation for all soils. Concentrations of [^{14}C]-aclonifen in aqueous and solid phase following the adsorption step are shown in the table below.

Table 7.1.3- 13: Definitive test: Concentration of [U-^{14}C-aniline]-aclonifen in aqueous and solid phase following 24 hours of adsorption

	Concentration (µg/mL)	Soil (µg/g)	Solution (µg/mL)
Conc. ID	**Soil ID**	**AX Sandy Loam**	
A	0.0505529	2.7113375	0.0166612
B	0.0258971	1.4816342	0.0073767
C	0.0049898	0.309625	0.0011194
D	0.0024724	0.1585432	0.0004906
E	0.0004861	0.0322307	0.0000832
	Soil ID	**HH Silt Loam**	
A	0.0505529	2.9047735	0.0142432
B	0.0258971	1.5473965	0.0065546
C	0.0049898	0.318062	0.001014
D	0.0024724	0.1628355	0.0004369
E	0.0004861	0.032829	0.0000758
	Soil ID	**HN Loam**	
A	0.0505529	3.2503707	0.0099233
B	0.0258971	1.6630349	0.0051091
C	0.0049898	0.3408289	0.0007331
D	0.0024724	0.1692781	0.0003564
E	0.0004861	0.0342163	0.0000584
	Soil ID	**WW Sandy Loam**	
A	0.0505529	2.8449772	0.0149907
B	0.0258971	1.4636766	0.0076011
C	0.0049898	0.3068762	0.0011538
D	0.0024724	0.1579536	0.0004979
E	0.0004861	0.0327076	0.0000773
	Soil ID	**DD Clay Loam**	
A	0.0505529	3.4978192	0.0068302
B	0.0258971	1.8054238	0.0033293
C	0.0049898	0.3609928	0.0004773
D	0.0024724	0.179354	0.0002304
E	0.0004861	0.03615	0.0000343

The adsorption constants K_f of the Freundlich isotherms ranged from 87.7 to 252.5 mL/g for the tested soils with associated Freundlich exponents 1/n ranging from 0.836 to 0.878. The corresponding correlation coefficients for the adsorption isotherms ranged from 0.9986 to 0.9995 indicating a linear fit to the measured data. When normalized for the organic carbon content of soil K_{foc} values ranged

from 4140 to 6480 mL/g. The Freundlich adsorption and desorption constants for aclonifen in soil are summarised below.

Table 7.1.3- 14: Adsorption constants of [U-^{14}C-aniline]-aclonifen in soil

Soil	Soil ID	Texture (USDA)	pH *	OC [%]	Clay [%]	K_F [mL/g]	K_{Foc} [mL/g]	1/n
[redacted] Hof AXXa	AX	Sandy loam	5.8	1.7	8	87.7	5156.9	0.8358
[redacted] am [redacted]	HH	Silt loam	6.2	2.0	16	111.9	5594.8	0.8522
[redacted] Hof	HN	Loam	5.6	2.8	15	181.5	6480.4	0.8578
[redacted] Hof Wurmwiese	WW	Sandy loam	5.0	1.9	15	92.4	4863.8	0.8400
[redacted] II	DD	Loam	7.0	6.1	26	252.5	4139.9	0.8615
Arithmetic mean						**145.2**	**5247.2**	**0.8535**
Geometric mean						**133.0**	**5189.8**	**0.8533**

* pH values were derived from aqueous 0.01 M $CaCl_2$ suspensions (soil characterization data)

The desorption behavior of aclonifen in the concentration range of two orders of magnitude was accurately described for all soils with the Freundlich equation (results shown in table below). The correlation coefficients of the individual isotherms ranged from 0.9985 to 0.9993. The calculated desorption constants $K_{F(des)}$ of the Freundlich isotherms ranged from 122.0 to 368.3 for the tested soils. The Freundlich exponents 1/n ranged from 0.8358 to 0.8784. The $K_{Foc(des)}$ values ranged from 6037.8 to 8493.2 mL/g. The $K_{Foc(des)}$ values were 1.3 to 1.5 times higher than the K_{Foc} values, indicating a strengthened binding of aclonifen once adsorbed to soil.

Table 7.1.3- 15: Summary of Freundlich desorption constants K_f and K_{foc} values

Desorption	Soil	AX	HH	HN	WW	DD
	Textural class	Sandy Loam	Silt Loam	Loam	Sandy Loam	Clay Loam
Cycle 1	$K_{f\,des}$	122	143.3	267.8	124.5	368.3
	1/n	0.8358	0.8399	0.8699	0.8435	0.8784
	R^2	0.9985	0.9993	0.9988	0.9985	0.9993
	$K_{foc\,des}$	7174.6	7162.5	8493.2	6551.6	6037.8

D. OECD 106 evaluation checklist:

Relevant quality checks were performed to evaluate the acceptability of the study according to the EFSA Technical Report on the Outcome of Pesticides Peer Review Meeting on the OECD 106 Evaluators Checklist (2017). These checks confirmed that the mass balance of 104.9-109.2% (mass balances 90-110%) and % adsorption of 68-100% were all acceptable (>20%). The LSC analytical method was considered acceptable over the range of concentrations measured. The lowest concentration tested was 29 times higher than the LOQ and the lowest measured value (1.2 Bq) was above the LOQ of the LSC (0.8 Bq). The use of the indirect method was appropriate based on a Kd * soil/solution ratio > 0.3 in all soils. Based upon this K_{fE} / K_f ratios were determined to be < 1.2 in all five soils. Confidence intervals for $_{ads}K_f$ and $_{ads}1/n$ did meet the quality criteria (confidence intervals should be narrow).

The graphical fits of the Freundlich equation based on the standard linear regression form using log-log transformed data alongside the associated residual plots were evaluated and found to be acceptable (see table below). The R^2 of the standard linear regressions ranged from 0.999 to 1.000. The visual fit of both the standard regression and the residual plots were acceptable.

The evaluation confirmed the study was acceptable according to all quality checks and thus it can be concluded the study is to be used in regulatory modelling. The results of the evaluation are summarised in the tables below.

Table 7.1.3- 16: Results of OECD 106 evaluation checklist

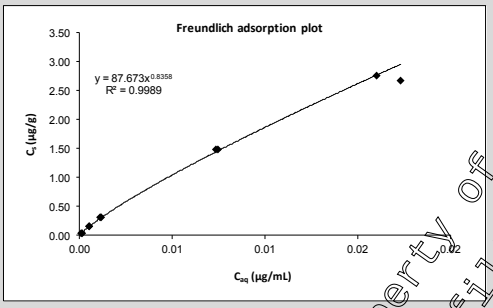
Soil	Units	Quality criteria	Sandy Loam (AX) [redacted]	Silt Loam (HH) [redacted]	Loam (HN) [redacted]	Sandy Loam (WW) Wurmwiese	Clay Loam (DD) [redacted]
Adsorption method (direct/indirect)	-	-	Indirect	Indirect	Indirect	Indirect	Indirect
Soil : solution ratio	g/mL	-	80:1	80:1	80:1	80:1	80:1
Mass balance of ^{14}C (at all tested concentrations)	%	90-110	104.9	106.4	107.4	106.5	109.2
Adsorbed percentage (δ)	%	>20%#	65.78-83.01	71.00-84.12	79.25-88.36	69.16-84.39	86.47-92.94
K_d x (soil : solution ratio) ##		>0.3#	1.92-4.89	2.45-5.53	3.82-7.59	2.24-5.41	6.39-13.17
K_{fE} / K_f	-	<1.2#	≤1.059	≤1.090	≤1.068	≤1.102	≤1.101
$^{ads}K_F$ (95% confidence interval)	L/kg	*	87.673 (74.9-102.6)	111.933 (94.8-132.2)	181.523 (149.3-220.7)	92.429 (81.5-104.9)	252.843 (224.3-284.3)
$^{ads}1/n$ (95% confidence interval)	-	*	0.836 (0.813-0.859)	0.852 (0.828-0.876)	0.878 (0.851-0.905)	0.840 (0.822-0.858)	0.862 (0.846-0.877)
$^{Ads}R^2$	-	>0.975	0.999	0.999	0.999	0.999	1.000
$^{ads}K_{F,OC}$	L/kg	-	5157.2	5596.7	6483.0	4864.7	4145.0
Visual fit to Freundlich isotherm		**	Acceptable	Acceptable	Acceptable	Acceptable	Good
Residual plots randomly distributed		**	Good	Good	Good	Good	Good

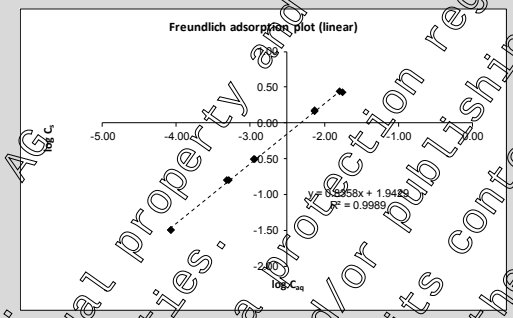
* Confidence intervals should be narrow.
#F factor - calculated using material balance data where F = 100 - (^{14}C aquatic [%] + ^{14}C extract [%]). As all material balances were >100% this resulted in negative values for F, these were used as positive values. The F value was not corrected for % parent in recovered radioactivity, preliminary tests resulted in 100% parent attributed to radioactivity.
** Note on assessing visual fits: Poor - an unacceptable fit, the fitted curve does not represent the trend of the data points and residuals show strong deviations from random distribution. Acceptable – the fitted curve describes the trend of the data points, residuals may show some deviation from random distribution but it is not significant. Good – the fitted curve closely follows all the data points, residuals are randomly distributed.
OCED 106 Calculation Tool V2 used

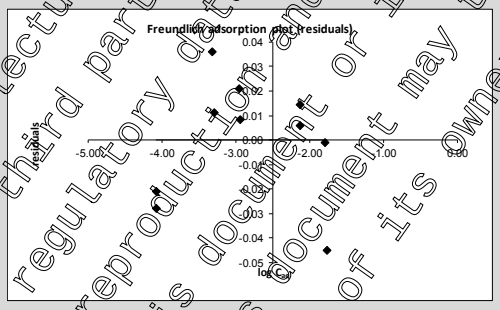
Freundlich plots from OECD 106 Checklist

Sandy loam soil (AX)

Linear plot parameters and derived adsorption values			
Slope =	0.8358	$logK_F$ =	1.9429
Intercept =	1.9429	K_F =	87.673
Correlation coefficient (r) =	0.9994	K_{Foc} =	5157.2
Coefficent of determination (r^2) =	0.9989	1/n =	0.8358

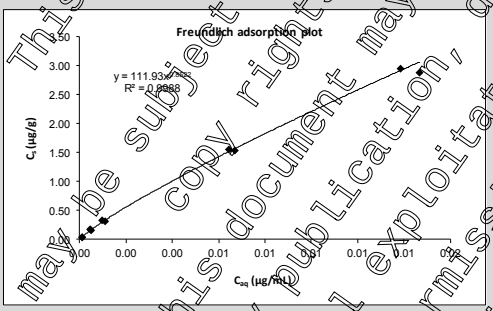


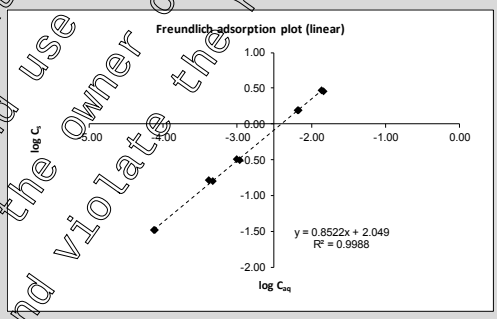


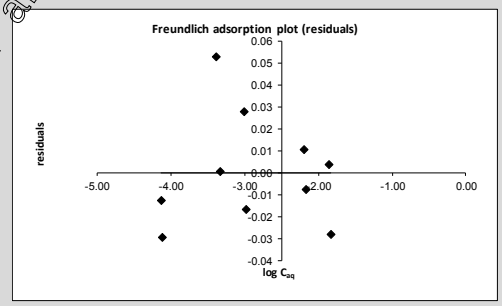


Silt loam soil (HH)

Linear plot parameters and derived adsorption values			
Slope =	0.8522	$logK_F$ =	2.0490
Intercept =	2.0490	K_F =	111.933
Correlation coefficient (r) =	0.9994	K_{Foc} =	55907
Coefficent of determination (r^2) =	0.9988	1/n =	0.8522

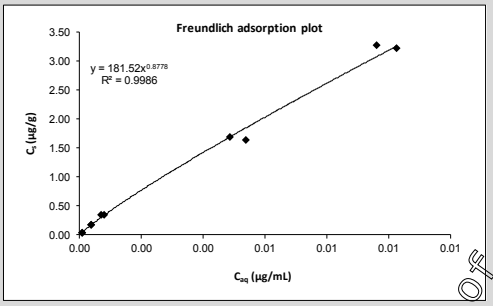


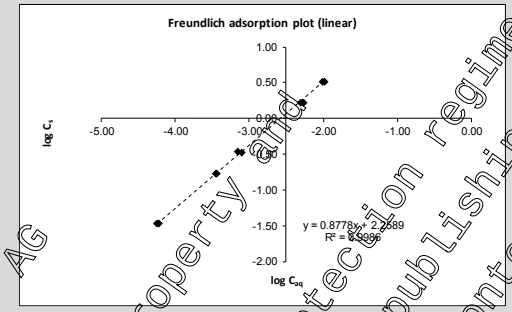


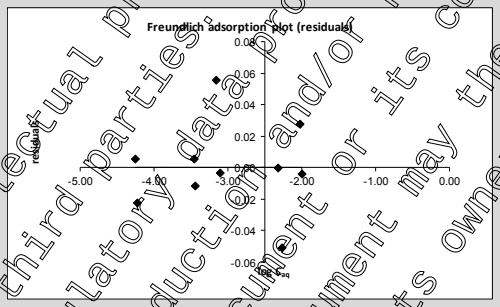


Loam soil (HN)

Linear plot parameters and derived adsorption values			
Slope =	0.8778	$logK_F$ =	2.2589
Intercept =	2.2589	K_F =	181.523
Correlation coefficient (r) =	0.9993	K_{Foc} =	6483.0
Coefficent of determination (r^2) =	0.9986	1/n =	0.8778

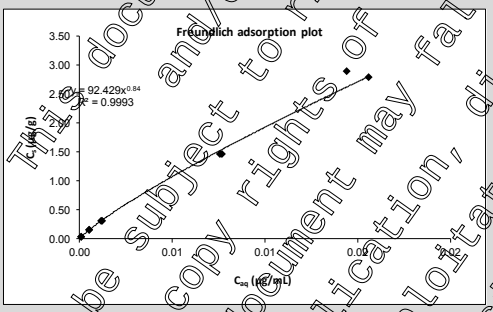


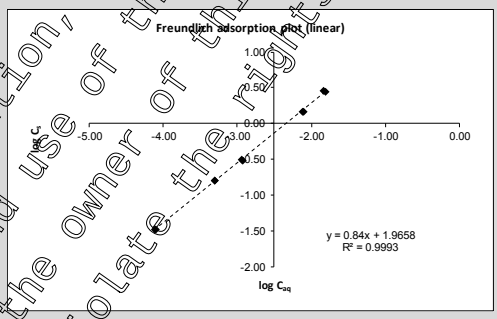


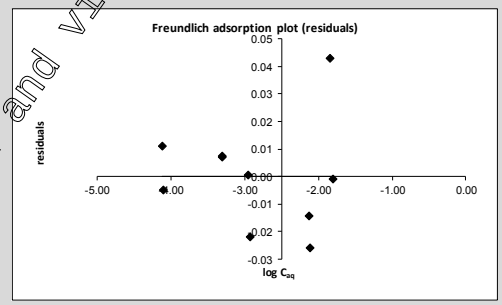


Sandy loam soil (WW)

Linear plot parameters and derived adsorption values			
Slope =	0.8400	$logK_F$ =	1.9658
Intercept =	1.9658	K_F =	92.429
Correlation coefficient (r) =	0.9996	K_{Foc} =	4864.7
Coefficent of determination (r^2) =	0.9993	1/n =	0.8400

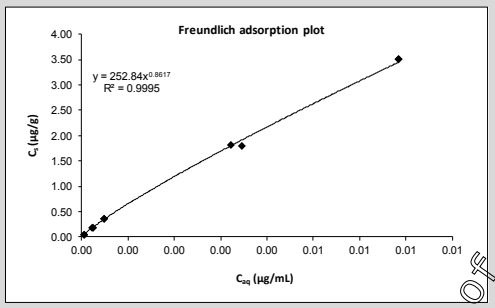


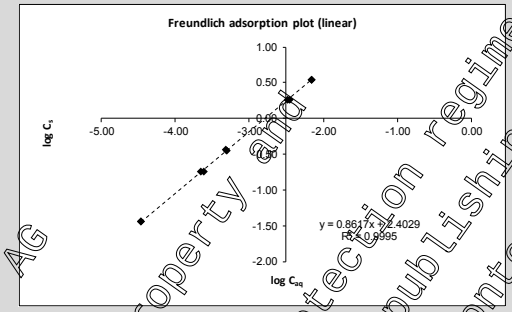




Clay loam soil (DD)

Linear plot parameters and derived adsorption values			
Slope =	0.8617	$logK_F$ =	2.4029
Intercept =	2.4029	K_F =	252.843
Correlation coefficient (r) =	0.9998	K_{Foc} =	4145.0
Coefficent of determination (r^2) =	0.9995	1/n =	0.8617





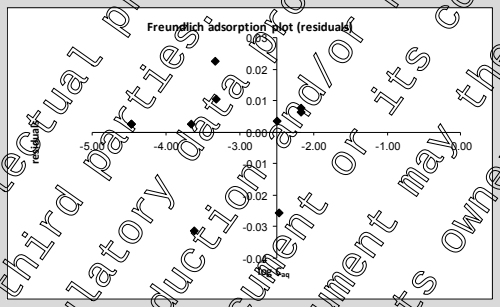


Table 7.1.3- 17: Summary of Quality Criteria and Regulatory Interpretation

Aclonifen		Quality Criteria			Overall Conclusion	Regulatory Interpretation
Soil Type	Soil Name	Met	Partially Met	Not Met		
Sandy Loam	AX	9	0	0	Met	Acceptable
Silt Loam	HH	9	0	0	Met	Acceptable
Loam	HN	9	0	0	Met	Acceptable
Sandy Loam	WW	9	0	0	Met	Acceptable
Clay Loam	DD	9	0	0	Met	Acceptable

Table 7.1.3- 18: Impact on Endpoints

Soil Type	Soil Name	K_{foc} (Reported)	K_{foc} (OECD tool)	1/n (Reported)	1/n (OECD tool)
Sandy Loam	AX	5156.9	5157.2	0.836	0.836
Silt Loam	HH	5594.8	5596.7	0.852	0.852
Loam	HN	6480.4	6483.0	0.878	0.878
Sandy Loam	WW	4863.8	4867.7	0.840	0.840
Clay Loam	DD	4139.9	4145.0	0.862	0.862
Arithmetic Mean		-	-	0.854	0.854
Geometric Mean		5189.8	5192.1	-	-
pH dependence		No	No	-	-

The small differences between the reported values and the OECD calculation tool (v2) are considered to be due to rounding in the calculations. The reported values have been used in modelling calculations.

III. CONCLUSION

The adsorption constants K_F of aclonifen ranged from 87.7 to 252.5 mL/g (geometric mean: 133.0 mL/g). The K_{Foc} values ranged from 4139.9 to 6480.4 mL/g (geometric mean: 5189.8 mL/g).

Depending on the soil, the desorption constants $K_{F(des)}$ of aclonifen were about 1.3 to 1.5 times higher than the respective adsorption constants, indicating a strengthened binding of aclonifen once adsorbed to soils representing conditions relevant for the environment.

There was no significant correlation between pH and adsorption for the investigated soils.

Aclonifen was stable during the test. The parental mass balance was ≥ 94.9% AR. No major degradation product was observed.

These results indicate that aclonifen is strongly absorbed to soil and is predicted to be immobile in soil according to the Briggs classifications.

The OECD 106 Checklist (v2) was used to evaluate the study. All 5 soils can be considered "met" with regard to the quality criteria and acceptable for regulatory use.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 106 (2000) and is considered valid to assess the adsorption and desorption characteristics of aclonifen in soil.

Assessment and conclusion by RMS:

CA 7.1.3.1.2 Adsorption and desorption of metabolites, breakdown and reaction products

The adsorption and desorption of the aclonifen metabolite M-01 (called BCS-AX74959 in the report) has been investigated (KCA 7.1.3.1.2/01).

Report reference	Author, Year	Comment
KCA 7.1.3.1.2/01 M-629733-01-1	[redacted] & [redacted] 2018	New data not yet reviewed under UP

This is a new study for Annex I renewal submission. The results are summarised below.

Report reference	Soil	Texture	pH	OC [%]	K_f	K_{OC}	1/n
KCA 7.1.3.1.2/01 M-629733-01-1	[redacted] Hof AXXa	Sandy loam	5.7	1.9	101.8	5359	0.8424
	[redacted]am [redacted]a	Silt loam	6.2	1.9	106.4	5601	0.8380
	[redacted]Hof	Loam	5.7	2.5	151.6	6062	0.8094
	[redacted] II	Clay loam	7.3	5.2	154.4	2969	0.8961
Arithmetic mean					128.5	4998	0.8465
Geometric mean					126.2	4821	0.8459

Data Point:	KCA 7.1.3.1.2/01
Report Author:	[redacted]; [redacted]
Report Year:	2018
Report Title:	[phenol-UL-14C]BCS-AX74959: Adsorption / Desorption on four soils
Report No:	EnSa-17-0366
Document No:	M-629733-01-1
Guideline(s) followed in study:	OECD Test Guideline No 106 (2000) US EPA OCSPP Test Guideline No 835.1230 (2008) Commission Regulation (EU) No 283/2013 in accordance with Regulation (EC) No 1107/2009 (2009 & 2013)
Deviations from current test guideline:	Current guideline: OECD 106 (2000) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The adsorption / desorption behaviour of [phenol-UL-^{14}C] M-01 (called BCS-AX74959 in the report) was studied in four soils in batch equilibrium experiments in the laboratory in the dark at 20 ± 2 °C. The soil characteristics were as follows:

Soil	Soil ID	Source	Texture (USDA)	pH *	OC [%]
[redacted] Hof AXXa	AX	[redacted], Germany	sandy loam	5.7	1.9
[redacted] am [redacted] 4a	HH	[redacted], Germany	silt loam	6.2	1.9
[redacted] Hof	HN	[redacted], Germany	loam	5.7	2.5
[redacted] II	DD	[redacted], Germany	clay loam	7.3	5.2

* pH values were derived from aqueous 0.01 M $CaCl_2$ suspensions

The adsorption phase of the study was carried out using air-dried gamma irradiated soils equilibrated in aqueous 0.01 M $CaCl_2$ solution with soil-to-solution ratios of 1/40 (soils AX and HH) and 1/100 (soils HN and DD). Test concentrations of approx. 0.52, 0.15, 0.05, 0.016 and 0.005 mg/L of M-01 were applied in aqueous 0.01 M $CaCl_2$ solution. The desorption phase was performed by supplying pre-adsorbed soil samples with fresh aqueous 0.01 M $CaCl_2$ solution. Adsorption took place for 24 hours (soils AX, HH and HN) and 2 hours (soil DD). Desorption took place for 2 hours for all soils. The test was performed in centrifuge tubes with screw caps.

The aqueous supernatant after adsorption and desorption was separated by centrifugation and the amount of test item in the supernatants was analysed by liquid scintillation counting (LSC). After the desorption step, the soil was dried and the residues were determined by combustion/LSC. The sorption parameters were calculated using Freundlich isotherms.

The test item was sufficiently stable throughout the study. Mean parental mass balances were 92.7, 91.9, 98.9 and 91.2% AR for soil AX, HH, HN and DD, respectively.

Mean material balances were 98.1% AR for soil AX (range from 95.5 to 101.6% AR), 98.3% AR for soil HH (range from 96.3 to 102.7% AR), 94.3% AR for soil HN (range from 91.7 to 96.4% AR) and 97.0% AR for soil DD (range from 92.7 to 105.5% AR).

In the definitive adsorption test 77.2 – 88.5% AR, 78.4 – 89.5% AR, 67.3 – 85.0% AR and 62.5 – 74.1% AR were adsorbed in soil AX, HH, HN and DD, respectively.

The calculated adsorption constants K_F of the Freundlich isotherms ranged from 101.817 to 154.386 mL/g (mean: 128.543 mL/g) for the tested soils. The Freundlich exponents 1/n ranged from 0.8094 to 0.8961 (mean: 0.8465), indicating that the concentration of the test item affected the adsorption behaviour in the examined concentration range.

In general the organic matter in soil, determined as organic carbon content, is the most important component responsible for binding organic chemicals. Therefore, the adsorption coefficients K_F were correlated with the organic carbon content of the soil to get a comparability of the adsorption behaviour in different soils. For M-01 the K_{Foc} values ranged from 2969 to 6062 mL/g (geometric mean: 4821 mL/g).

At the end of the adsorption and desorption, 6.9 to 16.7%, 5.9 to 16.3%, 9.1 to 23.2% and 11.8 to 20.0% of the initially adsorbed amount were desorbed in soil AX, HH, HN and DD, respectively.

The desorption $K_{F(des)}$ and the normalized $K_{Foc(des)}$ values were significantly higher (up to 2 times higher) than those obtained for adsorption, indicating that M-01 once adsorbed to soil is not readily desorbed.

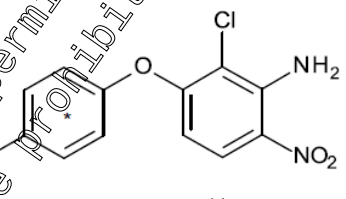
According to Briggs, M-01 can be classified as immobile for adsorption and for desorption.

Soil	Texture (USDA)	pH*	OC [%]	Clay [%]	K_F [mL/g]	K_{Foc} [mL/g]	1/n
[redacted] Hof AXXa	Sandy loam	5.7	1.9	8	101.8	5359	0.8424
[redacted] am [redacted] 4a	Silt loam	6.2	1.9	16	106.4	5601	0.8380
[redacted] Hof	Loam	5.7	2.5	18	151.6	6062	0.8094
[redacted] II	Clay loam	7.3	5.2	28	154.4	2969	0.8961
Arithmetic mean					**128.6**	**4998**	**0.8465**
Geometric mean					**126.2**	**4821**	**0.8459**

* pH values were derived from aqueous 0.01 M $CaCl_2$ suspensions (soil characterization data)

I. MATERIALS AND METHODS

1. Test Material: [phenol-UL-^{14}C] BCS-AX74959 (called M-01 in this dossier)



* indicates position of ^{14}C radiolabel

Chemical name (IUPAC): 4-(3-amino-2-chloro-4-nitro-phenoxy)phenol

Lot or batch number: KML 10307

Specific radioactivity: 6.08 MBq/mg

Radiochemical purity: 99%

CA registry number: Not stated

Stability of test compound: Stable, determined within study

Application vehicle: Calcium chloride

2. Soils Sorption tests were performed with four soils covering a range of pH, organic carbon content and texture. The characteristics of the European soils are summarised below.

Table 7.1.3- 19: Physico-chemical characteristics of the soils

Parameter	Results/Units			
Soil Designation	[redacted] Hof AXXa	[redacted] am 4a	[redacted] Hof	[redacted] II
Soil Taxonomic Classification (USDA)	Sandy, mixed, active, nonacid, mesic Inceptic Hapludalf	Coarse-silty, mixed, active, nonacid, mesic Typic Dystrudept	Loamy-skeletal, mixed, semiactive, mesic Dystric Eutrudept	Fine-loamy, mixed, active, frigid Typic Eutrudept
Textural Class (USDA)	sandy loam	silt loam	loam	clay loam
Sand [50 µm – 2 mm]	77%	21%	33%	33%
Silt [2 µm – 50 µm]	15%	63%	49%	39%
Clay [< 2 µm]	8%	16%	18%	28%
pH ($CaCl_2$)	5.7	6.2	5.7	7.3
pH (soil/water 1/1)	6.0	6.4	6.0	7.5
pH (saturated paste)	5.7	6.2	5.8	7.3
pH (soil/1 N KCl 1/1)	5.4	5.8	5.3	7.0
Organic Carbon (combustion)	1.9%	1.9%	2.5%	5.2%
Organic Matter [a]	3.3%	3.3%	4.3%	8.9%
Cation Exchange Capacity	8.1 meq/100 g	10.8 meq/100 g	9.1 meq/100 g	19.3 meq/100 g
Water Holding Capacity maximum (MWHC)	53.2 g H_2O *ad* 100 g DW	56.0 g H_2O *ad* 100 g DW	65.4 g H_2O *ad* 100 g DW	90.6 g H_2O *ad* 100 g DW
at 1/10 bar (pF 2.0)	14.6%	36.7%	28.9%	42.5%
Bulk Density (disturbed)	1.2 g/cm³	1.1 g/cm³	1.0 g/cm³	0.9 g/cm³

[a] % organic matter = % organic carbon x 1.724

B. STUDY DESIGN AND METHODS

1. Experimental dates:

10 Nov 2016 – 22 May 2018

2. Experimental design

The stability in aqueous solution, adsorption to test vessels, equilibration time and parental mass were determined in preliminary testing. The stability of M-01 was confirmed in 0.01M calcium chloride solution over 72 hours. The potential for adsorption of M-01 to the test vessels was tested over 24 hours and was found not to absorb to the test vessels. Teflon® centrifuge tubes (e.g. 42 mL) with Teflon® screw caps were used for all tests in this study.

The stability of M-01 in calcium chloride was assessed in the four soils over 72 hours at 20°C. After incubation for 72 hours the test item was detected with ≥ 97.2% of the applied radioactivity. Soil to solution ratios of 1:40 (soils AX and HH) and 1:100 (soils HN and DD) were regarded as adequate. The radioactivity adsorbed to soil was between 62.3 and 75.1% AR.

In the definitive test, with an equilibration time of 24 hours (soils AX, HH and HN) and 2 hours (Soil DD) the percentage adsorbed to soil ranged from 62.5 - 89.5%. The aqueous supernatant after

adsorption and desorption was separated by centrifugation and the amount of test item in the supernatants was analysed by liquid scintillation counting (LSC). Radio-HPLC was used to analyse the supernatants and soil extracts immediately after sampling. HPLC hyphenated to electrospray ionization mass spectrometry in single or multistage mode (ESI-MS(/MS)) and radio-detection was used for confirmation of the test item identity. After analysis, supernatants and soil extracts were stored at ≤ -18 °C in the dark. The soils were air-dried and residues were determined by combustion/LSC to establish the material balance. The sorption parameters were calculated using Freundlich isotherms.

Adsorption phase

Parameter		Description
Soil Condition		Soils were gently air-dried, sieved to < 2 mm, sterilized by gamma radiation and equilibrated to study conditions for 1 day with 20 mL aqueous 0.01 M $CaCl_2$ solution (corrected for soil moisture).
Have these soils been used for other laboratory studies?		The soils have been used in several degradation and sorption studies.
Soil sample weight		0.5 g (soils AX and HH) and 0.2 g (soils HN and DD) dry weight equivalents per replicate.
Equilibrium solution used		aqueous 0.01 M $CaCl_2$ solution.
Control used		N/A
Test item concentrations	Nominal application rates	Nominal concentrations in test solution: 0.005, 0.015, 0.05, 0.15 and 0.50 mg/L.
	Analytically measured concentrations	Concentrations in test solution: 0.005, 0.016, 0.05, 0.15 and 0.52 mg/L for soils AX, HH, HN; 0.006, 0.016, 0.05, 0.16 and 0.53 mg/L for soil DD.
Identity and concentration of co-solvent		N/A
Soil-to-solution ratio		1/40, i.e. 0.5 g soil dry weight equivalents (soils AX and HH) and 1/100, i.e. 0.2 g soil dry weight equivalents (soils HN and DD), to 20 mL solution (corrected for soil moisture).
pH of the equilibration solution	Initial	pH of aqueous 0.01 M $CaCl_2$ solution without soil: 6.82 - 7.55
	Final	pH with soil and test item after adsorption equilibrium: range 5.9 – 7.7
Number of replications	Controls	N/A
	Treatments	Duplicate
Equilibration	Time	Equilibration with untreated 0.01 M $CaCl_2$ for 1 day
	Temperature	19.5 °C
	Dark	Yes
	Shaking method	Mechanical overhead shaker, 300 rpm
	Shaking time	24 hours for soils AX, HH and HN and 2 hours for soil DD
Method of separation of supernatant		Centrifugation
Centrifugation	Speed	5000 x *g*
	Duration	10 min
	Method of separation of soil and solution	Supernatant was carefully decanted.

Desorption phase

Parameter	Description
Were the soil residues from the adsorption phase	Yes

used?		
Amount of test item present in the adsorbed state / adsorbed amount		The amounts of test item adsorbed to soil after adsorption ranged from 62.5 to 89.5% AR.
Number of desorption cycles		One desorption cycle was performed for each concentration.
Equilibrium solution and quantity used per treatment for desorption		The decanted solution was replaced by fresh aqueous 0.01 M $CaCl_2$ solution. A total volume of 20 mL was used as equilibration solution.
Soil-to-solution ratio		1/40, i.e. 0.5 g soil dry weight equivalents (soils AX and HH), and 1/100, i.e.0.2 g soil dry weight equivalents (soils HN and DD), to 20 mL solution (corrected for soil moisture).
Number of replications	Controls	N/A
	Treatments	Duplicate
Desorption equilibration	Temperature	19.5 °C
	Dark	Yes
	Shaking method	Mechanical overhead shaker, 300 rpm
	Shaking time	2 hours
Method of separation of supernatant		Centrifugation
Centrifugation	Speed	5000 x g
	Duration	10 min
	Method of separation of soil and solution	Supernatant was carefully decanted.

Analytical procedures

The purity of the test item in the application solution was determined by radio-HPLC in the preliminary tests. The parental mass balance was established in the preliminary test. Supernatants were analysed by LSC. The soil was extracted three times at ambient temperature by shaking for 30 minutes each and once by microwave-assisted extraction at 70 °C for 10 minutes using 20 mL acetonitrile / water 4/1 (v/v). Furthermore, one microwave-assisted extraction step was performed using 0.01 N HCl / methanol 1/1 (v/v) at 50 °C for 10 minutes. After each extraction step, extract and soil were separated by centrifugation and decantation. The supernatants (adsorption and desorption) and soil extracts were combined and the volumes determined. The radioactivity contents were determined by LSC and the stability of the test item was determined by HPLC/radio-detection to establish the parental mass balance.

The material balance was determined in the definitive test. Adsorption and desorption solutions were analysed by LSC, soils were air-dried and residues were determined by combustion/LSC to establish the material balance.

II. RESULTS AND DISCUSSION

A. Recovery of test item in preliminary test

Distribution of test item in combined solution and soil extracts, expressed as percentage of applied radioactivity [% AR] (mean) are presented in the table below.

Table 7.1.3- 20: Preliminary test: Total recovery of [U-^{14}C-aniline]-M-01 in solution samples and soil extracts

Compartment	Soil ID				
	AX	HH	HN	DD	Mean

Radioactivity in combined solution and soil extracts [% AR]	97.2	92.0	103.9	91.8	**96.2**
Test item in combined solution and soil extracts [% ROI]*	95.5	99.9	95.6	99.3	**97.6**
Test item in combined solution and soil extracts [% AR]	92.7	91.9	98.9	91.2	**93.7**
Non-extractable residues [% AR]	N/A	N/A	N/A	N/A	
Total recovery of radioactivity [% AR]	97.2	92.0	103.9	91.8	
Total recovery of test item [% AR]	**92.7**	**91.9**	**98.9**	**91.2**	**93.7**

*percentage of regions of interest [% ROI] from primary chromatographic method

B. Transformation of test substance:

The stability of test item and adsorption of test item to test vessel surface were determined prior to the definitive test in preliminary tests. Supernatants, controls as well as supernatants and soil extracts were analysed by LSC and HPLC/radio-detection immediately after sampling. Therefore, no storage stability investigations were necessary. The stability of the test item (control with no soil) was confirmed by radio-HPLC to be > 97% over the duration of the test.

C. Findings:

Within definitive tests, the amount of [^{14}C]M-01 recovered from the test system (adsorption and desorption supernatants and soil residue combustions) ranged from 95.5 to 101.6% AR for soil AX, 96.3 to 102.7% AR for soil HH, 91.7 to 96.4% AR for soil HN and 92.7 to 105.5% AR for soil DD (see table below).

Table 7.1.3- 21: Definitive test: Total recovery of [U-^{14}C-aniline]-M-01 in solution samples and soil extracts

Soil ID	AX	HH	HN	DD
*Conc. ID	[% AR]	[% AR]	[% AR]	[% AR]
A	99.8	97.8	96.4	96.7
B	98.2	102.7	91.7	94.4
C	95.5	97.3	94.9	92.7
D	95.5	96.3	93.9	95.7
E	101.6	97.4	94.7	105.5
Mean	98.1	98.3	94.3	97.0
SD	± 2.4	± 2.3	± 1.5	± 4.4

*A - 0.5, B - 0.15, C - 0.05, D - 0.16 & E – 0.005 mg/L (soils AX, HH, HN), E – 0.006 mg/L (soil DD)
SD = standard deviation

The definitive test was performed at a soil-to-solution ratio of 1:40 for soils AX and HH, 1:100 for soils HN and DD. Equilibrium of the test item was established after 24 hours shaking for soils AX, HH and HN, and 2 hours shaking for soil DD. All four soils had a desorption step of 2 hours. The adsorption behaviour of [^{14}C]M-01 was accurately measured using a nominal concentration range of 0.005 to 0.5 mg/L by the Freundlich equation for all soils. Concentrations of [^{14}C]-M-01 in aqueous and solid phase following the adsorption step are shown in the table below.

Table 7.1.3- 22: Definitive test: Concentration of [U-^{14}C-aniline]-M-01 in aqueous and solid phase following 24 hours of adsorption (2 hours for soil DD Clay Loam)

	Concentration (µg/mL)	Soil (µg/g)	Solution (µg/mL)
Conc. ID	**Soil ID**	**AX Sandy Loam**	
A	0.520	1.8033	0.0076
B	0.153	0.5412	0.0020
C	0.053	0.5420	0.0020
D	0.016	0.1876	0.0006
E	0.005	0.1874	0.0006
	Soil ID	**HH Silt Loam**	
A	0.520	1.8107	0.0074
B	0.153	0.5500	0.0018
C	0.053	0.5461	0.0019
D	0.016	0.1893	0.0006
E	0.005	0.1900	0.0005
	Soil ID	**HN Loam**	
A	0.520	4.1428	0.0112
B	0.153	1.2813	0.0027
C	0.053	1.2829	0.0027
D	0.016	0.4490	0.0008
E	0.005	0.4523	0.0008
	Soil ID	**DD Clay Loam**	
A	0.531	3.8408	0.0158
B	0.159	1.1655	0.0040
C	0.054	1.1549	0.0041
D	0.016	0.4480	0.0017
E	0.006	0.4572	0.0016

The adsorption constants K_f of the Freundlich isotherms ranged from 101.8 to 154.4 mL/g with associated Freundlich exponents 1/n ranging from 0.81 to 0.90. The corresponding correlation coefficients for the adsorption isotherms ranged from 0.996 to 0.999 indicating a linear fit to the measured data. When normalized for the organic carbon content of soil K_{foc} values ranged from 2969 to 6062 mL/g. The Freundlich adsorption and desorption constants for M-01 in soil are summarised below.

Table 7.1.3- 23: Adsorption constants of [U-^{14}C-aniline]-M-01 in soil

Soil	Soil ID	Texture (USDA)	pH *	OC [%]	Clay [%]	K_F [mL/g]	K_{Foc} [mL/g]	1/n
[redacted] Hof AXXa	AX	Sandy loam	5.7	1.9	8	101.8	5359	0.8424
[redacted] am [redacted] 4a	HH	Silt loam	6.2	1.9	16	106.4	5601	0.8380
[redacted] Hof	HN	Loam	5.7	2.5	18	151.6	6062	0.8094
[redacted] H	DD	Loam	7.3	5.2	28	154.4	2969	0.8961
Arithmetic mean						128.5	4998	0.8465
Geometric mean						126.2	4821	0.8459

* pH values were derived from aqueous 0.01 M $CaCl_2$ suspensions (soil characterization data)

The desorption K_{foc} values for soils HH, HN and DD were in the same order of magnitude as those for the adsorption K_{foc} values. However, soil AX was an order of magnitude higher compared to adsorption K_{foc} values. M-01 was strongly adsorbed to all soils with 5.9 to 23.2% remaining in solution after the adsorption cycle.

Table 7.1.3- 24: Summary of Freundlich desorption constants K_f and K_{foc} values

Desorption	Soil	AX	HH	HN	DD
	Textural class	Sandy Loam	Silt Loam	Loam	Clay Loam
Cycle 1	$K_{f\,des}$	122.395	125.559	198.495	327.838
	1/n	0.7995	0.8007	0.7866	0.8864
	R^2	0.998	0.9988	0.9992	0.9933
	$K_{foc\,des}$	64412	6608	7940	6305

D. OECD 106 evaluation checklist:

Relevant quality checks were performed to evaluate the acceptability of the study according to the EFSA Technical Report on the Outcome of Pesticides Peer Review Meeting on the OECD 106 Evaluators Checklist (2017). These checks confirmed that the mass balance of 91.2-98.9% (mass balances >90%) and % adsorption of 59.3-90.0% were all acceptable (<20%). The acceptability of the analytical method was confirmed over the entire range of concentrations measured (LOQ of 0.24 µg/L). The use of the indirect method was appropriate based on a Kd * soil/solution ratio > 0.3 in all soils. The graphical fits of the Freundlich equation based on the standard linear regression form using log-log transformed data alongside the associated residual plots were evaluated and found to be acceptable. The R^2 of the standard linear regressions ranged from 0.996 to 0.999 and the visual fit of both the standard regression and the residual plots were acceptable.

The evaluation confirmed the study was fully acceptable according to all quality checks and thus it can be concluded the study is appropriate for use in regulatory modelling. The results of the evaluation are summarised in the tables below.

Table 7.1.3- 25: Results of OECD 106 evaluation checklist

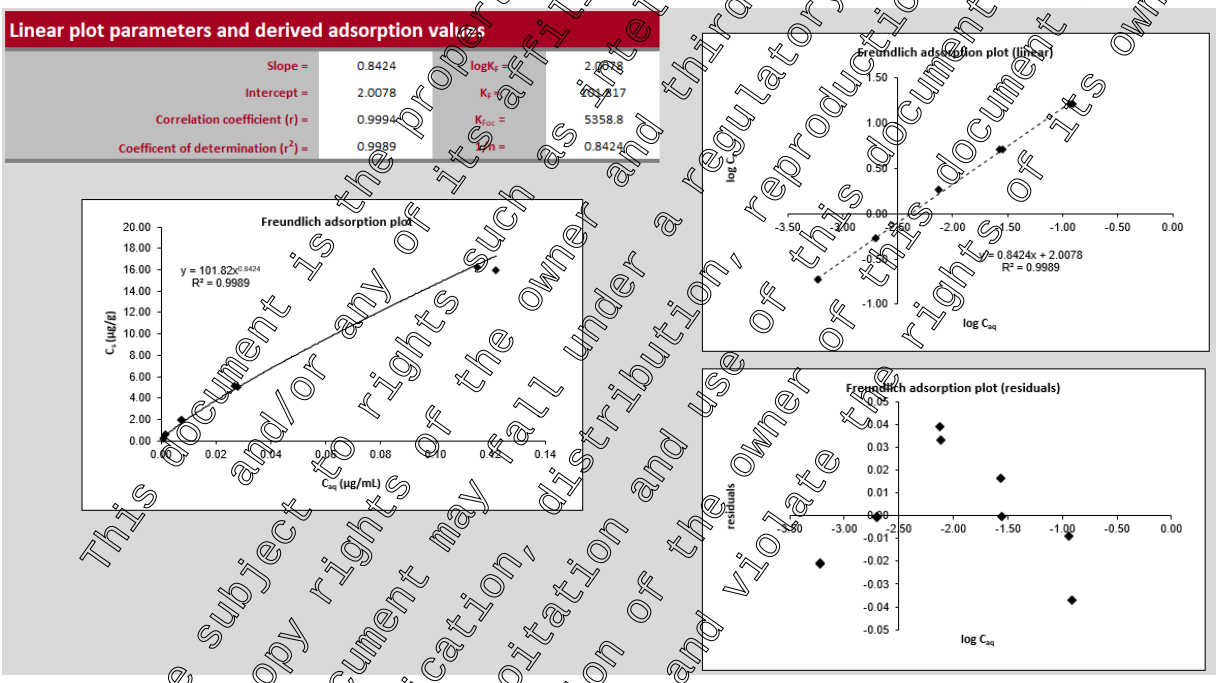
Soil	Units	Quality criteria	Sandy Loam AX	Silt Loam HH	Loam HN	Clay Loam DD
Adsorption method (direct/indirect)	-		Indirect	indirect	indirect	indirect
Soil : solution ratio	g/mL	-	40:1	40:1	100:1	100:1
Mass balance of ^{14}C (at all tested concentrations)	%	90-110%	98.1	98.3	94.3	97.0
Adsorbed percentage (δ)	%	>20%#	76.5-88.0	77.8-90.0	66.0-84.0	59.3-75.0
K_d x (soil:solution ratio)		0.3	3.25-7.82	3.49-9.50	1.94-5.65	1.46-2.91
K_{fE} / K_f	-	<1.2#	1.10 & 1.11	1.11 & 1.12	1.08	1.11-1.12
$^{ads}K_F$ (95% confidence interval)	L/kg	*	101.817 (90.348-114.742)	106.421 (94.206-120.216)	151.550 (135.735-169.208)	154.386 (125.515-189.897)
$^{ads}1/n$ (95% confidence interval)	-	*	0.842 (0.819-0.865)	0.838 (0.815-0.861)	0.809 (0.787-0.832)	0.896 (0.850-0.942)
$^{Ads}R^2$	-	>0.975	0.999	0.999	0.999	0.996
$^{ads}K_{F,OC}$	L/kg	-	5358.8	5601.1	6062.0	2969.0

Soil	Units	Quality criteria	Sandy Loam AX	Silt Loam HH	Loam HN	Clay Loam DD
Visual fit to Freundlich isotherm		**	Acceptable	Acceptable	Acceptable	Acceptable
Residual plots randomly distributed		**	Acceptable	Acceptable	Acceptable	Acceptable

* Confidence intervals should be narrow.
#F factor - calculated using preliminary parental mass balance data where F = 100 - (^{14}C aquatic [%] + ^{14}C extract [%]). For soil HN F = 103.9 - (^{14}C aquatic [%] + ^{14}C extract [%]).The F value was corrected for % parent in recovered radioactivity.
OCED 106 Calculation Tool v1 used.
** Note on assessing visual fits: Poor - an unacceptable fit, the fitted curve does not represent the trend of the data points and residuals show strong deviations from random distribution. Acceptable – the fitted curve describes the trend of the data points, residuals may show some deviation from random distribution but it is not significant. Good – the fitted curve closely follows all the data points, residuals are randomly distributed.

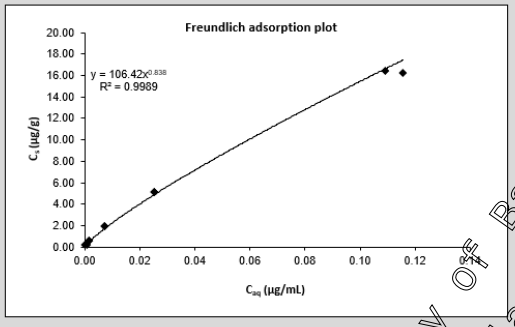
Freundlich plots from OECD 106 Checklist

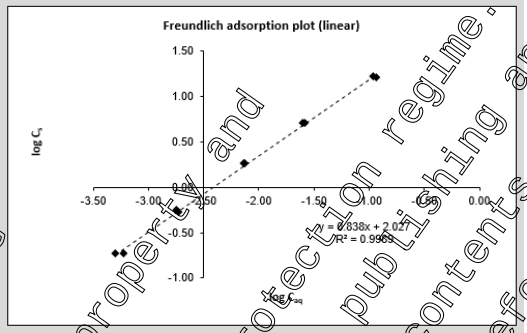
Sandy Loam AX

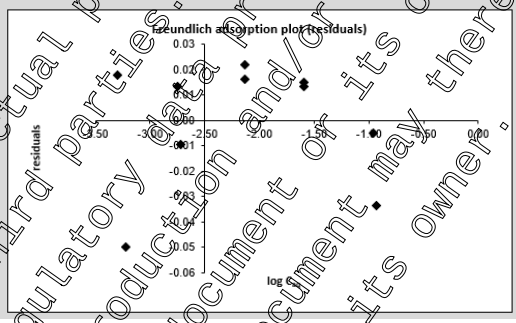


Silt Loam HH

Linear plot parameters and derived adsorption values			
Slope =	0.8380	$\log K_F$ =	2.0270
Intercept =	2.0270	K_F =	106.421
Correlation coefficient (r) =	0.9994	K_{FOC} =	5601.1
Coefficent of determination (r^2) =	0.9989	1/n =	0.8380

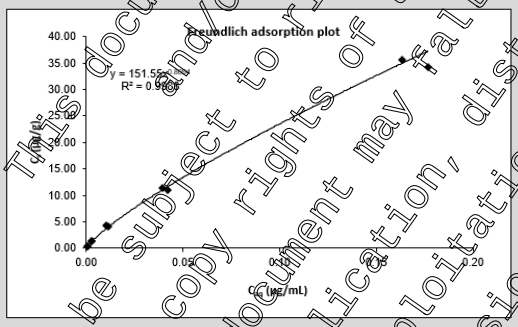


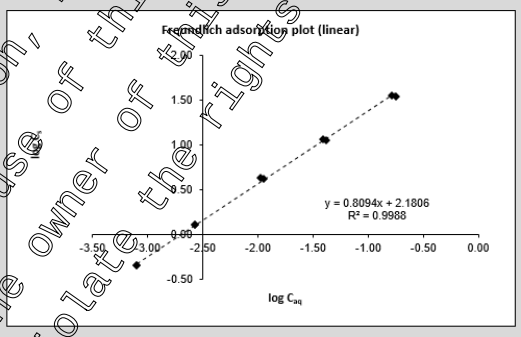


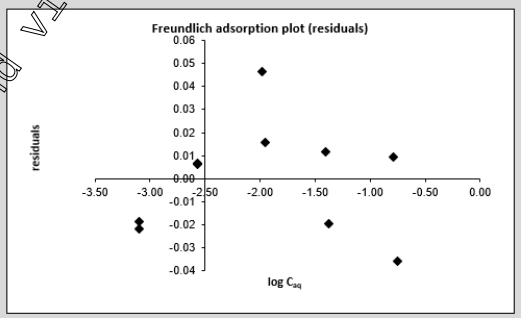


Loam HN

Linear plot parameters and derived adsorption values			
Slope =	0.8094	$\log K_F$ =	2.1806
Intercept =	2.1806	K_F =	151.980
Correlation coefficient (r) =	0.9994	K_{FOC} =	4052.0
Coefficent of determination (r^2) =	0.9988	1/n =	0.8094

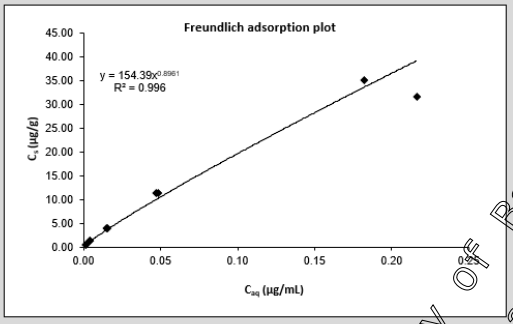


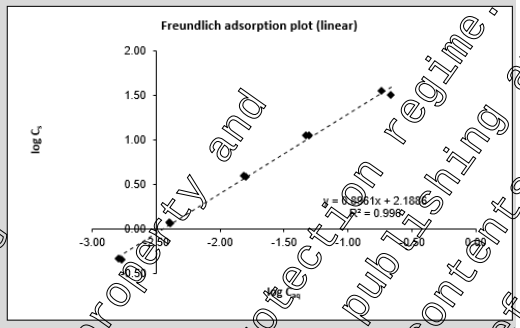




Clay Loam DD

Linear plot parameters and derived adsorption values			
Slope =	0.8961	$\log K_F$ =	2.1886
Intercept =	2.1886	K_F =	154.386
Correlation coefficient (r) =	0.9980	K_{Foc} =	2969.0
Coefficent of determination (r^2) =	0.9960	1/n =	0.8961





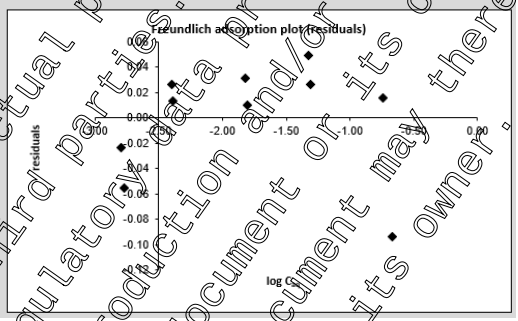


Table 7.1.3- 26: Summary of Quality Criteria and Regulatory Interpretation

M-01		Quality Criteria			Overall Conclusion	Regulatory Interpretation
Soil Type	Soil Name	Met	Partially Met	Not Met		
Sandy Loam	AX	9	0	0	Met	Acceptable
Silt Loam	HH	9	0	0	Met	Acceptable
Loam	HN	9	0	0	Met	Acceptable
Clay Loam	DD	9	0	0	Met	Acceptable

Table 7.1.3- 27: Impact on Endpoints

Soil Type	Soil Name	K_{foc} (Reported)	K_{foc} (OECD tool)	1/n (Reported)	1/n (OECD tool)
Sandy Loam	AX	5359	5359	0.8424	0.842
Silt Loam	HH	5601	5601	0.8380	0.838
Loam	HN	6062	6062	0.8094	0.809
Clay Loam	DD	2969	2969	0.8961	0.896
Arithmetic Mean		4998	4998	0.8465	0.846
Geometric Mean		4821	4821	0.8459	0.846
pH dependence		No	No		

The small differences between the reported values and the OECD calculation tool (v2) are considered to be due to rounding in the calculations. The reported values have been used in modelling calculations.

III. CONCLUSION

The adsorption constants K_F of M-01 for the tested soils calculated based on the Freundlich isotherms ranged from 101.8 to 154.4 mL/g (geometric mean: 126.2 mL/g). The respective K_{Foc} values ranged from 2969 to 6062 mL/g (geometric mean: 4821 mL/g). The desorption constants $K_{F(des)}$ of M-01 were

up to 2 times higher than the respective adsorption constants, indicating a strengthened binding of M-01 once adsorbed to soils representing conditions relevant for the environment. M-01 was stable during the test. The parental mass balance was ≥ 91.2% AR. No major degradation product was observed.

Using the Briggs classifications for the estimation of the mobility of chemicals in soil based on K_F and/or K_{Foc} values, M-01 can be classified as immobile for adsorption and for desorption.

All 4 soils can be considered 'met' with regard to the quality criteria and therefore acceptable for regulatory use.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 106 (2000) and is considered valid to assess the adsorption and desorption characteristics of the aclonifen metabolite M-01 in soil.

Assessment and conclusion by RMS:

CA 7.1.3.2 Aged sorption

An assessment of aged sorption is an optional higher tier study which is not required for aclonifen.

CA 7.1.4 Mobility in soil

CA 7.1.4.1 Column leaching studies

CA 7.1.4.1.1 Column leaching of the active substance

Column leaching studies are now only required when it is not possible to obtain reliable adsorption coefficient values due to weak adsorption. Reliable adsorption coefficients were obtained from OECD 106 studies with aclonifen which are provided in KCA 7.1.3.1.1. For procedural reasons the three previously submitted column leaching studies (and an English translation of an original German report) have to be included under Point KCA 7.1.4.1.1 in the current dossier (KCA 7.1.4.1.1/01, KCA 7.1.4.1.1/02, KCA 7.1.4.1.1/03 and KCA 7.1.4.1.1/04) but these studies have been fully superseded by the studies provided in KCA 7.1.3.1.1.

Data Point:	KCA 7.1.4.1.1/01
Report Author:	[redacted]
Report Year:	1981
Report Title:	Leaching properties of agrochemicals - CME 127
Report No:	R007353
Document No:	M-174739-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: OECD 312 (2004) Endpoint is Current Guideline; No longer required for the registration of active ingredients in the EU. Superseded by OECD 106 test. Reporting standard Current Guideline: Not acceptable. Current Guideline: No recoveries provided.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

In the previous submission (DAR, 2006), this study was evaluated and accepted as valid. However the study endpoint is no longer mandatory for the registration of active ingredients in the EU and hence a summary of this study is not presented in this dossier.

Data Point:	KCA 7.1.4.1.1/02
Report Author:	[redacted]
Report Year:	1986
Report Title:	Versickerungsverhalten des Pflanzenschutzmittels - Aclonifen
Report No:	R007354
Document No:	M-174741-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: OECD 312 (2004) Endpoint is no longer required for the registration of active ingredients in the EU. Superseded by OECD 106 test. Reporting standard not acceptable. No recoveries provided.
Previous evaluation:	yes, evaluated and accepted Study list relied on, Dec 2011 (BVL) R007354. Advise: 02-1 should be used as EN edition which is also listed in list relied upon
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

Data Point:	KCA 7.1.4.1.1/03
Report Author:	[redacted]
Report Year:	1986
Report Title:	Leaching properties of agrochemicals - Aclonifen (CME 127)
Report No:	C010546
Document No:	M-199204-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current Guideline: OECD 312 (2004) Endpoint is no longer required for the registration of active ingredients in the EU. Superseded by OECD 106 test. Reporting standard not acceptable. no recoveries provided.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

In the previous submission (DAR, 2006), this study (KCA 7.1.4.1.1/02) and its English translation (KCA 7.1.4.1.1/03) were evaluated and accepted as valid. However the study endpoint is no longer mandatory for the registration of active ingredients in the EU and hence a summary of this study and its translation are not presented in this dossier.

Data Point:	KCA 7.1.4.1.1/04
Report Author:	[redacted]
Report Year:	1993
Report Title:	ACLONIFEN: Aged leaching in five soils
Report No:	R007077
Document No:	M-174161-01-1
Guideline(s) followed in study:	USEPA (=EPA): 163-1
Deviations from current test guideline:	Current Guideline: OECD 312 (2004) Endpoint is no longer required for the registration of active ingredients in the EU. Superseded by OECD 106 test.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

In the previous submission (DAR, 2006), this study was evaluated and accepted as valid. However the study endpoint is no longer mandatory for the registration of active ingredients in the EU and hence a summary of this study is not presented in this dossier.

CA 7.1.4.1.2 Column leaching of metabolites, breakdown and reaction products

Column leaching studies are now only required when it is not possible to obtain reliable adsorption coefficient values due to weak adsorption. Reliable adsorption coefficients were obtained from an OECD 106 study with the aclonifen metabolite M-01 which is provided in KCA 7.1.3.1.2.

CA 7.1.4.2 Lysimeter studies

The potential mobility of aclonifen has been assessed by modelling and therefore a lysimeter study is not required.

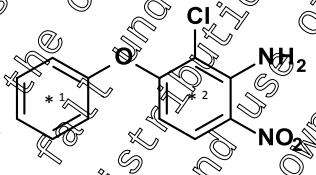
CA 7.1.4.3 Field leaching studies

The potential mobility of aclonifen has been assessed by modelling and therefore a field leaching study is not required.

CA 7.2 Fate and behaviour in water and sediment

The fate and behaviour of aclonifen in aquatic systems has been investigated under abiotic and biotic conditions in a series of laboratory studies. A number of studies were submitted for the first inclusion of aclonifen into Annex I of Council Directive 91/414/EEC and reviewed under uniform principles (DAR, Germany, 2006). In addition a number of new studies are provided for the current EU review. All valid environment fate studies are considered in the MCA 7 dossier.

Aquatic laboratory studies have generally been conducted with ^{14}C-aclonifen, uniformly labelled in either the phenoxy or aniline rings.



* Denotes position of [^{14}C]-radiolabel

1 = [Phenoxy-UL-^{14}C]-aclonifen 2 = [Aniline-UL-14C]-aclonifen

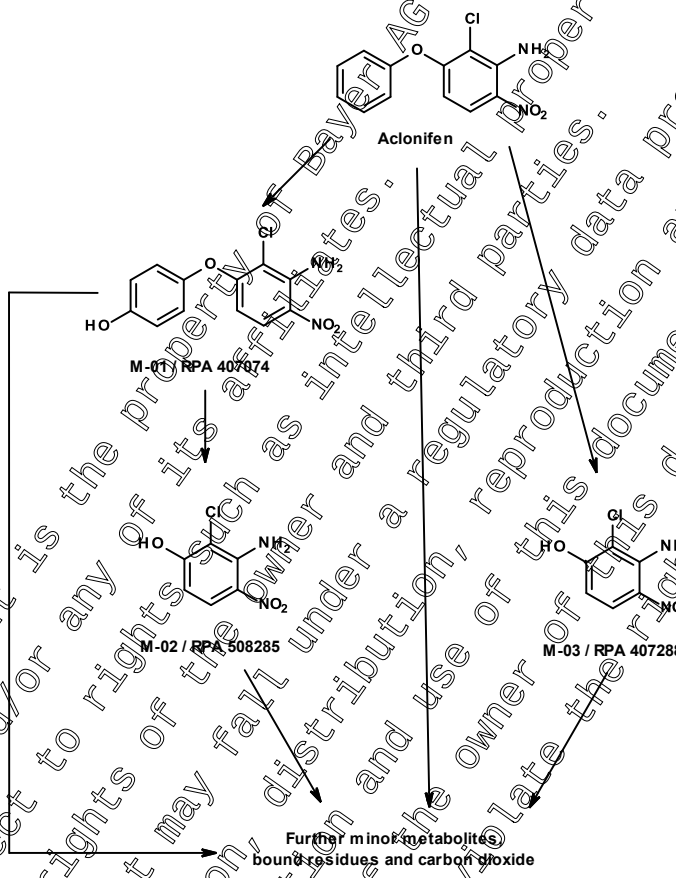
Under sterile aqueous conditions at temperatures of 22°C, 50°C and 70°C, aclonifen was found to be hydrolytically stable at pH 5, 7 and 9. The photolytic degradation of [aniline-UL-^{14}C]-aclonifen in water has been investigated under sterile conditions in phosphate buffer solution at pH 7. Aclonifen exhibited slow degradation when irradiated in sterile pH 7 buffer solution at 25 ºC, with up to 88 % of applied radioactivity still recovered as parent at the end of the study after 16 days (equivalent to 30 days natural sunlight). No major (>10%) metabolites were formed by photolysis in water.

Aclonifen was found to be not readily biodegradable under the stringent conditions of OECD guideline 301B in which only very limited opportunity for biodegradation and microbial acclimatisation was provided.

Aerobic mineralization studies and water sediment studies have been conducted with ^{14}C-aclonifen, uniformly labelled in either the phenoxy or aniline rings. In aerobic mineralization studies treated with [aniline-UL-^{14}C]-aclonifen, the metabolites M-01 and M-02 were observed as major metabolites (≥ 10%). In water sediment systems treated with [aniline-UL-^{14}C]-aclonifen, M-01, M-02 and M-03 were observed as minor metabolites. The combined sum of the cleaved metabolites M-02 and M-03 observed throughout the water sediment study was at a maximum of only 4%. No significant metabolites were observed in the suspended sediment aerobic mineralization or the water sediment studies treated with [phenoxy-UL-^{14}C]-aclonifen. Formation of unextractable bound residues in sediment was the major metabolic pathway in aquatic systems. Under sterile conditions aclonifen was relatively stable confirming that its metabolism is largely microbially mediated. Non-extractable

sediment bound residues were observed under sterile conditions at much lower levels than observed in microbially viable systems, indicative of metabolites of aclonifen also binding to the sediment matrix with time in microbially active systems. The metabolic pathway for aclonifen in aquatic systems is shown below.

Figure 7.2-1 Metabolic pathway for aclonifen in surface water



A new kinetic evaluation of the experimental data generated in two water sediment studies KCA 7.2.2.3/01 and KCA 7.2.2.3/06 has been conducted according to FOCUS kinetics guidance with the aim of deriving DT_{50} values for use as modelling and trigger endpoints ([redacted] & [redacted], 2019, KCA 7.2.2.3/08). The geometric mean modelling endpoints $DegT_{50}$ values for aclonifen are summarised in the table below.

Table 7.2- 1: Summary of modelling endpoint DT_{50} values for aclonifen in aquatic / sediment systems

Compound	Laboratory modelling endpoint DT_{50} (20 °C)		
	DT_{50} range (days)	Number of datasets (n)	Geometric mean DT_{50} (days) for exposure assessment
Total system	4.80 - 43.81	4	14.4
Water phase	0.83 -3.39	4	1.7
Sediment	8.43 – 69.49	4	26.1

CA 7.2.1 Route and rate of degradation in aquatic systems (chemical and photochemical degradation)

Data Point:	KCA 7.2.1/01
Report Author:	[redacted]; [redacted]
Report Year:	2019
Report Title:	Aclonifen - Effect of water treatment processes on the nature of residues present in groundwater and surface water
Report No:	EnSa-19-0595
Document No:	M-676096-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	not applicable
Acceptability/Reliability:	

Executive Summary

An assessment of the potential impact of drinking water treatment, considering both the exposure aspects and transformation chemistry, has been prepared for aclonifen.

The definition of residue relevant for assessment for aclonifen in groundwater and surface water is parent active substance only. No exposure of groundwater is predicted following application of aclonifen. The worst-case assumption for potential exposure of surface water at the field scale for aclonifen would be the regulatory acceptable concentration (RAC) of 1.36 µg/L. However raw water for drinking water purposes will usually be abstracted from dedicated groundwater and surface water sources where attenuation factors from conservative field scale predictions to actual exposure situation at drinking water abstraction points will be significant. Before being subjected to chemical-oxidative treatment steps, raw water sourced from a surface water body would typically undergo filtration steps. As aclonifen is a highly adsorptive compound (K_{oc} = 5727, Log P = 4.37), it would be expected that any contact with organic material in these pre-treatment steps would further significantly reduce any aclonifen presence, via simple physical adsorption processes. Consequently it is not expected any residues of the aclonifen would be present at the chemico-oxidative stages of drinking water processing in excess of 0.1 µg/L.

An evaluation of public monitoring data has shown findings of aclonifen are extremely rare with exceedance above the regulatory groundwater trigger of 0.1 µg/L at only 0.007% (12 out of 184651 analyses) and above the surface water Tier 1-RAC-SW also at 0.007% (22 out of 311942 analyses).

The diphenylether moiety is not known to raise any concerns for critical transformation reactions with chlorine or ozone-based water treatment. Literature searches did not identify any relevant publications on water treatment of aclonifen. One publication observed no reactivity of four diphenylether herbicides with chlorine or ozone. The structure of aclonifen does not match any of the previously known precursors of N-nitrosodimethylamine (NDMA), nor does it include any substructure moieties of concern.

Aclonifen residues is not expected to be presence in raw water abstracted for drinking water production. In the highly unlikely case that residues are present, there are no indications of any risk of forming N-nitrosamine type byproducts upon oxidative water processing from aclonifen residues.

I. MATERIALS AND METHODS

The first step in the assessment was a review of the predicted exposure concentrations at the field level for groundwater and surface water. This was refined by a review of the predicted exposure situation at raw water abstraction points and finally the impact of mechanical, physical, biological steps conducted prior to the eventual chemical steps on the predicted exposure situation at the chemical stages of raw water processing was assessed.

The next step in the assessment was an assessment of the behaviour of aclonifen residues upon chemical-oxidative water treatment. For the production of public drinking water, raw water is sourced from groundwater and/or surface water and is processed for purification and disinfection typically by a sequence of mechanical, physical, biological, and eventually chemical treatment steps. The chemical steps can be summarised as an exposure of pre-cleaned raw water to chemical oxidants, aiming for a transformation or mineralization of unwanted matrix components and micropollutants (e.g. to remove colour, odour, toxicity, and/or to meet other quality parameters), and for the inactivation of microbial life (i.e. control of waterborne diseases). In practice, two major groups of chemical reagents are in use; ozone-based treatment and chlorine-based treatment. An evaluation of public monitoring data for findings of aclonifen and public literature searches have been conducted to identify any relevant publications on water treatment of aclonifen.

Different classes of compounds have been assayed for their potential to form nitrosodimethylamine (NDMA) upon ozone treatment. Three types of structural moieties appear to have relevance as eventual NDMA precursors which can serve as structure alerts for screening purposes; N,N-dimethylhydrazine (UDMH) derivatives, N,N-dimethylsulfamide (DMS) derivatives and N,N-dimethylaminocarbamate (DMAC) derivatives. The structure of aclonifen was checked for exact matches, matching substructures or homologues that might form UDMH, DMS or DMAC.

II. RESULTS AND DISCUSSION

Predicted exposure concentrations at the field level

The definition of residue relevant for assessment for aclonifen in groundwater and surface water is parent active substance only. The first step was a review of the groundwater and surface water exposure situation at the field scale level for aclonifen based on the PEC_{gw} and PEC_{sw} values predicted for the most critical 'representative use' for EU Annex I inclusion. These values are provided in full in both Documents MCP Section 9.

The predicted environmental concentrations (PEC) for groundwater are presented below for the highest use rate.

Table 7.2.1- 1: FOCUS PEARL, PELMO and MACRO, PECgw results of aclonifen in legumes at 600 g/ha

Crop	Scenario	80th percentile PECgw at 1 m soil depth (µg/L)	
		Aclonifen	
		PEARL	PELMO
Beans (field) Peas (Animal)	[redacted]	<0.001 <0.001 <0.001 <0.001 <0.001 <0.001 <0.001 <0.001 <0.001	<0.001 <0.001 <0.001 <0.001 <0.001 <0.001 <0.001 <0.001 <0.001
MACRO	[redacted]	<0.001	

No exposure of groundwater is predicted following application of aclonifen.

The PEC values for surface water for FOCUS Step 4 are presented below for the highest use rate for cereals and peas.

Table 7.2.1- 2: Winter cereals: Single application FOCUS Step 4 PEC$_{sw}$ results for aclonifen, use rate 350 g/ha

PECsw (µg/L)	Scenario	Step 4 Aclonifen							
Nozzle reduction	Vegetated strip (m)	None	None	None	None	None	10 m	10 m	20 m
	No spray buffer (m)	0 m	5 m	10 m	15 m	20 m	10 m	15 m	20 m
None	D1 Ditch	2.22	0.602	0.349	0.218	0.166	0.319	0.218	0.166
50 %		1.11	0.301	0.159	0.109	0.083	0.159	0.109	0.083
75 %		0.585	0.150	0.080	0.054	0.041	0.080	0.054	0.041
90 %		0.222	0.060	0.032	0.022	0.017	0.032	0.022	0.017
None	D1 Stream	1.95	0.710	0.376	0.257	0.195	0.376	0.257	0.195
50 %		0.972	0.355	0.188	0.128	0.098	0.188	0.128	0.098
75 %		0.485	0.177	0.094	0.064	0.049	0.094	0.064	0.049
90 %		0.194	0.071	0.038	0.026	0.020	0.038	0.026	0.020
None	D2 Ditch	2.23	0.603	0.319	0.218	0.166	0.319	0.218	0.166
50 %		1.11	0.301	0.160	0.109	0.083	0.160	0.109	0.083
75 %		0.556	0.150	0.080	0.054	0.041	0.080	0.054	0.041
90 %		0.222	0.060	0.032	0.022	0.017	0.032	0.022	0.017
None	D2 Stream	1.98	0.723	0.383	0.262	0.199	0.383	0.262	0.199
50 %		0.990	0.361	0.191	0.131	0.099	0.191	0.131	0.099
75 %		0.494	0.180	0.096	0.065	0.050	0.096	0.065	0.050
90 %		0.198	0.072	0.038	0.026	0.020	0.038	0.026	0.020
None	D3 Ditch	2.19	0.594	0.315	0.215	0.163	0.315	0.215	0.163
50 %		1.10	0.297	0.157	0.107	0.082	0.157	0.107	0.082
75 %		0.547	0.148	0.079	0.054	0.041	0.079	0.054	0.041
90 %		0.219	0.059	0.031	0.021	0.016	0.031	0.021	0.016

PECsw (µg/L)	Scenario	Step 4 Aclonifen							
Nozzle reduction	Vegetated strip (m)	None	None	None	None	None	10 m	10 m	20 m
	No spray buffer (m)	0 m	5 m	10 m	15 m	20 m	10 m	15 m	20 m
None	D4 Pond	0.076	0.065	0.047	0.037	0.031	0.047	0.037	0.031
50 %		0.038	0.033	0.023	0.019	0.016	0.023	0.019	0.016
75 %		0.019	0.016	0.012	0.009	0.008	0.012	0.009	0.008
90 %		0.008	0.007	0.005	0.004	0.004	0.005	0.004	0.004
None	D4 Stream	1.90	0.694	0.368	0.251	0.191	0.368	0.251	0.191
50 %		0.950	0.346	0.184	0.125	0.095	0.184	0.125	0.095
75 %		0.474	0.173	0.092	0.063	0.048	0.092	0.063	0.048
90 %		0.190	0.069	0.037	0.032	0.032	0.037	0.032	0.032
None	D5 Pond	0.076	0.065	0.047	0.037	0.031	0.047	0.037	0.031
50 %		0.038	0.033	0.024	0.019	0.016	0.024	0.019	0.016
75 %		0.019	0.016	0.012	0.009	0.008	0.012	0.009	0.008
90 %		0.008	0.007	0.005	0.004	0.003	0.005	0.004	0.003
None	D5 Stream	2.05	0.748	0.397	0.271	0.206	0.397	0.271	0.206
50 %		1.02	0.374	0.198	0.135	0.103	0.198	0.135	0.103
75 %		0.512	0.187	0.099	0.068	0.051	0.099	0.068	0.051
90 %		0.205	0.075	0.040	0.027	0.021	0.040	0.027	0.021
None	D6 Ditch	2.22	0.800	0.318	0.217	0.165	0.318	0.217	0.165
50 %		1.11	0.300	0.159	0.108	0.083	0.159	0.108	0.083
75 %		0.553	0.150	0.079	0.062	0.062	0.079	0.062	0.062
90 %		0.221	0.062	0.062	0.062	0.062	0.062	0.062	0.062
None	R1 Pond	0.105	0.101	0.095	0.092	0.090	0.048	0.045	0.032
50 %		0.092	0.091	0.088	0.086	0.085	0.040	0.038	0.021
75 %		0.086	0.085	0.084	0.083	0.083	0.036	0.035	0.019
90 %		0.083	0.082	0.082	0.081	0.081	0.034	0.034	0.017
None	R1 Stream	1.44	0.527	0.477	0.477	0.477	0.279	0.214	0.145
50 %		0.722	0.477	0.477	0.477	0.477	0.214	0.214	0.111
75 %		0.477	0.477	0.477	0.477	0.477	0.214	0.214	0.111
90 %		0.477	0.477	0.477	0.477	0.477	0.214	0.214	0.111
None	R3 Stream	2.01	0.732	0.513	0.513	0.513	0.388	0.265	0.201
50 %		1.00	0.513	0.513	0.513	0.513	0.234	0.234	0.123
75 %		0.513	0.513	0.513	0.513	0.513	0.234	0.234	0.123
90 %		0.513	0.513	0.513	0.513	0.513	0.234	0.234	0.123
None	R4 Stream	1.45	0.701	0.701	0.701	0.701	0.316	0.316	0.165
50 %		0.726	0.701	0.701	0.701	0.701	0.316	0.316	0.165
75 %		0.701	0.701	0.701	0.701	0.701	0.316	0.316	0.165
90 %		0.701	0.701	0.701	0.701	0.701	0.316	0.316	0.165

Table 7.2.1- 3: Peas: Single application FOCUS Step 4 PEC_{sw} results for aclonifen, use rate 600 g/ha

PEC_{sw} (µg/L)	Scenario	Step 4 Aclonifen							
Nozzle reduction	Vegetated strip (m)	None	None	None	None	None	10 m	10 m	20 m
	No spray buffer (m)	0 m	5 m	10 m	15 m	20 m	10 m	15 m	20 m
None	D3 Ditch	3.12	1.02	0.541	0.369	0.281	0.541	0.369	0.281
50 %		1.56	0.510	0.270	0.185	0.140	0.270	0.185	0.140
75 %		0.778	0.255	0.135	0.092	0.070	0.135	0.092	0.070
90 %		0.311	0.102	0.054	0.037	0.028	0.054	0.037	0.028
None	D4 Pond	0.125	0.112	0.081	0.064	0.054	0.081	0.064	0.054
50 %		0.063	0.056	0.040	0.032	0.027	0.040	0.032	0.027
75 %		0.031	0.028	0.020	0.016	0.013	0.020	0.016	0.013
90 %		0.013	0.011	0.008	0.007	0.007	0.008	0.007	0.007
None	D4 Stream	2.54	1.07	0.565	0.386	0.294	0.565	0.386	0.294
50 %		1.27	0.533	0.282	0.193	0.147	0.282	0.193	0.147
75 %		0.633	0.266	0.141	0.096	0.073	0.141	0.096	0.073
90 %		0.253	0.106	0.056	0.051	0.051	0.056	0.051	0.051
None	D5 Pond	0.126	0.112	0.081	0.064	0.054	0.084	0.064	0.054
50 %		0.063	0.056	0.040	0.032	0.027	0.040	0.032	0.027
75 %		0.031	0.028	0.020	0.016	0.014	0.020	0.016	0.014
90 %		0.013	0.011	0.008	0.006	0.005	0.008	0.006	0.005
None	D5 Stream	3.60	1.09	0.578	0.395	0.300	0.578	0.395	0.300
50 %		1.30	0.545	0.289	0.197	0.150	0.289	0.197	0.150
75 %		0.648	0.272	0.144	0.099	0.075	0.144	0.099	0.075
90 %		0.259	0.109	0.058	0.039	0.030	0.058	0.039	0.030
None	D6 Ditch	3.12	1.02	0.541	0.370	0.281	0.541	0.370	0.281
50 %		1.56	0.510	0.270	0.185	0.140	0.270	0.185	0.140
75 %		0.779	0.255	0.135	0.092	0.070	0.135	0.092	0.070
90 %		0.311	0.102	0.054	0.043	0.043	0.054	0.043	0.043
None	R1 Pond	0.133	0.119	0.088	0.079	0.079	0.083	0.067	0.055
50 %		0.079	0.079	0.079	0.079	0.079	0.043	0.035	0.028
75 %		0.079	0.079	0.079	0.079	0.079	0.032	0.032	0.016
90 %		0.079	0.079	0.079	0.079	0.079	0.032	0.032	0.016
None	R1 Stream	2.16	0.907	0.707	0.707	0.707	0.481	0.328	0.250
50 %		1.08	0.707	0.707	0.707	0.707	0.318	0.318	0.166
75 %		0.707	0.707	0.707	0.707	0.707	0.318	0.318	0.166
90 %		0.707	0.707	0.707	0.707	0.707	0.318	0.318	0.166
None	R2 Stream	2.87	1.21	0.639	0.436	0.332	0.639	0.436	0.332
50 %		1.43	0.602	0.319	0.218	0.186	0.319	0.218	0.166
75 %		0.715	0.301	0.186	0.186	0.186	0.159	0.109	0.083

PECsw (µg/L)	Scenario	Step 4 Aclonifen							
Nozzle reduction	Vegetated strip (m)	None	None	None	None	None	10 m	10 m	20 m
	No spray buffer (m)	0 m	5 m	10 m	15 m	20 m	10 m	15 m	20 m
90 %		0.286	0.186	0.186	0.186	0.186	0.085	0.085	0.044
None	R3 Stream	3.05	1.28	0.680	0.502	0.502	0.680	0.464	0.353
50 %		1.52	0.641	0.502	0.502	0.502	0.340	0.232	0.176
75 %		0.761	0.502	0.502	0.502	0.502	0.229	0.229	0.120
90 %		0.502	0.502	0.502	0.502	0.502	0.229	0.229	0.120
None	R4 Stream	2.15	1.16	1.16	1.16	1.16	0.516	0.516	0.268
50 %		1.16	1.16	1.16	1.16	1.16	0.516	0.516	0.268
75 %		1.16	1.16	1.16	1.16	1.16	0.516	0.516	0.268
90 %		1.16	1.16	1.16	1.16	1.16	0.516	0.516	0.268

To reflect a situation "*consequent on application consistent with good plant protection practice and having regard to realistic conditions of use*" (1107/2009, Article 4(3b)), it should also be considered that registration of products containing aclonifen will establish an ecotoxicological assessment for the protection of aquatic habitats. Where needed, this assessment will impose measures for mitigating the exposure at the field scale to an upper bound value defined as the regulatory acceptable concentration (RAC). In practice, therefore the worst-case assumption for potential exposure of surface water at the field scale for aclonifen would be the RAC of 1.36 µg/L.

Predicted exposure situation at raw water abstraction points

The second step was a review of the exposure situation expected at raw water abstraction points of relevance for technical drinking water production. Raw water for drinking water purposes will usually be abstracted from protected groundwater and surface water sources that are dedicated for this use. The attenuation factor from conservative field scale predictions to actual exposure situation at drinking water abstraction points will be significant. Within recent EU review processes explicit dilution factors have been proposed and accepted by Rapporteur Member States and EFSA within an overall range from 1:10 – 1:1000000 for various active substances.

The maximum edge-of-field water body exposure situation for aclonifen (1.36 µg/L) indicates that attenuation by only factor 15 upon the PECsw -> PECraw transfer would already be sufficient not exceed the parametric value of 0.1 µg/L at the raw water abstraction point. Aclonifen exposure levels which might be expected at raw water abstraction points would therefore not trigger generic concerns for water treatment, would remain well within the range previously considered acceptable for other components, and would be by far below the limits specified for surrogate disinfection by-products (DBP) in the drinking water regulation.

Predicted exposure situation at the chemical stages of raw water processing

The exposure assessment identified surface water as the predominant, though overall low, exposure route of potential relevance for the assessment of water treatment. Before being subjected to chemical-oxidative treatment steps, raw water sourced from a surface water body would typically undergo either a bank filtration process, pass a sand filtration step, or other filtering systems for pre-cleaning, potentially followed by flocculation. As aclonifen is a highly adsorptive compound (K_{oc} = 5727, Log P = 4.37), it would be expected that any contact with organic material in these pre-treatment steps would further significantly reduce any aclonifen presence, via simple physical adsorption processes. It is not expected any residues of the aclonifen would be present at the chemico-oxidative stages of drinking water processing in excess of 0.1 µg/L.

Environmental monitoring information

An evaluation of public monitoring data has shown findings of aclonifen in public databases are not common (see KCA 7.5/01, [redacted], [redacted] & May, 2019). The results from the groundwater monitoring data search indicates that aclonifen concentrations above the regulatory trigger of 0.1 µg/L are exceedingly rare (12 samples; 0.007% of 184651 analyses) and at most 10.4 µg/L. The results from the surface water monitoring data search indicate that aclonifen concentrations above the Tier 1-RAC-SW (1.36 µg/L) are exceedingly rare (22 samples, 0.007% of 311942 analyses) and are at most 65 µg/L.

Literature information related to Aclonifen behaviour upon water treatment

A public literature search was conducted on aclonifen (see Document MCA Section 9) which did not identify any relevant publications on water treatment of aclonifen. A literature search conducted for diphenylether herbicides located one publication (Okumura, 1992) in which no reactivity with chlorine or ozone was observed for four diphenylether herbicides. It can therefore be concluded that the diphenylether moiety, the characteristic core structure of aclonifen's herbicide class, is not known to raise any particular concern for critical transformation reactions under the conditions of chlorine and/or ozone-based water treatment.

Evaluation for possible risk of N-nitrosamine generation

The structure of aclonifen does not match any of the previously known precursors of N-nitrosodimethylamine (NDMA). Aclonifen does not include any substructure moieties that would be expected prone to cleavage forming N,N-dimethylhydrazine ('unsymmetrical dimethylhydrazine', UDMH), N,N-dimethylsulfamide (DMS) or N,N-dimethyl-O-aminocarbamate (DMAC). The parent molecule does not contain a dimethyl-substituted amine moiety. Moreover, the aclonifen structure does not include any homologues or analogues to the UDMH, DMS, DMAC moieties. The two nitrogen atoms present are not interconnected by a potential leaving group. No environmental transformation reactions are known that might convert aclonifen to form one of the known alerting motifs.

III. CONCLUSION

In conclusion, it is not to be expected that aclonifen residues even in the highly unlikely case of a presence in raw water abstracted for drinking water production would bear a risk of forming N-nitrosamine type byproducts upon oxidative water processing.

Assessment and conclusion by applicant:

The position paper is considered valid to aid assessment of the potential impact of drinking water treatment.

Assessment and conclusion by RMS:

Aclonifen is hydrolytically stable at pH 5, 7 and 9 and photolytically stable in water at pH 7 and at 25°C. No major metabolites are formed by hydrolysis or photolysis in water.

CA 7.2.1.1 Hydrolytic degradation

The hydrolytic degradation of aclonifen has been investigated in Study KCA 7.2.1.1/01 which was evaluated during the previous EU review and is still considered acceptable.

Report	Author, Year	Aniline	Phenoxy	Comment

reference		Label	Label	
KCA 7.2.1.1/01 M-174330-01-1	[redacted], P., [redacted], E. & [redacted] J., 1991	✓	×	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.

Data Point:	KCA 7.2.1.1/01
Report Author:	[redacted]; [redacted]; [redacted]
Report Year:	1991
Report Title:	Herbicides: Aclonifen-14C Hydrolysis under aqueous conditions at 22 degrees C., 50 degrees C. and 70 degrees C.
Report No:	R007157
Document No:	M-174330-01-1
Guideline(s) followed in study:	BBA: 55; OECD: 111
Deviations from current test guideline:	Current Guideline: OECD 111 (2004) Acidic hydrolysis experiment conducted at pH 5 not pH 4.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2014 (RMS, DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive summary:

The hydrolysis of [^{14}C]- labelled aclonifen, was investigated in sterile buffers at pH 5, 7 and 9 at a nominal concentration of approximately 1 mg/L.

The test samples were incubated in the dark, under sterile conditions at 22 ± 1, 50 ± 1 and 70 ± 1°C for up to 31 days. Duplicate samples for each pH and temperature were incubated for 0, 1, 3, 7, 14 and 31 days. The pH of each solution was confirmed at sampling. The samples were extracted with hexane and an aliquot of the extract was counted by LSC to confirm quantitative recovery of the radioactivity.

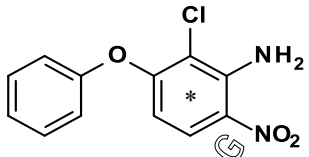
Good radiochemical balances were achieved with mean recoveries of applied radioactivity of 112.0, 108.6 and 107.7% for pH 5, 7 and 9, respectively at 22°C, 113.6, 110.1 and 106.3% for pH 5, 7 and 9, respectively at 50°C and 112.2, 111.8 and 108.3% for pH 5, 7 and 9, respectively at 70°C. The pH values of the samples were maintained throughout the study.

Under sterile aqueous conditions at temperatures of 22°C, 50°C and 70°C, aclonifen was found to be 105.1 stable at pH 5, 7 and 9.

I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: [U-^{14}C-aniline]-aclonifen



* indicates position of ^{14}C radiolabel

Chemical name (IUPAC):	2-chloro-6-nitro-3-phenoxyaniline
Specific activity:	18.55 mCi/mmol or 6.86 MBq/mg
Lot or batch number:	KWC 1747
Radiochemical purity:	≥ 99.6%
CA registry number:	74070-46-5
Stability of test compound:	Stable, determined within study
Application vehicle:	Radiolabelled material dissolved in sterile buffers

B. STUDY DESIGN AND METHODS

1. In-life dates:

19 September 1990 – 21 March 1991

2. Experimental design

This study was performed in 0.035M pH 5 phthalate buffer, 0.05M pH 7 phosphate buffer and 0.05M pH 9 borate buffer. The 0.035M pH 5 phthalate buffer was prepared as follows, 14.2954 g of potassium hydrogen phthalate was mixed with 1.383 g sodium hydroxide and diluted to 2000 mL with HPLC Grade water. The pH 7 phosphate buffer was prepared as follows 10.29254 g of disodium hydrogen phosphate was mixed with 2.6458 g potassium dihydrogen phosphate and diluted to 2000 mL with HPLC Grade water. The pH 9 borate buffer was prepared as follows, 6.1824 g of boric acid was mixed with 7.45 g potassium chloride and 1.6308 g of sodium hydroxide and diluted to 2000 mL with HPLC Grade water. The buffers were sterilised by autoclaving prior to use.

Experimental design

Parameter		Description
Duration of the test		31 days
Buffer condition		Sterilised by autoclaving
Sample size (mL per test vessel)		10 mL
Test concentration (mg ai/mL total buffer)		0.91 mg/mL pH 5, 0.88 mg/mL pH 7 and 0.76 mg/mL pH 9
Control conditions sterility samples		No
Number of replicates		2
Test apparatus		Sterile glass vials, sterilised by autoclaving
Incubation conditions		0.035M pH 5 phthalate buffer, 0.05M pH 7 phosphate buffer, 0.05M pH 9 borate buffer
Traps for CO_2 & organic volatiles		No volatile traps
Test material application	Identity of solvent	The [^{14}C]-labelled aclonifen was dissolved in 500 ml of stock sterile buffers at a nominal concentration of approximately 1 mg/L
	Volume of test solution used/treatment	
Indication of test material adsorbing to walls of test apparatus		As there is a tendency for aclonifen to adsorbed from aqueous solution onto glass and plastic surfaces, during prolonged contact at elevated temperatures. The incubated samples were extracted thoroughly with hexane, the hexane was initially used to wash the interior surfaces of the sample vials.
Experimental conditions	Temperature (°C)	22 ± 1, 50 ± 1 and 70 ± 1°C
	Continuous darkness	Yes
	Agitation	Yes

Sampling

Parameter		Details
Sampling intervals for the parent/transformation products		0, 1, 3, 7, 14 and 31 days
Sampling procedure		The samples were extracted with hexane and an aliquot of the extract was counted by LSC to confirm quantitative recovery of the radioactivity. The samples were concentrated and one of the duplicates was analysed by HPLC, whilst the second was analysed by TLC
Collection of CO_2 and other volatiles		Not collected
Measurements intervals	pH measurement	The pH of each solution was confirmed at sampling.
	Sterility check	Not checked

Description of analytical procedures

Aqueous samples were radioassayed using LSC and analysed by HPLC (co-chromatography with unlabelled aclonifen) to determine the levels of parent and significant degradates in each sample.

II. RESULTS AND DISCUSSION

The recoveries at the beginning and end of the study are summarised below.

Mass Balance

Total radioactivity	Sum of activity in the treatment solutions
Recovery at 0 DAT	116.1% AR
Overall recovery (all samples)	110.1% AR

Volatilisation

$^{14}CO_2$ and other volatiles	No trapping of volatiles

Transformation of Parent Material

Good radiochemical balances were achieved with mean recoveries of applied radioactivity of 112.0, 108.6 and 107.7% for pH 5, 7 and 9, respectively at 22°C, 113.6, 110.1 and 106.3% for pH 5, 7 and 9, respectively at 50°C and 112.2, 111.8 and 108.3% for pH 5, 7 and 9, respectively at 70°C. The pH values of the samples were maintained throughout the study. No check on sterility is reported. The distribution and recovery of aclonifen with time in buffer is presented in Table 7.2.1.1- 1.

Table 7.2.1.1- 1: Distribution and recovery of aclonifen in sterile buffer solutions of pH 5, pH 7 and pH 9 at 22°C, 50°C and 70°C

Sampling interval	% of Applied Radioactivity								
	22°C			50°C			70°C		
	pH 5	pH 7	pH 9	pH 5	pH 7	pH 9	pH 5	pH 7	pH 9
0	106.3	19.5	116.5	127.5	111.0	118.3	109.9	117.6	108.6
1 day	129.3[A]	109.1	105.4	124.9	115.7	106.8	116.4	112.1	115.4
3 day	118.6	116.6	113.1	117.2	113.4	118.9	117.6	113.1	122.9
7 day	113.7	103.9	101.2	107.5	117.1	101.3	114.6	118.1	103.1
14 day	107.4	87.8	103.8	102.3	101.4	96.5	114.0	109.1	102.9
31 day	105.5	101.4	106.4	102.8	102.8	96.2	100.8	100.4	96.6
Mean	112.0	108.6	107.7	113.6	110.1	106.3	112.2	111.8	108.3
Overall mean: 110.1%									

[A] The percentage figure for 22°C, pH 5 (129.16 %) is for a single replicate sample; the result for the second replicate was anomalously high.

The HPLC and TLC analyses of the hexane extracts showed only the presence of unchanged [^{14}C]-aclonifen. No other components were detected in measurable quantities. The LC/MS results confirmed the identity of the aclonifen in the pooled 31 day sample.

III. CONCLUSION

Under sterile aqueous conditions, at temperatures of 22°C, 50°C and 70°C, aclonifen was found to be hydrolytically stable at pH 5, 7 and 9.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD Guideline 111 (1981) and is considered valid to assess the hydrolysis of aclonifen as a function of pH.

Assessment and conclusion by RMS:

CA 7.2.1.2 Direct photochemical degradation

The aqueous photolysis of aclonifen has been investigated in Study KCA 7.2.1.2/01 which was evaluated during the previous EU review and is still considered acceptable. The photolytic degradation of aclonifen in water at pH 7 and at 25 °C occurs only at a very slow rate. No major metabolites are formed by photolysis in water. The quantum yield and environmental photolytic half-life of aclonifen in water have been assessed in documents KCA 7.2.1.2/02 and KCA 7.2.1.2/04. Document KCA 7.2.1.2/03 was submitted for first approval of aclonifen, but has now been superseded by KCA 7.2.1.2/04 as it was found to contain an error.

Report reference	Author, Year	Aniline Label	Phenoxy Label	Comment
KCA 7.2.1.2/01 M-229777-01-1	[redacted], A. & [redacted], 2003	✓	✓	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.2.1.2/02 M-174432-01-1	[redacted] P.	-	-	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.2.1.2/03 M-174430-01-1	[redacted], M.	-	-	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Superseded by M-676286-01-1.
KCA 7.2.1.2/04 M-676286-01-1	[redacted]	-	-	New data not yet reviewed under UP.

Cleavage of aclonifen or its minor soil metabolite M-01 could lead to the formation of phenol (or hydroquinone from cleavage of M-01). A review of the published information on phenol and hydroquinone under photolytic conditions is provided in KCA 7.2.1.2/05 ([redacted], 2019; M-676285-02-1).

Data Point:	KCA 7.2.1.2/01
Report Author:	[redacted]
Report Year:	2003
Report Title:	(14C)-aclonifen : aqueous photolysis at pH 7
Report No:	C031518
Document No:	M-229777-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current guideline: OECD 316 (2008) No deviation
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2017 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive summary:

The photolysis of [^{14}C]-aclonifen was investigated in aqueous solution at pH 7. The study was conducted, under sterile conditions, at 25 ± 2°C, with continuous illumination under artificial sunlight for a period of 16 days (calculated to be equivalent to 30 days of natural summer sunlight in the European Union at latitude 50°N). The irradiation source was a Heraeus suntest with xenon lamp, filtered to remove wavelengths below 290 nm. The mean irradiation intensity (290 to 800 nm) over the duration of the study was measured and found to be 316 W/m^2.

Test solutions were prepared in 0.025M, pH 7 phosphate buffer. The [^{14}C]-aclonifen was added in acetonitrile solution such that the initial concentration of aclonifen was 0.7 mg/L with 0.2% acetonitrile present as a co-solvent.

The test solutions were contained in jacketed vessels of 50 mL capacity with a quartz window. The temperature was maintained at 25 ± 1°C by circulation of coolant through the outer jacket. Each vessel was connected individually to a flow through system, such that moist, CO_2 free, air was passed over the surface of the test solution and out through a series of three traps [ethylene glycol and KOH (x2)], designed to collect any volatile products generated. Single samples were taken at 0, 1, 2, 3, 7, 10, 14 and 16 days. A second set of control samples were similarly set up and incubated in the dark.

Upon sampling, the radioactivity in the aqueous solution was quantified by LSC and analysed directly by HPLC.

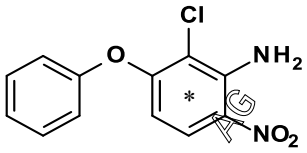
The radioactivity recoveries remained above 93.1% (mean 94.6%) and above 90.4% (mean 95.5%) for the irradiated and non-irradiated samples respectively, throughout the 16 day experiment. No significant quantities of volatile products were collected in either the irradiated experiment (2% of applied radioactivity) or the non-irradiated experiment (0%). The HPLC analysis showed that the amount of aclonifen present in the irradiated samples declined to 87.6% of applied radioactivity after 16 days continuous irradiation. Two minor photodegradates were detected. These were polar in nature and reached maximal values of 5.2% and 1.5% of applied radioactivity after 14 days irradiation. By the final sampling at day 16 these had declined to 4.0% and 0.9%. No degradation of aclonifen was observed in the non-irradiated system

It was concluded that the photolytic degradation of aclonifen in water at pH 7 and at 25°C occurs only at a very slow rate. No major metabolites are formed by photolysis in water. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in SFO DT_{50} values of 105.1 days under irradiated conditions, which was equivalent to 198 days based upon 1 suntest day being equivalent to 1.88 days sunlight in Europe.

I. MATERIALS AND METHODS

A. MATERIALS

1. Test Material: [U-^{14}C-aniline]-aclonifen



* indicates position of ^{14}C radiolabel

Chemical name (IUPAC): 2-chloro-6-nitro-3-phenoxyaniline
Lot or batch number: SEL/1202
Specific radioactivity: 6.59 MBq/mg
Radiochemical Purity: 100%
CA registry number: 74070-46-5
Stability of test compound: Stable, determined within study
2. Buffer: 0.025 M pH 7 phosphate buffer

B. STUDY DESIGN AND METHODS

1. In-life dates:

20 February 2003 – 2 April 2003

2. Test System

This study was performed in 0.025M pH 7 phosphate buffer. The buffer was prepared from 6.8g potassium di-hydrogen phosphate, dissolved in 2L water and adjusted to pH 7.0 + 0.05 with 1M aqueous potassium hydroxide. The buffer was sterilised by filtration through a 0.22 µm Millipore Steritop Sterile filter.

Parameter		Description
Nature of light source		Xenon lamp
Emission wavelength spectrum		300 - 800 nm
Light intensity measurement (300 - 800 nm)		The mean irradiance (300 – 800 nm) over the irradiation period was 300 W/m²
Filters used		UV filter that cuts out wavelengths of < 290 nm
Relationship to natural sunlight		Similar spectral distribution
Duration of the test		16 days
Test system		50 mL of sterile pH 7.0 buffer (sterilised by filtration through a 0.22 µm Millipore Steritop Sterile filter)
Test concentration		0.7 mg L^{-1}
Control conditions		Darkness
Number of replicates	Irradiated	1
	Dark Controls	1
Test apparatus	Irradiated	Water jacketed glass photolysis vessels with quartz lids
	Dark Controls	100 mL volumetric flasks
Traps for CO_2 & organic volatiles		An ethylene glycol trap followed by two potassium hydroxide (2M) traps

Test material application	Identity of solvent	Acetonitrile, 0.2% v/v in final volume
	Volume of application solution	100 µL
	Application method	In order to maintain the sterility of the samples, the treatment was carried out in a laminar flow cabinet. With each treatment the cap was removed from the vessel and the buffer inside was treated with l00 µL of the radio-labelled treatment solution.
	Evaporation of application solvent	Not applicable
Indication of test material adsorbing to walls of test apparatus		None seen
Experimental conditions	Temperature (°C)	25°C
	Continuous irradiation	Yes

Sampling

Parameter	Details
Sampling intervals for the parent/transformation products	0, 1, 2, 3, 7, 10, 14 and 16 days irradiated 0, 1, 2, 3, 7, 10, 14 and 16 days irradiated non-irradiated
Sampling procedure	A single sample was removed at each timepoint and transferred to a measuring cylinder, the volume measured and the radioactivity quantified by LSC of an aliquot. The photolysis vessel was then rinsed with acetonitrile and the radioactive content was similarly quantified by LSC.
Collection of CO_2 and other volatiles	An ethylene glycol trap flowed by two potassium hydroxide (2M) traps.
Sterility check	Samples were checked for sterility at 1, 2, 3, 7, 10, 14 and 16 days and found to be sterile.
Sample storage before analysis	Analysis of test solutions was started on the day of sampling. Solutions not undergoing analysis were stored frozen at - 15°C.

Description of analytical procedures

For the 0, 1, 2 and 3 day samples the aqueous solution and acetonitrile wash were analysed separately, whereas for the 7, 10 14 and 16 day these were combined prior to analysis. The aqueous and mixed aqueous/acetonitrile solutions were analysed directly by HPLC; the separate acetonitrile washes were concentrated prior to analysis. The identity of the aclonifen remaining in these samples was confirmed by comparison with an authentic reference standard. The day 16 samples, both irradiated and non-irradiated, were also analysed by TLC to confirm this structural assignment.

Degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics. DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the current recommendations of the FOCUS work group (2006, 2014) using the software KinGUI (version 2.1). Rate constants for aclonifen degradation were calculated on the assumption of first order kinetics.

II. RESULTS AND DISCUSSION

The total recoveries and distribution of radioactivity are shown in detail in Table 7.2.1.2- 1 to Table 7.2.1.2- 2. The recoveries and trap contents at the beginning and end of the study are summarised below.

Mass Balance

Total radioactivity	Sum of activity in the treated solutions, acetonitrile vessel washings and traps
Recovery at 0 DAT	Dark Control: 97.3% AR
Overall recovery (all samples)	Irradiated: Range 93.1% to 97.3%, Average 94.3% AR Dark controls: Range 90.4% to 97.4%, Average 93.5% AR

Volatilisation

Volatiles	Irradiated: 2.0% AR at the end of the experiment Dark controls: No volatiles seen

Transformation of Parent Material

The HPLC analysis showed that the amount of aclonifen present in the irradiated samples declined to 87.6% of applied radioactivity after 16 days continuous irradiation (Table 7.2.1.2- 3). Two minor photodegradates were detected. These were polar in nature and reached maximal values of 5.2% and 1.5% of applied radioactivity after 14 days irradiation. By the final sampling at day 16 these had declined to 4.0% and 0.9%. No degradation of aclonifen was observed in the non-irradiated system (Table 7.2.1.2- 4).

Table 7.2.1.2- 1: Distribution and recovery of radioactivity, irradiated samples as % of applied radioactivity

	DAT							
Test Point (days)	Initial	1	2	3	7	10	14	16
Equivalent days natural sunlight	0.0	1.9	3.8	5.6	13.2	18.8	26.3	30.0
Water	95.91	89.78	88.48	91.75	90.46	89.89	87.88	88.75
ACN wash	1.38	3.78	4.60	3.77	4.21	3.73	3.35	3.83
Volatile traps	0.00	0.00	0.07	0.14	0.55	0.85	1.99	2.02
Total	97.30	93.51	93.14	95.66	95.23	94.48	93.22	94.60

Table 7.2.1.2- 2: Distribution and recovery of radioactivity, non-irradiated samples as % of applied radioactivity

	DAT							
Test Point (days)	Initial	1	2	3	7	10	14	16
Water	95.91	92.75	86.43	92.97	94.95	93.87	92.70	95.32
ACN wash	1.38	2.97	3.96	2.36	2.06	2.22	2.35	2.10
Volatile traps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total	97.30	95.73	90.39	95.33	97.01	96.09	95.05	97.42

Table 7.2.1.2- 3: Composition of radioactivity by HPLC for the irradiated samples as % of applied radioactivity

	DAT							
Test Point (days)	Initial	1	2	3	7	10	14	16
Total in water & acetonitrile wash	97.30	93.51	93.07	95.52	94.68	93.62	91.23	92.58
Aclonifen	97.30	93.51	93.07	93.85	92.51	90.06	84.47	87.65
Unknown 1 (RRT = 0.09)	n.d.	n.d.	n.d.	0.75	1.71	2.58	5.22	4.02
Unknown 2 (RRT = 0.11)	n.d.	n.d.	n.d.	0.93	0.46	0.98	1.55	0.91

n.d. = none detected, RRT = retention time relative to aclonifen

Table 7.2.1.2- 4: Composition of radioactivity by HPLC for the non-irradiated samples as % of applied radioactivity

	DAT						
Test Point (days)	1	2	3	7	10	14	16
Total in water & acetonitrile wash	95.73	90.39	95.33	97.01	96.09	95.05	97.42
Aclonifen	95.73	90.39	95.33	97.01	96.09	95.05	97.42

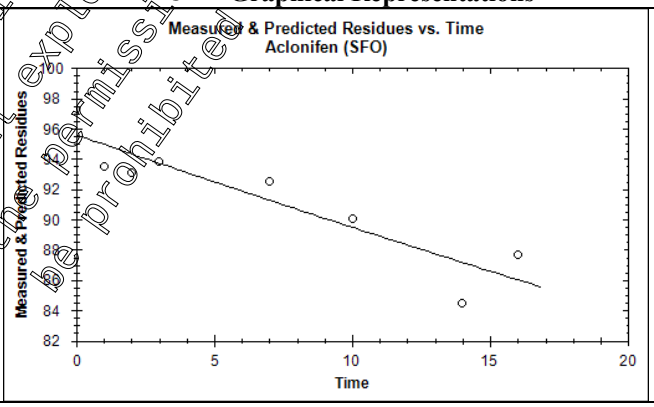
Degradation kinetics

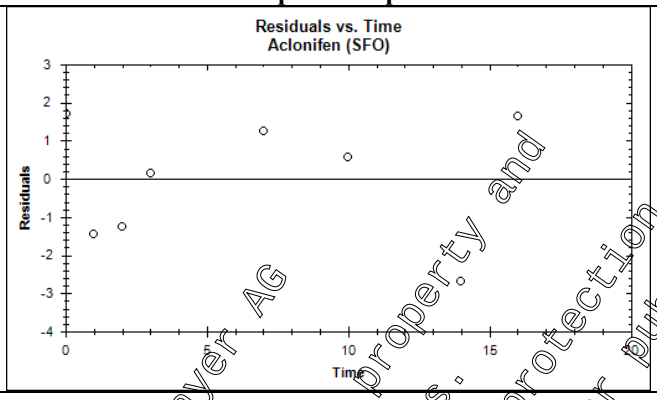
Aclonifen slowly degraded under irradiated conditions. No degradation of [^{14}C]-aclonifen occurred during incubation in the dark. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014) resulted in SFO DT_{50} value of 105.1 days under irradiated conditions.

Table 7.2.1.2- 5: DT_{50} values for aclonifen in aqueous photolysis study

Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	χ^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
SFO	95.6	k = 0.006596	1.3	3.45e-11	0.004247	0.009	105.1	349.1

Table 7.2.1.2- 6: Degradation of aclonifen under photolytic conditions with time

Trial / Best Fit Model	Graphical Representations
Irradiated / SFO	

Trial / Best Fit Model	Graphical Representations
	

The half-life for the decline of aclonifen in the irradiated experiment was calculated to be equivalent to 198 days natural summer sunlight (European Union at latitude 50°N) according to first order kinetics.

III. CONCLUSION

It was concluded that the photolytic degradation of aclonifen in water at pH 7 and at 25°C occurs only at a very slow rate. No major metabolites are formed by photolysis in water.

Assessment and conclusion by applicant:

The study is considered valid to aid assessment of the photodegradation of aclonifen in the environment.

Assessment and conclusion by RMS:

Data Point:	KCA 7.2.1.2/02
Report Author:	[redacted]
Report Year:	1993
Report Title:	Determination of the direct phototransformation of Aclonifen in water
Report No:	R007202
Document No:	M-174432-01-1
Guideline(s) followed in study:	BBA, IV, 6-1 (July 1990); OECD: Draft-test guideline: Phototransformation of Chemicals in Water (January 1990)
Deviations from current test guideline:	Current guideline: Not applicable Current guidance for MoA: SANCO/3029/99 rev.4. (2000) Precision data not reported in full detail
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

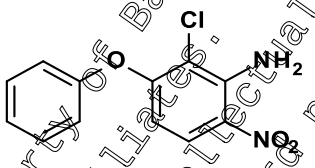
Executive summary:

The quantum yield Φ (PHI) of direct photolysis of aclonifen in aqueous solution was determined using Suntest irradiation apparatus and the computer programme Quantaus. The quantum yield Φ (PHI) was determined to be 5.19 E^{-06}. A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), half-life DT_{50} under the conditions of the experiment of 36.25 hours.

I MATERIALS AND METHODS

A. MATERIALS

1. Test Material: Aclonifen



Chemical name (IUPAC): 2-chloro-6-nitro-3-phenoxyaniline

Lot or batch number: MOY 1521

Chemical Purity: 99.7%

CA registry number: 74070-46-5

Stability of test compound: Stable, determined within study

B. STUDY DESIGN AND METHODS

1. In-life dates:

14 December 1992 - 8 January 1993

2. Test System

A solution of aclonifen in deionised water, containing 1% (v/v) dioxane as co-solvent, was prepared at a concentration of 2.643 mg/L. The irradiation source was a Heraeus Suntest CPS with 1.8kW xenon lamp, filtered to remove wavelengths below 290 nm (maximum irradiance 765 W/m^2). Sub-samples were transferred to irradiation cuvettes (glass with quartz cover). One sample was analysed without irradiation (i.e. zero time sample), whilst the others were placed in the irradiation chamber on a thermostated sample tablet at 20 ± 2°C. Single samples were taken for analysis at 0.25, 1, 2, 6, 24 and 48 hours. The samples were acidified to approximately pH 1 by addition of 6M HCl and were extracted into ethyl acetate. The combined extracts were evaporated to dryness and reconstituted in ethyl acetate. Final quantification was by gas chromatograph with nitrogen-phosphorous detector (NPD).

An actinometer solution, composed of 0.01M uranyl nitrate and 0.05M oxalic acid, was used to determine the number of photons penetrating the test solution cells. The oxalic acid was determined, both before and after irradiation, by titration with an acidic solution of $KMnO_4$ (0.02M). Two actinometer solutions were irradiated, one at the beginning and one at the end of the irradiation experiment. The actinometer solutions were irradiated for 15 minutes.

Degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics. DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the current recommendations of the FOCUS

work group (2006, 2014) using the software KinGUI (version 2.1). Rate constants for aclonifen degradation were calculated on the assumption of first order kinetics.

II. RESULTS AND DISCUSION

The disappearance of aclonifen as a function of irradiation time is given in Table 7.2.1.2- 7.

Table 7.2.1.2- 7: Aclonifen concentration as function of irradiation time

Irradiation time (hours)	0	0.25	1.0	2.0	6.0	24.0	48.0
Aclonifen (mg/L)	2.58	2.46	1.64	1.24	1.86	1.21	0.94

From the analytical results for the actinometric solutions it was determined that the number of photolysed molecules (mean of the two results) was 1.49 x 10^{20} in the 15 minutes exposure. The quantum yield for the actinometer is 0.56 and thus it was possible to determine the number of incident photons and to use this to calculate the quantum yield for aclonifen. This calculation was performed using a computer programme named QUANTAUS (v. 1.10). The quantum yield was determined to be 5.19 x 10^{-6}.

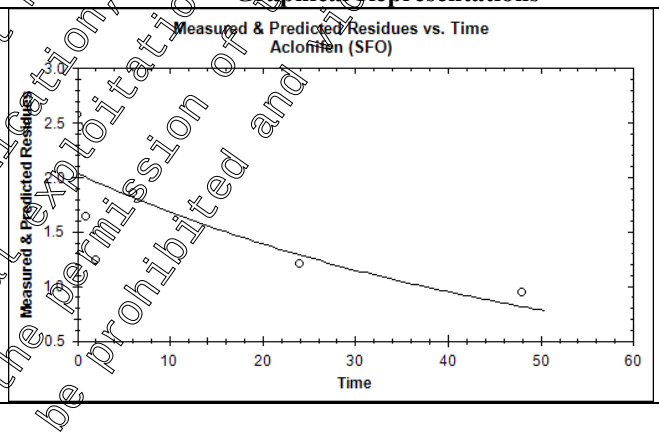
Degradation kinetics

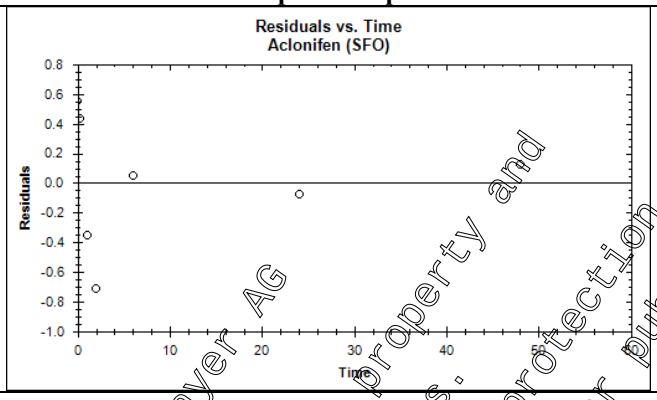
A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in a half-life of 36.25 hours under the conditions of the experiment.

Table 7.2.1.2- 8: DT_{50} values for aclonifen in quantum yield study

Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [hours]	DT_{90} [hours]
SFO	2.03	k = 0.019119	18.9	0.063601	-0.001388	0.040	36.25	120.4

Table 7.2.1.2- 9: Degradation of aclonifen in quantum yield study with time

Trial / Best Fit Model	Graphical Representations
Irradiated / SFO	

Trial / Best Fit Model	Graphical Representations
	

III. CONCLUSION

The quantum yield of direct photolysis of aclonifen in aqueous solution was determined to be 5.10 x 10 ^{6}.

Assessment and conclusion by applicant:

The study is considered valid to aid assessment of the photodegradation of aclonifen in the environment.

Assessment and conclusion by RMS:

KCA 7.2.1.2/03 is a position paper calculating the environmental photolytic half-life of aclonifen in water (M-174430-01-1, 7-cJ)ceajai 1993). It was submitted in the first inclusion of aclonifen into Annex I of Council Directive 91/414/EEC but was subsequently found to contain an error. For procedural reasons it has to be included in the current dossier however it is now superseded by a new statement with the correct calculation. Consequently, the document is not summarised here.

Data Point:	KCA 7.2.1.2/04
Report Author:	[redacted]
Report Year:	2019
Report Title:	Aclonifen - Statement on the environmental photolytic half-life of aclonifen in water
Report No:	VC/19/025B
Document No:	M-676286-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities.
Acceptability/Reliability:	

Executive Summary

The environmental photolytic half-life of aclonifen has been calculated from the quantum yield and molar extinction coefficients in the range 292.5 to 800 nm. This calculation was performed using ABIWAS (v.3.0, Fraunhofer IME). The calculation applies to low concentrations of the chemical in the upper layers of a water body where there is no attenuation of the sunlight intensity. The sunlight data used is appropriate for central Europe (latitude 55°C). The results show that the mean DT_{50} value for the photolytic degradation for Aclonifen ranges from 12.8 days in June to the equivalent of 208 days in December.

I. MATERIALS AND METHODS

The environmental photolytic half-life of aclonifen in water has been calculated using the program ABIWAS (v3.0) as proposed in the OECD 316 guideline: "Phototransformation of Chemicals in Water – Direct Photolysis". The program is based on the original publication of Herrmann *et al*.

ABIWAS 3.0 calculates the abiotic degradation of chemicals in water based on the concentration of the chemical, the molar extinction coefficients of the chemical at appropriate wavelengths and its quantum yield. The implemented standard scenario in ABIWAS 3.0 contains light data based on central Europe (55°) and uses water data without absorption (extinction equal to zero). The results therefore apply to the upper layers of a water body where attenuation of the incident light is minimal.

The spectral data for aclonifen was taken from Jendezejczak N, & [redacted] M., 1992 and the quantum yield of 5.19×10^{-4} from Offizorz P. & Wschiansky A., 1993. A concentration of 0.01 µmol/L was assumed for the calculation, this low concentration being used to minimise light attenuation by the compound.

II. RESULTS AND DISCUSSION

The mean photolytic half-lives calculated for each month are summarised below.

Table 7.2.1.2- 10: Mean calculated $DegT_{50}$ values for aclonifen in upper layer of water body [central Europe (55°)]

Month	days	Month	days	Month	days	Month	days
Jan	131.21	Apr	18.41	Jul	14.43	Oct	47.15
Feb	64.76	May	14.47	Aug	14.88	Nov	109.20
Mar	31.17	Jun	12.85	Sep	25.63	Dec	208.12

The results show that the mean DT_{50} value for the photolytic degradation of aclonifen in the upper layers of a water body in central Europe (latitude 55°C) ranges from 12.8 days in June to the equivalent of 208 days in December.

III. CONCLUSION

The environmental photolytic half-life of aclonifen has been calculated from the quantum yield and molar extinction coefficients in the range 292.5 to 800 nm using ABIWAS (v3.0). The results show that the mean DT_{50} value for the photolytic degradation of aclonifen in the upper layers of a water body in central Europe (latitude 55°C) ranges from 12.8 days in June to the equivalent of 208 days in December.

Assessment and conclusion by applicant:

The statement is considered valid to aid assessment of the photodegradation of aclonifen in the environment.

Assessment and conclusion by RMS:

Data Point:	KCA 7.2.1.2/05
Report Author:	[redacted]
Report Year:	2020
Report Title:	Aclonifen - Statement on the fate of phenol and hydroquinone in the environment
Report No:	AC/19/025H
Document No:	M-676285-02-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	not applicable
Acceptability/Reliability:	

Executive Summary

Any cleavage of the diphenyl ether linkage would result in the formation of phenol and hydroquinone from the phenyl ring. This document summarizes the published information on the fate of phenol and hydroquinone in anaerobic soil and photodegradation in aqueous solutions. The information on the photodegradation in aqueous solutions is summarised here.

Microbial degradation was observed to be a more important transformation process than photolysis for phenol in surface water in both winter and summer. The microbial and photolysis transformation rate constants for phenol were 0.03 h^{-1} (DT_{50} = 28 hours) and 0.016 h^{-1} (DT_{50} = 43 hours), respectively. In winter the decrease in surface irradiance and lower water temperatures result in a decrease in both photolysis and microbial degradation rates.

In natural aquatic systems under dilute environmentally relevant concentrations, direct photolysis of phenol will produce 1,4-hydroquinone and 1,2-hydroquinone (catechol). Photooxidation of phenol with light in the wavelength range of 310–420 nm may be sensitized by humic acid.

Direct photolysis of 1,4- hydroquinone will either give no net change to due recombination of photogenerated radicals, or the radicals will go on to react with other dissolved constituents. The photoproduct 2,2',5,5'-tetrahydroxybiphenyl would not be expected to form at environmentally relevant concentrations.

I. MATERIALS AND METHODS

First an overview of the fate and behaviour of phenol and hydroquinone in surface water, including hydrolysis, mineralization and autoxidation as well as photolytic degradation is provided in the statement. Then fully detailed summaries of five publications are provided as shown below.

Reference 1.	Hwang H-M; Hodson R E; Lee R F Degradation of phenol and chlorophenols by sunlight and microbes in estuarine water Environmental Science and Technology, (1986) Vol. 20, No. 10, pp. 1002-1007.
Reference 2.	Kępczynski, Mariusz; Czosnyka, Alicja; Nowakowska, Maria Photooxidation of phenol in aqueous nanodispersion of humic acid Journal of Photochemistry and Photobiology, A: Chemistry (2007), 185(2-3), 198-205
Reference 3.	Rayne Sierra; Forest Kaya; Friesen Ken J. Mechanistic aspects regarding the direct aqueous environmental photochemistry of phenol and its simple halogenated derivatives. A review. Environment International, (2009 Feb) Vol. 35, No. 2, pp. 425-37. Electronic Publication Date: 18 Oct 2008
Reference 4.	Joschek, H.-I.; Miller, Sidney I.; Photooxidation of phenol, cresols, and dihydroxybenzenes Journal of the American Chemical Society (1966), 88(14), 3273-81
Reference 5.	Boule, P.; Rossi, A.; Pilichowski, J. F.; Grabner, G.; Photoreactivity of hydroquinone in aqueous solution New Journal of Chemistry (1992), 16(11), 1053-62

A short overview of the results pertinent to the fate of phenol and hydroquinone under photolytic conditions is provided below.

II. RESULTS AND DISCUSSION

In a paper by Hwang *et al.* (1986) the rates of photolysis and microbial degradation of phenol were determined in estuarine water. Microbial degradation was the primary process for transformation of phenol. In the summer the microbial and photolysis transformation rate constants for phenol were 0.03 h^{-1} (DT_{50} = 28 hours) and 0.016 h^{-1} (DT_{50} = 43 hours), respectivel. Winter photolysis and microbial degradation rates were lower than the summer values.

Kepczynski *et al.* (2007) studied the photooxidation of phenol sensitized by Aldrich humic acid (AHA) in aqueous solutions at neutral and basic pH. Solutions containing phenol and humic acid of various concentrations were irradiated with monochromatic light at 253.7 nm or with polychromatic light within the wavelength range of 310-420 nm. The quantum yields of phenol photodegradation under these conditions were determined. At the wavelength of 253.7 nm direct degradation. of phenol was much more effective than that sensitized by humic acid. With polychromatic light the photooxidation was found to be strongly dependent on pH of aqueous solution and independent on humic acid concentration.

In Rayne, Forest *et al*., (1992) the aqueous photochemistry of phenol and its simple halogenated derivatives, including 1,4-hydroquinone were reviewed. Two key publications were reviewed for phenol and hydroquinone (Joschek and Miller (1966); Boule *et al*., (1992)). In natural aquatic systems under dilute environmentally relevant concentrations, direct photolysis of phenol will product 1,4 hydroquinone and 1,2-hydroquinone (catechol). Direct photolysis of 1,4- hydroquinone will either give no net change to due recombination of photogenerated radicals, or the radicals will go on to react with other dissolved constituents.

The key publications were also summarised individually. In Joschek & Miller (1966) it was established that continuous irradiation of aqueous solutions of phenol with a mercury lamp in the presence of oxygen, dinitrogen oxide and cupric acetate or under nitrogen yielded rather similar products. Seven photodegradation products of phenol were identified. Irradiation of hydroquinone produced only one dimerization product.

In Boule *et al.* (1992) it was established in the absence of oxygen, the quantum yield of hydroquinone is very low (0.002). The main photoproduct was 2,5,2',5'-tetrahydroxybiphenyl (THBP) from the reaction of excited hydroquinone with benzoquinone. In air-saturated solution the main photoproducts were benzoquinone, hydroxybenzoquinone and hydrogen peroxide. The quantum yield for the phototransformation of hydroquinone was higher with NO_3^- as the oxidant than with O_2. Oxidation by NO_3^- was inhibited by O_2 and lead mainly to benzoquinone. It was observed that hydroquinone could photosensitize the conversion of 3-chlorophenol to resorcinol, which was attributed to a triplet-triplet energy transfer. 3-chlorophenol also inhibited the formation of THBP and the oxidation of hydroquinone by NO_3^-. These results point to the hydroquinone triplet state as intermediate in all these reactions. The triplet-triplet absorption of hydroquinone and its reactions with O_2 and NO_3^- were observed by nanosecond flash photolysis. In-cage recombination of semiquinone and superoxide radicals produced in triplet quenching by O_2 was proposed to account for the formation of hydroxybenzoquinone.

III. CONCLUSION

Any cleavage of the diphenyl ether linkage would result in the formation of phenol and hydroquinone from the phenyl ring. A review of the published data on phenol and hydroquinone enabled conclusions on their fate and behaviour in the environment to be made. Neither compound will persist or accumulate in the environment under photolytic conditions.

Assessment and conclusion by applicant:

The position paper is considered valid to aid assessment of the photodegradation of phenol and hydroquinone in aqueous solutions.

Assessment and conclusion by RMS:

CA 7.2.1.3 Indirect photochemical degradation

An assessment of indirect photochemical degradation is an optional higher tier study which is not required for aclonifen.

CA 7.2.2 Route and rate of biological degradation in aquatic systems

CA 7.2.2.1 "Ready biodegradability"

The ready biodegradability of aclonifen has been investigated in study KCA 7.2.2.1/01 which was evaluated during the previous EU review and is still considered acceptable.

Report reference	Author, Year	Comment
KCA 7.2.2.1/01 M-218236-01-2	[redacted], 1990	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable

Data Point:	KCA 7.2.2.1/01
Report Author:	[redacted]
Report Year:	1990
Report Title:	Ready biodegradability of Aclonifen with activated sludge (28 days)
Report No:	R003640
Document No:	M-218236-01-2
Guideline(s) followed in study:	OECD: 301 B
Deviations from current test guideline:	Current Guideline: OECD: 301 B None
Previous evaluation:	yes, evaluated and accepted Source: Study first relied upon, December 2006 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive summary:

The ready biodegradability of aclonifen was determined according to the Sturm test (OECD guideline 301B).

Aclonifen was incubated in the test medium, inoculated with activated sludge (from a municipal purification plant at [redacted]), at concentrations of 5 and 10 mg/L. The released carbon dioxide was monitored for a period of 28 days and quantified by precipitation as $BaCO_3$ followed by back titration of $Ba(OH)_2$ with 0.05 M HCl. A parallel experiment was performed using sodium acetate as a reference substance to validate the test results.

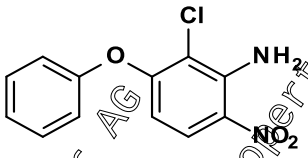
The test was valid (ready biodegradability of the reference substance > 60%). With a test result of < 60% for aclonifen after 28 days, the compound was classified as being not readily biodegradable.

Aclonifen was found to be not readily biodegradable.

I. MATERIALS AND METHODS

A. MATERIALS

Test Material: Aclonifen



Test material: 2-chloro-6-nitro-3-phenoxy-aniline

Lot/Batch #: DA 618

Purity: 91.3% w/w

Appearance: White - yellow

Stability of test compound: Shown to be stable under the conditions of the test

B. STUDY DESIGN AND METHODS

1. In-life dates:

08 August 1990 – 05 September 1990

2. Experimental design:

Aclonifen was incubated in the test medium, inoculated with activated sludge (from a municipal purification plant at [redacted]) at concentrations of 5 and 10 mg/L. The released carbon dioxide was monitored for a period of 28 days and quantified by precipitation as $BaCO_3$ followed by back titration of $Ba(OH)_2$ with 0.05 M HCl. A parallel experiment was performed using sodium acetate as a reference substance to validate the test results.

II. RESULTS AND DISCUSSION

The results, expressed as a percentage of the maximum theoretical CO_2 production, for both aclonifen and the reference substance (sodium acetate) are shown in Table 7.2.2.1- 1.

Table 7.2.2.1- 1: Ready biodegradability expressed as percentage of maximum theoretical CO2 production

Time (days)	Aclonifen (5 mg/L)	Aclonifen (10 mg/L)	Sodium acetate (20 mg/L)
7	9%	4%	70%
16	15%	0%	70%
28	22%	0%	74%

The test was valid (ready biodegradability of the reference substance > 60%). With a test result of < 60% for aclonifen after 28 days, the compound was classified as being not readily biodegradable.

III. CONCLUSION

Aclonifen was found to be not readily biodegradable.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD Guideline and is considered valid to assess the ready biodegradability of aclonifen.

Assessment and conclusion by RMS:

CA 7.2.2.2 Aerobic mineralisation in surface water

An assessment of aerobic mineralisation in surface water is new study requirement under Regulation 1107/2009. Two aerobic mineralisation studies (OECD 309) with aclonifen were performed for Annex I Renewal, a 'pelagic' test system (KCA 7.2.2.2/01) representative of the water column of the open waters or oceans and a 'suspended sediment' test system (KCA 7.2.2.2./02) representative of most surface waters according to OECD Test Guideline 309.

In the 'pelagic' test system the aerobic mineralisation of aclonifen was investigated in natural water at pH 7.1. The results indicated that aclonifen was slowly degraded in both low and high concentration tests but did not significantly mineralise (<1% AR) over the study duration. DT_{50} values for aclonifen in pelagic water were 205.5 and 361 days. The aclonifen metabolite M-01 was formed at a maximum of 10% AR along with 3 other minor unidentified metabolites (<3.5% AR).

However exposure of aclonifen to open water is not expected as the compound is very strongly adsorbed (mean K_{oc} > 5500) & immobile in soil. Any residues unintentionally reaching surface waters will not reach open water such as lakes, reservoirs, estuaries or the sea.

In the 'suspended sediment' test system the aerobic mineralisation of aclonifen was investigated in natural water at pH 6.9. The results indicated that aclonifen was readily metabolised in both low and high concentration tests but did not significantly mineralise (<5% AR) over the study duration. DT_{50} values for aclonifen in suspended sediment water were 25.7 and 39.2 days. The aclonifen metabolite M-02 was formed at a maximum of 17% AR in flasks treated with [aniline-UL-^{14}C]-aclonifen. No significant metabolites were observed in flasks treated with [phenoxy-UL-^{14}C]-aclonifen.

Studies KCA 7.2.2.2/01 and KCA 7.1.1.1/02 are provided as new data not yet reviewed under uniform principles.

Report reference	Author, Year	Aniline Label	Phenoxy Label	Comment
KCA 7.2.2.2/01 M-551820-01-1	[redacted] & [redacted] D., 2016	✓	✓	New data not yet reviewed under UP.
KCA 7.2.2.2/02 M-674035-01-1	[redacted] & [redacted] 2020	✓	✓	New data not yet reviewed under UP.

Data Point:	KCA 7.2.2.2/01
Report Author:	[redacted]; [redacted]
Report Year:	2016
Report Title:	[Aniline-UL-14C]aclonifen: Aerobic mineralization in surface water
Report No:	MECLN007
Document No:	M-551820-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 309 Commission Regulation (EU) No 283/2013 in accordance with Regulation (EC) No 1107/2009 US EPA OCSPP Guideline No 835.SUPP
Deviations from current test guideline:	Current guideline: OECD 309 (2004) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of [aniline-UL-^{14}C]-aclonifen were studied in surface water under aerobic conditions (pelagic). Two test concentrations were incubated in the laboratory in the dark at 20 ± 2 °C for 62 days.

Two study application rates were used which included 10.7 µg aclonifen/L and 100.8 µg aclonifen/L surface water for low and high concentration test systems, respectively.

The test systems consisted of 250-mL flasks each containing 100 mL of surface water equipped with traps (permeable to oxygen) for the collection of carbon dioxide and volatile organic compounds. The surface water in the test systems was kept in motion during the entire study period to maintain aerobicity.

Duplicate samples were analysed at 0, 7, 15, 22, 29, 41, 48 and 62 days of incubation. Additionally, sterile test systems were processed and analysed at 62 days after treatment. The concentration of aclonifen and degradation products in surface water were determined by liquid scintillation counting (LSC) and by HPLC/ radiodetection analysis. The redox potential, pH and oxygen content of the surface water were measured throughout the study on 0, 7, 15, 22, 29, 41, 48 and 62 days.

Mean material balances were 99.3% AR for low concentration test systems (range from 97.2 to 101.0% AR) and 98.4% AR for high concentration test systems (range from 97.3 to 99.8% AR).

In low concentration test systems, the mean amount of aclonifen in surface water decreased from day 0 to day 62 from 99.8 to 84.2% AR. In high concentration test systems, the mean amount of aclonifen in surface water decreased from day 0 to day 62 from 99.8 to 88.5% AR. In sterile samples, the mean amount of aclonifen in surface water at day 62 was 98.9% AR in low concentration test systems and 100.2% AR in high concentration test systems.

For low concentration samples the mean amount of carbon dioxide increased throughout the study to a maximum of 0.8% AR at day 62. For high concentration samples, the mean amount of carbon dioxide increased throughout the study to a maximum of 0.5% AR at day 62.

Besides the formation of carbon dioxide, one degradation product was identified as M-01 (called AE 0561851 in the report) and occurred at a maximum of 9.9% AR (day 41, low concentration). In high concentration samples M-01 was identified with a maximum occurrence of 5.3% AR (day 48). The total unidentified residues amounted to a maximum of 6.5% and 4.9% AR for the low and high

concentrations, respectively, with no individual component exceeding 3.5% AR. In sterile samples, no degradation products were formed. This indicates that all degradation was the result of microbial activity within each test system.

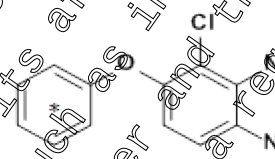
The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. The experimental data was best described by the simple first order (SFO) kinetic model. The DT_{50} values of aclonifen in the tested surface water under aerobic conditions were 205.5 and 361 days for low and high concentrations, respectively. Aclonifen is degraded slowly in both low and high concentration samples under conditions representative of the water column of waters or oceans.

However exposure of aclonifen to open water is not expected as it is very strongly adsorbed (K_{oc} > 5000) and immobile in soil. Any residues unintentionally reaching surface waters will not reach open water such as lakes, reservoirs, estuaries or the sea.

I MATERIALS AND METHODS

A. MATERIALS

1. Test material: [Aniline-UL-^{14}C]-aclonifen



* Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC)	2-chloro-6-nitro-3-phenoxy-aniline
CA registry number:	74070-46-5
Lot or batch number:	C-1193
Specific activity:	47.139 mCi/mMole
Radiochemical purity:	99%
Stability of test compound:	Shown to be stable under the conditions of the test

2. Water: Surface water was freshly collected from Beaver Dam Lake, Wake Forest, North Carolina, USA in August 2015. Prior to use, it was sieved through a 0.001 mm sieve. The water was stored for four days prior to the start of the equilibration.

Table 7.2.2.2- 1: Physicochemical Parameters of the Water/Sediment Systems

Parameter	Beaver Dam, NC
Geographic Location	Beaver Dam Lake, Wake Forest, North Carolina, USA
Batch	082715-W
GPS Coordinates	[redacted]

Water Temperature [°C]	28.9
pH	7.14
Redox Potential @ 21.0 °C, pH 6.9 E_{obs} [mV]	252.6
Oxygen saturation [mg/L]	6.5
Total Organic Carbon (TOC) [ppm]	7.6
Dissolved Organic Carbon (DOC) [ppm]	7.3
Biochemical Oxygen Demand (BOD_5) [mg/L]	2.2
Total Nitrogen [ppm]	0.7
Total Phosphorus [ppm]	0.8

B. STUDY DESIGN

In-life dates: June 2017 – November 2017

Experimental conditions

Parameter	Description
Duration of test	62 days
Water conditions	Fresh surface water
Water sample Volume	100 mL

Parameter		Description
Concentration in test system	Nominal [µg test substance / test system]	1 (low concentration samples) and 10 (high concentration samples)
	Actual [µg test substance / test system]	1.1 (low concentration samples) and 10.1 (high concentration samples)
Control conditions	Microbial activity samples	Samples for determination of microbial activity: surface water treated with control substance (0.9 µg/test system, corresponding to 9.5 µg/L)
	Sterile samples	Sterilized test systems treated with test substance (1.1 and 10.1 µg/test system
Number of replications	Degradation samples	Duplicate samples for each sampling interval
	Microbial activity samples	Duplicate samples for each sampling interval (Bio + and Bio -)
	Sterile samples	Duplicate samples for each sampling interval
Test apparatus		250 mL glass flasks
Traps for volatiles		The traps were filled with soda lime and polyurethane foam plug. The traps were permeable for oxygen.
Test material application (including sterile samples)	Identity of solvent	Methanol
	Volume of application solution	Low concentration: 110 µL of "Low Conc" per test system High concentration: 130 µL of "High Conc" per test system
	Application method	Application to the water surface using a gastight syringe
	Evaporation of application solvent	Treatment solutions created by evaporating standard to dryness, then diluting to final volume with methanol for degradation treatment solutions and water for microbial activity treatment solution.
Control substance application (microbial activity samples)	Solvent of application solution of control substance	Water
	Volume of application solution (control substance) used per treatment	100 µL per test system
	Application method	Application to the water surface using a gastight syringe
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	19.4°C
	Lighting	Dark
Other details		The water was kept in smooth motion. Sterile samples were autoclaved.

Sampling

Parameter		Details
Sampling intervals	Degradation samples	Duplicate test systems of each test concentration were processed and analysed at 0, 7, 15, 22, 29, 41, 48, and 62 days after treatment (DAT).
	Sterile samples	62 DAT
	Microbial activity samples	0 and 14 DAT
Water system sampling procedures		See below
Collection of CO_2 and other volatiles		Soda lime for absorption of carbon dioxide and polyurethane foam for adsorption of volatile organic compounds.
Sampling intervals / times for	Redox potential, oxygen saturation and pH	At each sampling interval
	TOC, DOC, total nitrogen, total phosphorus, BOD5	At start of the study for untreated surface water
	Sterility checks	Sterile samples at DAT 62
	Other	N/A
Sample storage before analysis		Samples were initially analysed by LSC and HPLC/radiodetection within two days after sampling [A]. Therefore no storage stability investigations were necessary.

Description of analytical procedures

At each sampling interval an aliquot of water was initially removed, and the concentration of any dissolved carbon dioxide determined. The radioactive content of the water was then determined by LSC. Once the physical measurements had been determined the water phase was transferred to a measuring cylinder and the test vessel rinsed with acetonitrile. The acetonitrile washes were then combined with the water sample. The amounts of test item and degradation products in the water extracts were determined by HPLC/radiodetection analysis. The limit of detection for the primary chromatographic method was determined to be 2.7% AR.

Degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2.

II. RESULTS AND DISCUSSION

A. Mass Balance

Mean material balances were 99.3% AR for low concentration test systems (range from 97.2 to 101.0% AR) and 98.4% AR for high concentration test systems (range from 97.3 to 99.8% AR). A summary of the recoveries at each sampling time interval is provided in Table 7.2.2.2- 2 and Table 7.2.2.2- 3.

B. Findings

In low concentration test systems, the mean amount of aclonifen in surface water decreased from day 0 to day 62 from 99.8 to 84.2% AR. In high concentration test systems, the mean amount of aclonifen in surface water decreased from day 0 to day 62 from 99.8 to 88.5% AR. In sterile samples, the mean amount of aclonifen in surface water at day 62 was 98.9% AR in low concentration test systems and 100.2% AR in high concentration test systems (Table 7.2.2.2- 4 and Table 7.2.2.2- 5).

Degradation of aclonifen was accompanied by the formation of one major degradation product, identified as M-01 and occurred at a maximum of 9.9% AR (day 41, low concentration). In high

concentration samples the degradation product M-01 was identified with a maximum occurrence of 5.3% AR (day 48). The total unidentified residues amounted to a maximum of 6.5% and 4.9% AR for the low and high concentrations, respectively, with no individual component exceeding 3.5% AR. In sterile samples, no degradation products were formed. This indicates that all degradation was the result of microbial activity within each test system.

For low concentration samples the mean amount of carbon dioxide increased throughout the study to a maximum of 0.8% AR at day 62. For high concentration samples, the mean amount of carbon dioxide increased throughout the study to a maximum of 0.5% AR at day 62. In sterile samples the mean amount of carbon dioxide formed after 62 days was ≤ 0.1% AR at both concentrations. Formation of volatile organic compounds (VOC) was insignificant as demonstrated by values of ≤0.1% AR at all sampling intervals for both concentrations, as well as in the sterile samples.

Table 7.2.2.2- 2: Material balance of radioactivity in surface water under aerobic conditions at low concentration (including sterile samples) expressed as percentage of applied radioactivity

	Rep No.	DAT								
		0	**7**	**15**	**22**	**29**	**41**	**48**	**62**	**62 sterile**
Volatiles										
Carbon Dioxide	A	n.a	0.1	0.2	0.3	0.1	0.5	0.6	0.8	0.1
	B	n.a	0.1	0.2	0.3	0.1	0.5	0.6	0.9	0.1
	Mean	**n.a**	**0.1**	**0.2**	**0.3**	**0.1**	**0.5**	**0.6**	**0.9**	**0.1**
Volatile Organic Compounds	A	n.a	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
	B	n.a	0.1	0.1	0.1	0.1	0.1	0.2	0.1	0.1
	Mean	**n.a**	**0.1**	**0.1**	**0.1**	**0.1**	**0.1**	**0.1**	**0.1**	**0.1**
Total Volatiles	A	n.a	0.2	0.3	0.5	0.2	0.6	0.7	0.9	0.2
	B	n.a	0.2	0.2	0.5	0.1	0.6	0.8	1.0	0.2
	Mean	**n.a**	**0.2**	**0.2**	**0.5**	**0.2**	**0.6**	**0.8**	**0.9**	**0.2**
Water [A]	A	101.9	101.6	95.8	96.9	100.4	98.1	99.6	98.2	99.0
	B	97.7	100.0	98.2	98.9	98.6	97.9	99.8	98.0	98.8
	Mean	**99.8**	**100.8**	**97.0**	**97.9**	**99.5**	**98.0**	**99.7**	**98.1**	**98.9**
Material Balance	A	101.9	101.8	96.0	97.4	100.6	98.7	100.3	99.1	99.2
	B	97.7	100.2	98.4	99.3	98.8	98.5	100.6	99.0	99.0
	Mean	**99.8**	**101.0**	**97.2**	**98.4**	**99.7**	**98.6**	**100.4**	**99.0**	**99.1**
Overall Mean 9.3 ± 1.2 (excludes sterile samples)										

n.a.: not analysed, DAT: days after treatment
[A] Includes acetonitrile rinse, min 1.2% to max 4.8% of AR

Table 7.2.2.2- 3: Material balance of radioactivity in surface water under aerobic conditions at high concentration (including sterile samples) expressed as percentage of applied radioactivity

	Rep No.	DAT								
		0	7	15	22	29	41	48	62	62 sterile
Volatiles										
Carbon Dioxide	A	n.a	<0.1	0.1	0.2	0.2	0.3	0.2	0.5	<0.1
	B	n.a	<0.1	0.1	0.2	0.3	0.3	0.4	0.5	0.1
	Mean	**n.a**	**<0.1**	**0.1**	**0.2**	**0.3**	**0.3**	**0.3**	**0.5**	**<0.1**
Volatile Organic Compounds	A	n.a	<0.1	<0.1	<0.1	0.1	<0.1	<0.1	0.1	0.1
	B	n.a	<0.1	<0.1	<0.1	0.1	<0.1	<0.1	<0.1	0.1
	Mean	**n.a**	**<0.1**	**<0.1**	**<0.1**	**0.1**	**<0.1**	**<0.1**	**0.1**	**0.1**
Total Volatiles	A	n.a	0.1	0.1	0.2	0.3	0.4	0.3	0.6	0.1
	B	n.a	0.1	0.1	0.2	0.4	0.3	0.4	0.5	0.1
	Mean	**n.a**	**0.1**	**0.1**	**0.2**	**0.3**	**0.4**	**0.3**	**0.6**	**0.1**
Water [A]	A	97.6	98.9	97.3	97.8	97.9	98.3	96.8	99.0	96.6
	B	101.9	98.9	98.8	96.8	98.6	97.8	97.2	96.5	103.9
	Mean	**99.8**	**98.9**	**98.1**	**97.3**	**98.2**	**98.0**	**97.0**	**97.8**	**100.2**
Material Balance	A	97.6	98.9	97.4	98.0	98.2	98.7	97.0	99.7	96.7
	B	101.9	98.9	98.9	97.0	98.9	98.1	97.7	97.1	104.0
	Mean	**99.8**	**98.9**	**98.2**	**97.5**	**98.6**	**98.4**	**97.3**	**98.4**	**100.3**
Overall Mean 98.4 ± 0.7 (excludes sterile samples)										

n.a.: not analysed, DAT: days after treatment
[A] Includes acetonitrile rinse, min 1.2% to max 4.8% of AR

Table 7.2.2.2- 4: Degradation of aclonifen in system in surface water under aerobic conditions at low concentration (including sterile samples)

Compound	DAT (Mean % AR ± SD)								
	0	7	15	22	29	41	48	62	62 sterile
Aclonifen	99.8 ± 2.1	100.8 ± 0.8	97.0 ± 1.2	93.5 ± 1.4	93.2 ± 2.4	86.1 ± 2.5	84.4 ± 2.3	84.2 ± 5.1	98.9 ± 0.1
M-01 [A]	n.d.	n.d.	n.d.	4.4 ± 0.4	6.3 ± 1.5	9.9 ± 0.6	8.8 ± 1.5	4.4 ± 0.6	n.d.
Unknown 1 [B]	n.d.	n.d.	n.d.	n.d.	n.d.	1.2 ± 0.0	2.2 ± 0.2	2.6 ± 0.0	n.d.
Unknown 2 [C]	n.d.	n.d.	n.d.	n.d.	n.d.	0.8 ± 0.0	3.0 ± 0.8	2.6 ± 0.2	n.d.
Unknown 3 [D]	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1.3 ± 0.0	n.d.	n.d.
Sum Unidentified Residues	n.d.	n.d.	n.d.	< LOD	LOD	2.0 ± 0.0	6.5 ± 0.8	5.1 ± 2.8	n.d.
Total Water Residues	99.8 ± 2.1	100.8 ± 0.8	97.0 ± 1.2	97.9 ± 1.0	99.5 ± 0.9	98.0 ± 0.1	99.7 ± 0.1	98.1 ± 0.1	98.9 ± 0.1
Carbon Dioxide	n.a.	0.1 ± 0.0	0.2 ± 0.0	0.3 ± 0.0	0.1 ± 0.0	0.5 ± 0.0	0.6 ± 0.0	0.8 ± 0.1	0.1 ± 0.0
Volatile Organic Cpds [E]	n.a.	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0
Material Balance	99.8 ± 2.1	101.8 ± 0.0	97.2 ± 1.2	98.4 ± 1.0	99.7 ± 0.9	98.6 ± 0.1	100.4 ± 0.1	99.0 ± 0.0	99.1 ± 0.1

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
[A] Called AE 0561851 in the report
[B] Called Minor Degradate 1 in the report
[C] Called Minor Degradate 2 in the report
[D] Called Minor Degradate 3 in the report
[E] Sum of radioactivity in soda lime and polyurethane foam

Table 7.2.2.2- 5: Degradation of aclonifen in system in surface water under aerobic conditions at high concentration (including sterile samples).

Compound	DAT (Mean % AR ± SD)								
	0	7	15	22	29	41	48	62	62 sterile
Aclonifen	99.8 ± 2.1	98.8 ± 0.0	98.1 ± 0.8	94.4 ± 0.5	92.9 ± 1.4	96.0 ± 1.8	88.9 ± 0.0	88.5 ± 3.2	100.2 ± 3.7
M-01 [A]	n.d.	n.d.	n.d.	3.0 ± 0.0	3.3 ± 0.1	2.1 ± 0.0	5.3 ± 1.1	4.4 ± 0.2	n.d.
Unknown 1 [B]	n.d.	n.d.	n.d.	n.d.	1.3 ± 0.1	n.d.	2.1 ± 0.0	5.5 ± 0.3	n.d.
Unknown 2 [C]	n.d.	n.d.	n.d.	n.d.	0.8 ± 0.0	n.d.	1.8 ± 0.2	1.5 ± 0.0	n.d.
Sum Unidentified Residues	n.d.	n.d.	n.d.	LOD	2.1 ± 1.0	< LOD	2.8 ± 0.9	4.9 ± 1.8	n.d.
Total Water Residues	99.8 ± 2.1	98.8 ± 0.8	98.1 ± 0.8	97.3 ± 0.5	98.2 ± 0.3	98.0 ± 0.2	97.0 ± 0.2	97.8 ± 1.3	100.2 ± 3.7
Carbon Dioxide	n.a.	0.1 ± 0.0	0.1 ± 0.0	0.2 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.3 ± 0.1	0.5 ± 0.0	< 0.1 ± 0.0
Volatile Organic Cpds [D]	n.a.	0.1 ± 0.0	< 0.1 ± 0.0	< 0.1 ± 0.0	0.1 ± 0.0	< 0.1 ± 0.0	< 0.1 ± 0.0	0.1 ± 0.0	0.1 ± 0.0
Material Balance	99.8 ± 2.1	98.8 ± 0.0	98.2 ± 0.8	97.5 ± 0.5	98.6 ± 0.4	98.4 ± 0.3	97.3 ± 0.3	98.4 ± 1.3	100.3 ± 3.7

n.a.: not analysed, n.d.: not detected, DAT: days after treatment
[A] Called AE 0561851 in the report
[B] Called Minor Degradate 1 in the report
[C] Called Minor Degradate 2 in the report
[D] Sum of radioactivity in soda lime and polyurethane foam

Degradation kinetics

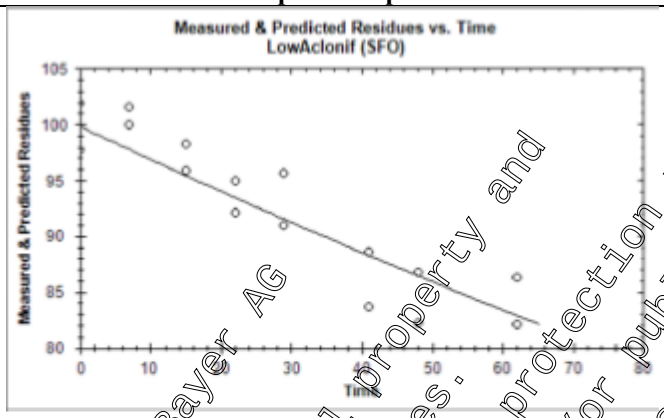
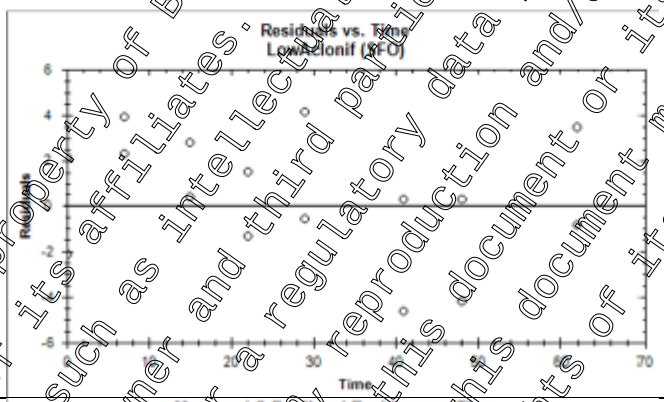
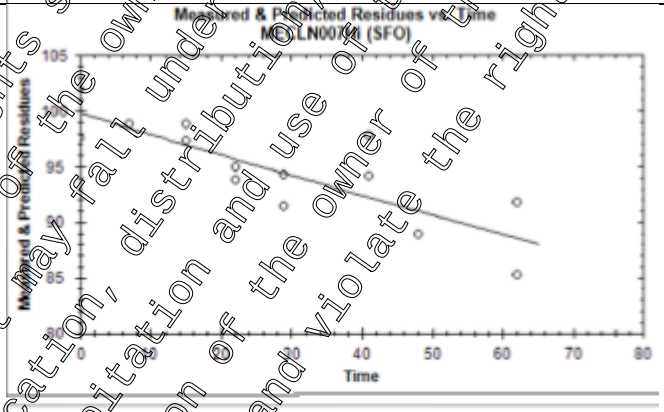
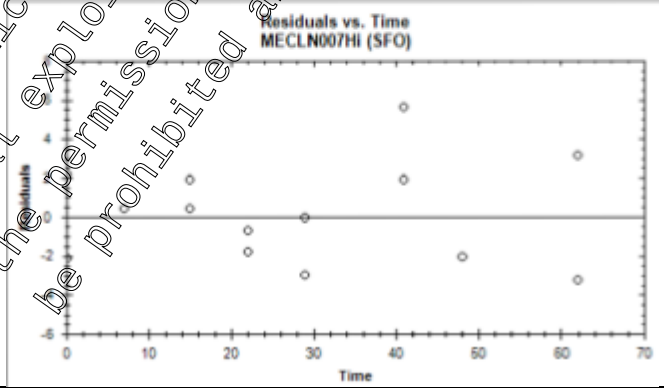
The reported DT_{50} values are summarised in Table 7.2.2.2- 6. The degradation of aclonifen in the water followed simple first order (SFO) kinetics in both systems according to the lowest chi^2 error values and visual assessments.

Table 7.2.2.2- 6: Summary of the kinetic evaluation of the degradation of aclonifen in water systems under aerobic conditions

Test system	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	DT_{50} [days]	DT_{90} [days]
Low	SFO	99.8	3.373E-03	1.36	1.45E-07	205.5	682.7
High	SFO	99.8	0.0019203	1.37	3.25E-08	361	>1000

Table 7.2.2.2- 7: Degradation of aclonifen in water systems under aerobic conditions with time

Trial / Best Fit Model	Graphical Representations

Trial / Best Fit Model	Graphical Representations
Low concentration / SFO	 
High concentration / SFO	 

III. CONCLUSION

Aclonifen was degraded at both the low and high test concentrations. The DT_{50} values of aclonifen in the tested surface water under aerobic conditions were 205.5 and 361 days, respectively.

Formation of volatiles such as carbon dioxide was minimal and reached a maximum of 0.9 % AR at day 62 in the lower concentration.

Apart from carbon dioxide, one metabolite was identified as M-01 which occurred at a maximum of 9.9% AR (at day 41, low concentration). In high concentration samples M-01 was detected at a maximum of 5.3% AR (day 48). The total unidentified residues amounted to a maximum of 6.5% and 4.9% AR for the low and high concentrations, respectively, with no individual component exceeding 3.5% AR.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD Test Guideline No. 309 in a pelagic test system. Aclonifen was degraded slowly in both concentration under conditions representative of the water column of open waters or oceans. However exposure of aclonifen to open water is not expected as it is very strongly adsorbed (K_{oc} > 5000) and immobile in soil. Any residues unintentionally reaching surface waters will not reach open water such as lakes, reservoirs, estuaries or the sea. No drainage from fields is expected. Any exposure will be limited to unintended contamination by spray drift or runoff to surface waters such as ditches, ponds or streams bordering fields in which the compound is applied. The study is not considered appropriate to assess the persistence of aclonifen in water bodies to which the compound may reasonably be expected to be exposed to.

Assessment and conclusion by RMS:

Data Point:	KCA 7.2.2.2/02
Report Author:	[redacted]
Report Year:	2020
Report Title:	Aclonifen: Aerobic mineralization in surface water (suspended sediment test)
Report No:	EnSa-19-0252
Document No:	M-674035-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 309 (2004); Commission Regulation (EU) No 283/2013 in accordance with Regulation (EC) No 1107/2009 (2013)
Deviations from current test guideline:	Current guideline: OECD 309 (2004) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of [aniline-UL-^{14}C]aclonifen were studied at two test concentrations in turbid surface water [redacted], Germany (water pH 6.9, total organic

carbon < 2.0 mgL,) under aerobic conditions (suspended sediment test) in the laboratory in the dark at 20 ± 2 °C for 60 days.

A nominal application rates of 1 µg/L and 10 µg/L were applied for low and high concentration samples, respectively.

The test was performed in test systems consisting of Erlenmeyer flasks with baffles each containing 100 mL of surface water, supplemented with approx. 0.33 g of sediment, and equipped with traps (permeable for oxygen) for the collection of carbon dioxide and volatile organic compounds. The surface water in the test systems was kept in motion during the entire study period.

Duplicate samples of each test concentration were processed and analysed 0, 1, 3, 7, 14, 30 and 60 days after treatment (DAT). Sterile samples were processed at study end (DAT-60) for both concentrations.

At each sampling interval, water/sediment suspension was at first transferred and extracted at ambient temperature with acetonitrile (in the following referred to as suspension extract). Afterwards, the sediment was extracted at ambient temperature twice with acetonitrile/water 1/1 (*v/v*) and once each with acetonitrile and methanol, respectively. The amounts of test item and degradation products in suspension extract and sediment extracts were determined by liquid scintillation counting (LSC) and by HPLC/radiodetection analysis. The amounts of volatiles and non-extractable residues were determined by LSC and combustion/LSC, respectively.

To complete the understanding of the degradation behaviour of aclonifen in surface water under aerobic conditions, additional test systems were treated with [phenoxy-UL-^{14}C]- aclonifen and processed 49 and 61 days after treatment.

For flasks treated with [aniline-UL-^{14}C]-aclonifen, mean material balances were 98.3% AR for low concentration test systems (range from 91.4 to 110% AR) and 93.4% AR for high concentration test systems (range from 81.3 to 105% AR). Mean material balances of sterile samples were 110% AR for low concentration test systems and 119% AR for high concentration test systems.

The amounts of carbon dioxide were 4.6 and 2.4% AR at study end (DAT-60) for low and high concentration samples, respectively. The amounts of carbon dioxide formed in sterile samples after 60 days were 0.7% AR and 0.2% AR for low and high concentration samples, respectively. Formation of volatile organic compounds (VOC) was insignificant as demonstrated by values of ≤ 2.4% AR at all sampling intervals for both concentrations in degradation samples as well as in sterile samples.

Extractable residues in the total system (suspension extract and sediment extracts) decreased from DAT-0 to DAT-60 from 95.5 to 60.3% AR for low concentration samples and from 99.7 to 55.9% AR for high concentration samples. In sterile samples (DAT-60), residues in the total system amounted to 97.5% AR for low concentration samples and 116% AR for high concentration samples.

Non-extractable residues (NER) increased from DAT-0 to DAT-60 from 1.1 to 26.4% AR for low concentration samples and from 0.2 to 24.8% AR for high concentration samples. In sterile test system, non-extractable residues (NER) at DAT-60 accounted for 11.4 and 1.9% AR in low and high concentration test systems, respectively.

The amount of aclonifen in the total system decreased from DAT-0 to DAT-60 from 95.5 to 42.9% AR for low concentration samples and from 99.7 to 31.3% AR for high concentration samples. In sterile samples (DAT-60), the amount of aclonifen in the total system was not detectable for low concentration samples and 94.5% AR for high concentration samples.

Degradation of aclonifen was accompanied by the formation of one degradation product, identified as M-02 (called aclonifen-des-phenyl in the report) with a maximum occurrence in the total system of 17.4% AR at DAT-60 in low concentration samples and 14.4% AR at DAT-60 in high concentration samples. The total unidentified residues amounted to a maximum of 24.2% AR in low concentration samples which consisted of diffuse radioactivity only. In high concentration samples total unidentified residues were <LOD. In sterile samples, no degradation products of aclonifen were determined in the total system of both concentrations.

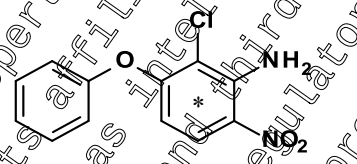
The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. The experimental data could be best described by a single first order (SFO) kinetic model for high concentration samples and a double first order in parallel (DFOP) kinetic model kinetics for low concentration samples. The DT_{50} values for the degradation of aclonifen in the total system were 25.7 and 39.2 days for low and high concentrations, respectively.

For flasks treated with [phenoxy-UL-^{14}C]-aclonifen, HPLC analysis of extractable residues in the total system (suspension extract and sediment extracts) after 49 and 61 days of incubation showed that aclonifen disappeared nearly completely and no other compounds were determined. Thus, it was proven that the degradation behaviour of aclonifen in surface water is completely understood.

I MATERIALS AND METHODS

A. MATERIALS

1. Test material: [aniline-UL-^{14}C]-aclonifen



* Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC) 2-chloro-6-nitro-3-phenoxy-aniline

CA registry number: 74070-46-5

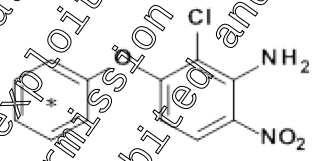
Lot or batch number: KML 10075

Specific activity: 6.59 MBq/mg

Radiochemical purity: > 98%

Stability of test compound: Shown to be stable under the conditions of the test

Additional test material: [phenoxy-UL-^{14}C]-aclonifen



* Denotes position of [^{14}C]-radiolabel

Lot or batch number: KML 10393

Specific activity: 4.52 MBq/mg

Radiochemical purity: >98%

Stability of test compound: Shown to be stable under the conditions of the test

2. Water/sediment:

Surface water and sediment was freshly collected from [redacted], North Rhine-Westphalia, Germany in November 2016. Water and sediment were sampled from approximately 0 to 1 m depth of water. The upper sediment layer was collected 0 to 5 cm Prior to use, sediment was sieved through a 2 mm sieve and the surface water through a 0.063 mm sieve. The water was stored for 1 day prior to the start of the equilibration.

Table 7.2.2.3- 1: Physicochemical Parameters of the Water/Sediment Systems

Parameter [units]		Results
Water Designation		[redacted]
Geographic Location		[redacted] / North Rhine-Westphalia / Germany
GPS Coordinates		[redacted]
Properties of Water		
Water Temperature [°C]		9.5
pH		6.9
Redox Potential E_{obs} [mV]		194
Oxygen Saturation [%]		88.9
Total Organic Carbon (TOC) [mg/L]		< 2.0
Dissolved Organic Carbon (DOC) [mg/L]		< 2.0
Biochemical Oxygen Demand (BOD_5) [mg/L]		N/A [1]
Total Nitrogen [mg/L]		1.2
Total Phosphorus [mg/L]		0.05
Properties of Sediment		
Texture Class (USDA)		Silt loam
Sand [%]	[50 µm – 2 mm]	36
Silt [%]	[2 µm – 50 µm]	51
Clay [%]	[< 2 µm]	13
pH (sediment / 0.01 M $CaCl_2$ 1/2)		5.4
pH		6.9
TOC [g/kg]		69.8
Cation Exchange Capacity [meq/100 g]		9.2
Redox Potential E_H [mV]		-1.01
Microbial biomass [mg microbial CO_2 per h per kg of sediment dw]		67.6

[1] not applicable as TOC was < 5 mg/L

B. STUDY DESIGN

In-life dates: 01 November 2016 – 17 December 2019

Experimental conditions

Parameter	Description
Duration of test	60 days
Water and sediment conditions	Fresh water and sediment samples

Parameter		Description
Concentration in test system	Nominal [µg test item / L]	1 (low concentration samples) and 10 (high concentration samples)
	Actual [µg test item / L]	1.1 (low concentration samples) and 9.8 (high concentration samples)
Control conditions (if used)	Microbial activity samples	Samples for determination of microbial activity: surface water treated with control item (1 µg/test system, corresponding to 10 µg/L)
	Solvent control microbial activity samples	Samples treated with control item (1 µg/test system, corresponding to 10 µg/L) and solvent (10 µL acetone)
	Sterile samples	Sterilized test systems treated with test item (1.1 and 9.8 µg/L)
Number of replications	Degradation samples	Duplicate samples for each sampling interval
	Microbial activity samples	Duplicate samples for each sampling interval
	Solvent control microbial activity samples	Duplicate samples for each sampling interval
	Sterile samples	Duplicate samples for each sampling interval
Test apparatus		250 mL Erlenmeyer flasks with baffles
Sediment sample weight [VSediment and mSediment DW]		0.33 g wet sediment per replicate corresponded to approx. 0.1 g dry weight.
Water sample Volume [V_{Water}]		100 mL per replicate
Test material application	Identity of solvent	acetone/water 1/10 (v/v)
	Volume of application solution	Low conc.: 102 µL of application solution High conc.: 104 µL of application solution
	Application method	Application to the water surface using an adjustable pipette
Traps for CO_2 and organic volatiles		The traps were filled with soda lime and polyurethane foam plug. The traps were permeable for oxygen.
Control item application (microbial activity samples)	Solvent of application solution of control item	Water
	Volume of application solution (control item) used per treatment	48 µL per test system
	Solvent for treatment of solvent control microbial activity samples	10 µL acetone
	Application method	Application to the water surface using an adjustable pipette
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	19.4°C
	Continuous darkness (Yes/No)	Yes

Sampling

Parameter		Details
Sampling intervals for the parent/transformation products		Duplicate samples of each test concentration were processed and analysed 0, 1, 3, 7, 14, 30 and 60 days after treatment (DAT). Sterile samples were processed at study end (DAT-60) for both concentrations
Sampling procedure		See below
Collection of CO_2 and other volatiles		Soda lime for absorption of carbon dioxide and polyurethane foam for adsorption of volatile organic compounds.
Sampling intervals / times for	Redox potential, oxygen saturation and pH	Each sampling interval
	TOC, DOC, total nitrogen, total phosphorus, BOD_5	At start of equilibration for untreated surface water
	Sterility checks	Sterile samples at DAT-60
	Other	N/A
Sample storage before analysis		The samples were processed immediately after sampling; first HPLC/radiodetection analysis of samples was performed within 6 days after sampling. Samples were stored at < -18 °C in the dark up to 32 days for re-analysis.

Description of analytical procedures

The total sample (water and sediment) suspension extracts was transferred into a centrifuge beaker. Then, the test vessel was rinsed with 20 mL acetonitrile and the rinse was combined with the sample. The sample was then extracted for 5 min using a polytron followed by centrifugation and decantation, an aliquot of water was removed, and the concentration of any dissolved carbon dioxide determined. The remaining sediment was extracted twice at ambient temperature using acetonitrile/water 1/1 (v/v) followed by two additional extractions with acetonitrile and methanol. Following extraction, sediment samples were dried and homogenised, and the remaining unextracted radioactivity quantified by combustion.

The amounts of test item and degradation products in the suspension extracts and the sediment extracts were determined by liquid scintillation counting (LSC) and by HPLC/radiodetection analysis following concentration by rotary evaporation. The limit of detection for the primary chromatographic method was determined to be 2.6% AR and 3.5% AR in the high and low concentration samples respectively.

Degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2.

II. RESULTS AND DISCUSSION

A. Mass Balance

Mean material balances were 98.3% AR for low concentration test systems (range from 91.4 to 110% AR) and 93.4% AR for high concentration test systems (range from 81.3 to 105% AR). Mean material balances of sterile samples were 110% AR for low concentration test systems and 119% AR for high concentration test systems.

Material balances in the range of 80 to 120% AR were considered acceptable in the present study which deviates from the targeted 90 to 110% AR specified in the applicable OECD test guideline 309. However, these values are specified as targets and not as a fixed criterium for acceptance of the study. Due to challenges caused by the interplay of low test item concentrations (1 and 10 µg/L), the application of low amounts of sediment (1 g/L) and the need for sample pre-concentration before measurement, the targeted 90 to 110% AR were not achieved at all times. Therefore, the range of material balances considered acceptable was set to 80-120% AR. Material balances of individual samples going below or exceeding the specified range were excluded from the evaluation.

A summary of the recoveries is provided in Table 7.2.2.2- 8 and Table 7.2.2.2- 9.

B. Findings

The residues in suspension extract decreased from DAT-0 to DAT-60 from 82.4 to 40.2% AR in low concentration test systems and from 88.4 to 44.6% AR in high concentration test systems. In sterile samples (DAT-60), residues in suspension extract amounted to 76.1% AR in low concentration test systems and 94.5% AR in high concentration test systems.

The amount of aclonifen in suspension extract decreased from DAT-0 to DAT-60 from 82.4 to 22.8% AR in low concentration test systems and from 88.4 to 20.0% AR in high concentration test systems. In sterile samples (DAT-60), the amount of aclonifen in suspension extract was not detectable in low concentration test systems and 94.5% AR in high concentration test systems.

Degradation of aclonifen was accompanied by the formation of one degradation product, identified as M-02 with a maximum occurrence in suspension extract of 17.4% AR at DAT-60 in low concentration samples and 14.4% AR at DAT-60 in high concentration samples. The total unidentified residues amounted to a maximum of 9.1% AR and no single component exceeded 3.0% AR at any sampling interval for both concentrations. In sterile samples, aclonifen-des-phenyl was not determined for both concentrations.

Extractable residues in the sediment of low concentration samples increased from 13.1% AR at DAT-0 to 24.2% AR at DAT-7 and decreased then to 20.1% AR at DAT-60. Extractable residues in the sediment of high concentration samples increased from 11.3% AR at DAT-0 to 17.0% AR at DAT-14 and decreased then to 11.3% AR at DAT-60. In sterile samples (DAT-60), residues in the sediment amounted to 21.3% AR for low concentration samples and 21.6% AR for high concentration samples.

The amount of aclonifen in the sediment of low concentration samples increased from 13.1% AR at DAT-0 to 20.1% AR at DAT-60 and was not-detectable from DAT-3 to DAT-14. The amount of aclonifen for high concentration samples increased from 11.3% AR at DAT-0 to 17.0% AR at DAT-14 and decreased then to 11.3% AR at DAT-60. In sterile samples (DAT-60), the amount of aclonifen in the sediment extracts was not detectable for both concentrations. No degradation products of aclonifen were identified in sediment extracts. The total unidentified residues amounted to a maximum of 24.2% AR in low concentration samples, which consisted of diffuse radioactivity only. In high concentration samples total unidentified residues were <LOD. In sterile samples, no degradation products of aclonifen were determined in the sediment of both concentrations

The amounts of carbon dioxide were 4.6 and 2.4% AR at study end (DAT-60) for low and high concentration samples, respectively. The amounts of carbon dioxide formed in sterile samples after 60 days were 0.7% AR and 0.2% AR for low and high concentration samples, respectively. Formation of volatile organic compounds (VOC) was insignificant as demonstrated by values of ≤ 2.4% AR at all sampling intervals for both concentrations in degradation samples as well as in sterile samples.

Extractable residues in the total system (suspension extract and sediment extracts) decreased from DAT-0 to DAT-60 from 95.5 to 60.3% AR for low concentration samples and from 99.7 to 55.9% AR for high concentration samples. In sterile samples (DAT-60), residues in the total system amounted to 97.5% AR for low concentration samples and 116% AR for high concentration samples.

The amount of aclonifen in the total system decreased from DAT-0 to DAT-60 from 95.5 to 42.9% AR for low concentration samples and from 99.7 to 31.3% AR for high concentration samples.

In sterile samples (DAT-60), the amount of aclonifen in the total system was not detectable for low concentration samples and 94.5% AR for high concentration samples.

Degradation of aclonifen was accompanied by the formation of one degradation product, identified as M-02 with a maximum occurrence in the total system of 17.4% AR at DAT-60 in low concentration samples and 14.4% AR at DAT-60 in high concentration samples. The total unidentified residues amounted to a maximum of 24.2% AR in low concentration samples which consisted of diffuse radioactivity only. In high concentration samples total unidentified residues were <LOD. In sterile samples, no degradation products of aclonifen were determined in the total system of both concentrations.

Non-extractable residues (NER) increased from DAT-0 to DAT-60 from 1.1 to 26.4% AR for low concentration samples and from 0.2 to 24.8% AR for high concentration samples. In sterile test system, non-extractable residues (NER) at DAT-60 accounted for 11.4 and 1.9% AR in low and high concentration test systems, respectively.

A summary of the degradation of aclonifen in the systems is provided in Table 7.2.2.2- 10 and Table 7.2.2.2- 11.

To fully understand the degradation behaviour of aclonifen in surface water under aerobic conditions, duplicate test systems were treated with a second radiolabel, i.e. [phenoxy-UL-^{14}C]aclonifen, at a final concentration of 11 µg/mL. Test systems were incubated, processed and analyzed after 49 and 61 days as described for [aniline-UL-^{14}C]aclonifen. HPLC/radiodetection analyses of water and sediment extracts after 49 and 61 days of incubation showed that aclonifen disappeared nearly completely and no other compounds were determined. Thus, it was proven that the degradation behaviour of aclonifen in surface water is completely understood.

Table 7.2.2.2- 8: Material balance of radioactivity in surface water under aerobic conditions at low concentration expressed as percentage of applied radioactivity

	Repl. No.	DAT							
		0	1[1]	3[1]	7	14	30	60	60 sterile
Volatiles									
Carbon Dioxide	A	n.a.	1.1	0.8	0.6	~~1.0~~	~~2.1~~	4.0	0.8
	B	n.a.	~~0.5~~	0.3	~~0.4~~	0.9	~~8.4~~	~~5.3~~	0.6
	Mean	**n.a.**	**1.1**	**0.6**	**0.5**	**0.9**	**~~5.3~~**	**4.6**	**0.7**
Volatile Organic Compounds	A	n.a.	0.9	0.7	1.7	~~0.3~~	~~1.5~~	0.7	0.5
	B	n.a.	~~0.8~~	0.7	~~3.2~~	0.7	~~1.0~~	0.7	0.6
	Mean	**n.a.**	**0.9**	**0.7**	**2.4**	**0.3**	**~~1.3~~**	**0.7**	**0.6**
Total Volatiles	A	n.a.	2.1	1.5	2.2	~~1.3~~	~~3.6~~	4.7	1.3
	B	n.a.	~~1.4~~	1.1	~~3.6~~	1.1	~~9.4~~	~~6.0~~	1.2
	Mean	**n.a.**	**2.1**	**1.3**	**2.9**	**1.1**	**~~6.5~~**	**5.3**	**1.3**
Water and Sediment Extractable Residues									
Water / Sediment Suspension Extract	A	84.1	85.0	72.0	77.3	~~78.4~~	~~83.6~~	41.2	74.2
	B	80.7	~~87.1~~	74.8	77.9	63.7	~~77.3~~	39.2	78.1
	Mean	**82.4**	**85.0**	**73.4**	**77.6**	**63.7**	**~~80.5~~**	**40.2**	**76.1**
Sediment Extractable Residues									
Ambient Extract 1	A	11.5	13.3	16.9	14.8	~~27.3~~	~~11.7~~	15.2	19.4
	B	14.6	~~37.5~~	16.9	17.1	16.0	~~11.2~~	13.2	11.3
	Mean	**13.1**	**13.3**	**16.9**	**15.9**	**16.0**	**~~11.4~~**	**14.2**	**15.4**
Ambient Extract 2	A	n.a.	n.a.	8.9	8.1	~~4.8~~	~~5.1~~	6.2	6.1
	B	n.a.	~~n.a.~~	10.7	8.5	5.1	~~4.7~~	5.6	5.8
	Mean	**n.a.**	**n.a.**	**9.8**	**8.3**	**5.1**	**~~4.9~~**	**5.9**	**5.9**
Total Sediment Extractable Residues	A	11.5	13.3	25.8	22.8	~~32.0~~	~~16.8~~	21.4	25.5
	B	14.6	~~37.1~~	33.5	25.6	21.2	~~15.8~~	18.8	17.1
	Mean	**13.1**	**13.3**	**14.7**	**24.2**	**21.2**	**~~16.3~~**	**20.1**	**21.3**
Total Extractable Residues	A	95.6	98.3	97.8	100.2	~~110.4~~	~~100.3~~	62.6	99.7
	B	95.3	~~124.2~~	78.4	103.4	84.8	~~93.2~~	58.0	95.2
	Mean	**95.5**	**98.3**	**88.1**	**101.8**	**84.8**	**~~96.8~~**	**60.3**	**97.5**
Non-Extractable Residues	A	0.8	1.9	1.6	5.2	~~11.5~~	~~19.1~~	25.0	19.2
	B	1.3	~~2.6~~	2.5	5.3	11.7	~~21.0~~	27.9	3.6
	Mean	**1.1**	**1.9**	**2.1**	**5.2**	**11.7**	**~~20.0~~**	**26.4**	**11.4**
Material Balance	A	96.4	102.3	100.9	107.6	~~123.3~~	~~123.0~~	92.2	120.3
	B	96.6	~~128.2~~	82.0	112.4	97.7	~~123.6~~	91.9	99.9
	Mean	**96.5**	**102.3**	**91.4**	**110.0**	**97.7**	**~~123.3~~**	**92.1**	**110.1**
Mean 98.3 ± 10.7 (excludes sterile samples)									

n.a.: not analyzed, DAT: days after treatment

Samples with material balances <80% and >120% were excluded from the evaluation.

[1] For replicates B of day 3 and day 7, the radioactivity assigned to a contamination from a parallel study was subtracted from the total sediment extractable residues, total extractable residues and material balance. Thus, for these samples the sum of both individual extracts does not match the total sediment extractable residues.

Material balances in the range of 80 to 120% AR were considered acceptable. Due to challenges caused by the interplay of low test item concentrations, the application of low amounts of sediment and the need for sample pre-concentration, the targeted 90 to 110% AR were not achieved at all times.

Table 7.2.2.2- 9: Material balance of radioactivity in surface water under aerobic conditions at high concentration expressed as percentage of applied radioactivity

	Repl. No.	DAT							
		0	1[1]	3	7[1]	14	30	60	60 sterile
Volatiles									
Carbon Dioxide	A	n.a.	0.2	3.7	0.3	0.5	1.1	1.7	1.1
	B	n.a.	0.2	4.3	0.3	0.5	1.4	3.1	0.2
	Mean	**n.a.**	**0.2**	**4.0**	**0.3**	**0.5**	**1.3**	**2.4**	**0.2**
Volatile Organic Compounds	A	n.a.	0.2	0.1	0.1	0.1	0.1	0.1	1.1
	B	n.a.	0.1	< 0.1	0.1	0.1	0.2	0.1	0.9
	Mean	**n.a.**	**0.1**	**< 0.1**	**0.1**	**0.1**	**0.2**	**0.1**	**0.9**
Total Volatiles	A	n.a.	0.3	3.7	0.4	0.6	1.2	1.8	2.1
	B	n.a.	0.2	4.3	0.4	0.6	1.7	3.2	1.0
	Mean	**n.a.**	**0.3**	**4.0**	**0.4**	**0.6**	**1.4**	**2.5**	**1.0**
Water and Sediment Extractable Residues									
Water / Sediment Suspension Extract	A	87.3	65.1	66.8	85.9	67.4	86.9	47.2	822.6
	B	89.5	68.0	69.1	81.0	65.4	59.6	41.9	94.5
	Mean	**88.4**	**66.5**	**67.9**	**83.5**	**66.4**	**73.3**	**44.6**	**94.5**
Sediment Extractable Residues									
Ambient Extract 1	A	11.2	22.5	14.2	17.9	16.4	11.0	9.5	119.7
	B	11.5	16.4	14.3	16.0	15.8	13.3	11.0	20.5
	Mean	**11.3**	**19.4**	**14.3**	**16.9**	**16.1**	**12.2**	**10.2**	**20.5**
Ambient Extract 2	A	n.a.	n.a.	1.3	1.3	1.1	0.9	1.0	3.1
	B	n.a.	n.a.	1.3	1.6	0.7	1.2	1.1	1.1
	Mean	**n.a.**	**n.a.**	**1.3**	**1.4**	**0.9**	**1.0**	**1.1**	**1.1**
Total Sediment Extractable Residues	A	11.2	15.2	15.5	11.6	17.5	11.9	10.5	122.7
	B	11.5	11.9	15.6	17.6	16.6	14.5	12.1	21.6
	Mean	**11.3**	**13.6**	**15.6**	**14.6**	**17.0**	**13.2**	**11.3**	**21.6**
Total Extractable Residues	A	98.5	80.3	82.2	97.5	84.9	98.8	57.7	945.4
	B	100.9	79.9	84.7	98.6	82.0	74.1	54.0	116.1
	Mean	**99.7**	**80.1**	**83.5**	**98.0**	**83.5**	**86.5**	**55.9**	**116.1**
Non-Extractable Residues	A	0.2	0.9	1.2	2.6	8.9	12.9	22.6	8.6
	B	0.1	0.9	1.3	5.2	9.1	21.7	27.1	1.9
	Mean	**0.2**	**0.9**	**1.3**	**3.9**	**9.0**	**17.3**	**24.8**	1.9
Material Balance	A	98.7	81.6	87.2	100.6	94.3	112.9	82.1	956.0
	B	101.1	81.0	90.3	104.2	91.7	97.5	84.3	119.0
	Mean	**99.9**	**81.3**	**88.8**	**102.4**	**93.0**	**105.2**	**83.2**	**119.0**
Mean 93.4 ± 9.4 (excludes sterile samples)									

n.d.: not detected, n.a.: not analyzed, DAT: days after treatment

[1] For both replicates of day 1 and replicate A of day 7, the radioactivity assigned to the artefact was subtracted from the total sediment extractable residues, total extractable residues and material balance. Thus, for these samples the sum of both individual extracts does not match the total sediment extractable residues.

Material balances in the range of 80 to 120% AR were considered acceptable. Due to challenges caused by the interplay of low test item concentrations, the application of low amounts of sediment and the need for sample pre-concentration, the targeted 90 to 110% AR were not achieved at all times.

Table 7.2.2.2- 10: Degradation of aclonifen in surface water under aerobic conditions at low concentration expressed as percentage of applied radioactivity

Compound	Source	Repl No.	DAE							
			0	1	3	7	14	30	60	60 sterile
Aclonifen	Water/ Sediment Suspension	Mean	82.4	85.0	73.4	77.6	53.4	~~61.0~~	22.8	n.d.
		SD	± 1.7		± 1.4	± 0.3		~~± 6.4~~	± 0.4	
	Sediment	Mean	13.1	13.3	n.d.	n.d.	n.d.	~~16.3~~	20.1	n.d.
		SD	± 1.6					~~± 0.5~~	± 1.3	
	Entire System	Mean	95.5	98.3	73.4	77.6	53.4	~~77.3~~	42.9	n.d.
		SD	± 0.2		± 1.4	± 0.3		~~± 6.8~~	± 0.9	
M-02	Water/ Sediment Suspension	Mean	n.d.	n.d.	n.d.	n.d.	10.2	~~19.5~~	17.4	n.d.
		SD						~~± 3.2~~	± 1.4	
	Sediment	Mean	n.d.	n.d.	< LOD	n.d.	n.d.	~~n.d.~~	n.d.	n.d.
		SD								
	Entire System	Mean [4]	n.d.	n.d.	< LOD	n.d.	10.2	~~19.5~~	17.4	n.d.
		SD						~~± 3.2~~	± 1.4	
Sum of Unid./Diff. Residues [1]	Water/ Sediment Suspension	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	~~n.d.~~	n.d.	76.1
		SD								± 2.0
	Sediment	Mean	n.d.	n.d.	12.9	24.2	21.2	~~n.d.~~	n.d.	21.3
		SD			± 0.0	± 1.4				± 4.2
	Entire System	Mean [4]	n.d.	n.d.	12.9	24.2	21.2	~~n.d.~~	n.d.	97.5
		SD			± 0.0	± 1.4				± 2.3
Total Extractable Residues [2]	Water/ Sediment Suspension	Mean	82.4	85.0	73.4	77.6	63.7	~~80.5~~	40.2	76.1
		SD	± 1.7		± 1.4	± 0.3		~~± 3.1~~	± 1.0	± 2.0
	Sediment	Mean	13.1	13.3	14.7	24.2	21.2	~~16.3~~	20.1	21.3
		SD	± 1.6		± 1.1	± 1.4		~~± 0.5~~	± 1.3	± 4.2
	Entire System	Mean [4]	95.5	98.3	88.1	101.8	84.8	~~96.8~~	60.3	97.5
		SD	± 0.2		± 9.7	± 1.6		~~± 3.6~~	± 2.3	± 2.3
Carbon Dioxide [3]		Mean	n.a.	1.1	0.6	0.5	0.9	~~5.3~~	4.6	0.7
		SD			± 0.2	± 0.1		~~± 3.2~~	± 0.6	± 0.1
Volatile Organic Compounds [3]		Mean	n.a.	0.9	0.7	2.4	0.3	~~1.3~~	0.7	0.6
		SD			± 0.0	± 0.8		~~± 0.2~~	± 0.0	± 0.1
Non-Extractable Residues [3]		Mean	1.1	1.9	2.1	5.2	11.7	~~20.0~~	26.4	11.4
		SD	± 0.2		± 0.5	± 0.1		~~± 0.9~~	± 1.5	± 7.8
Total Recovery [2]		Mean	96.5	102.3	91.4	110.0	97.7	~~123.3~~	92.1	110.1
		SD	± 0.1		± 9.5	± 2.4		~~± 0.3~~	± 0.2	± 10.2

n.d.: not detected, DAT: days after treatment, SD: standard deviation

Samples with material balances <80% and >120% were excluded from the evaluation.

[1] Minor components are summed up to sum of unidentified / diffuse residues

[2] Difference to Material Balance values due to rounding errors as well as clean up and chromatographic losses.

[3] Values taken from Material Balance.

[4] Mean values of the entire system could be unequal compared to the sum of the mean values of water and sediment, because mean values of the entire system were calculated from the single replicate values of the entire system and not by summation of the mean values of water and sediment.

Table 7.2.2.2- 11: Degradation of aclonifen in surface water under aerobic conditions at high concentration expressed as percentage of applied radioactivity

Compound	Source	Repl No.	DAE							
			0	1	3	7	14	30	60	60 sterile
Aclonifen	Water/ Sediment Suspension	Mean	88.4	66.5	67.9	83.5	61.7	55.8	20.0	94.5
		SD	± 1.1	± 1.4	± 1.2	± 2.4	± 0.3	± 9.9	± 4.5	
	Sediment	Mean	11.3	12.0	15.6	14.6	17.0	13.2	11.3	n.d.
		SD	± 0.1	± 3.3	± 0.1	± 3.0	± 0.5	± 1.3	± 0.8	
	Entire System	Mean	99.7	78.5	83.5	98.0	78.8	69.0	31.3	94.5
		SD	± 1.2	± 1.8	± 1.2	± 0.5	± 0.8	± 8.6	± 3.7	
M-02	Water/ Sediment Suspension	Mean	n.d.	n.d.	n.d.	n.d.	4.7	13.5	14.4	n.d.
		SD					± 0.6	± 3.0	± 0.2	
	Sediment	Mean	n.d.	< LOD	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
		SD								
	Entire System	Mean [4]	n.d.	< LOD	n.d.	n.d.	4.7	13.5	14.4	n.d.
		SD					± 0.6	± 3.0	± 0.2	
Sum of Unid./Diff. Residues [1]	Water/ Sediment Suspension	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	3.9	9.1	n.d.
		SD						± 0.7	± 3.1	
	Sediment	Mean	n.d.	< LOD	n.d.	< LOD	n.d.	n.d.	n.d.	72.2
		SD								
	Entire System	Mean [4]	n.d.	< LOD	n.d.	< LOD	n.d.	3.9	9.1	72.2
		SD						± 0.7	± 3.1	
Total Extractable Residues [2]	Water/ Sediment Suspension	Mean	88.4	66.5	67.9	83.5	66.4	73.3	43.5	94.5
		SD	± 1.1	± 1.4	± 1.2	± 2.4	± 1.0	± 13.6	± 1.6	
	Sediment	Mean	11.3	12.0	15.6	14.6	17.0	13.2	11.3	72.2
		SD	± 0.1	± 3.3	± 0.1	± 3.0	± 0.5	± 1.3	± 0.8	
	Entire System	Mean [4]	99.7	78.5	83.5	98.0	83.5	86.5	54.8	116.1
		SD	± 1.2	± 1.8	± 1.2	± 0.5	± 1.4	± 12.3	± 0.8	
Carbon Dioxide [3]		Mean	n.a.	0.2	4.0	0.3	0.5	1.3	2.4	0.2
		SD		± 0.0	± 0.3	± 0.0	± 0.0	± 0.2	± 0.7	
Volatile Organic Compounds [3]		Mean	n.a.	0.1	< 0.1	0.1	0.1	0.2	0.1	0.9
		SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	
Non-Extractable Residues [3]		Mean	0.2	0.9	1.3	3.9	9.0	17.3	24.8	1.9
		SD	± 0.0	± 0.0	± 0.0	± 1.3	± 0.1	± 4.4	± 2.3	
Total Recovery [2]		Mean	99.9	79.7	88.7	102.4	93.0	105.2	82.1	119.0
		SD	± 1.2	± 1.9	± 1.6	± 1.8	± 1.3	± 7.7	± 2.1	

n.d.: not detected, DAT: days after treatment, SD: standard deviation

Samples with material balances <80% and >120% were excluded from the evaluation.

[1] Minor components are summed up to sum of unidentified / diffuse residues

[2] Difference to Material Balance values due to rounding errors as well as clean up and chromatographic losses.

[3] Values taken from Material Balance.

[4] Mean values of the entire system could be unequal compared to the sum of the mean values of water and sediment, because mean values of the entire system were calculated from the single replicate values of the entire system and not by summation of the mean values of water and sediment.

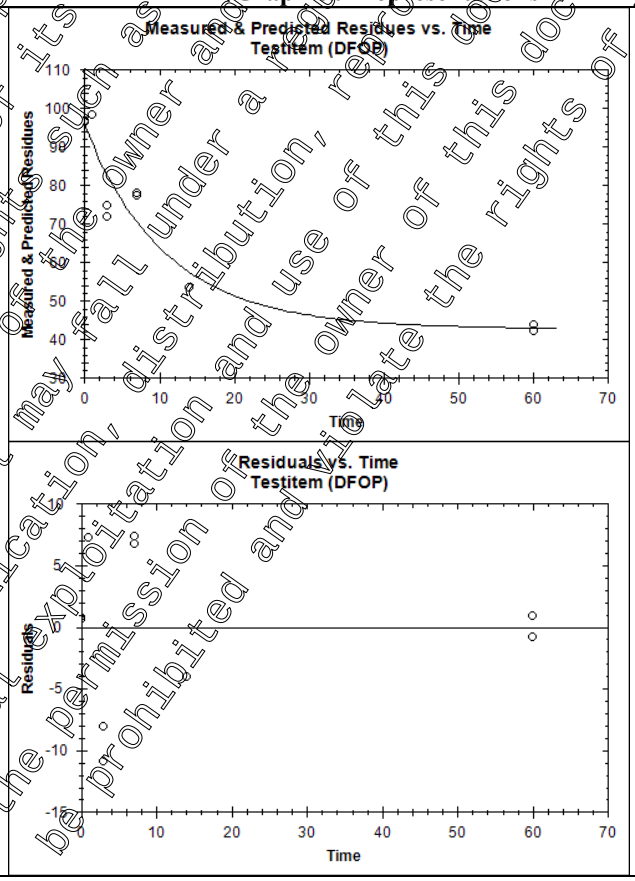
Degradation kinetics

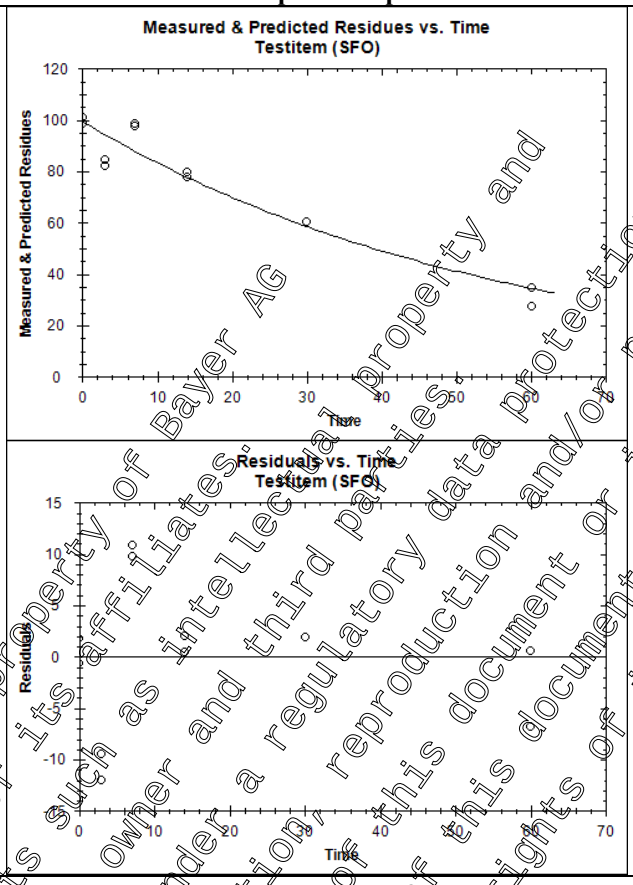
The reported DT_{50} values are summarised in Table 7.2.2.2- 6. The degradation of aclonifen followed single first order (SFO) kinetics for high concentration samples and double first order in parallel (DFOP) kinetics for low concentration samples, according to the lowest chi² error values and visual assessments.

Table 7.2.2.2- 12: Summary of the kinetic evaluation of the degradation of aclonifen in water systems under aerobic conditions

Test system	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	DT_{50} [days]	DT_{90} [days]
Low	DFOP	95.8	k1: 0.093872 k2: 0.00328 g: 0.545123	8.02	0.272 0.495	25.7	>1000
High	SFO	99.3	k: 0.017674	6.63	1.77e-05	39.2	130

Table 7.2.2.2- 13: Degradation of aclonifen in water systems under aerobic conditions with time

Trial / Best Fit Model	Graphical Representations
Low concentration / SFO	

Trial / Best Fit Model	Graphical Representations
High concentration / SFO	

III. CONCLUSION

In the total system (surface water), aclonifen was degraded well with calculated best fit DT_{50} values o of 25.7 and 39 days for low and high aclonifen concentrations, respectively.

Formation of carbon dioxide was up to 4.6% AR at study end indicating the potential for a complete mineralization of aclonifen and its degradation products.

Formation of non-extractable residues (NER) was up to 26.4% AR at study end, which is an indication for biotic degradation of aclonifen. In sterile samples NER accounted to a maximum of 11.4% AR after 60 days, indicating that formation of NER is enhanced by microbial activity and thus considered as transformation and detoxification of aclonifen.

Degradation of aclonifen was accompanied by the formation of one degradation product, identified as M-02 with maximum occurrence in suspension extract of 17.4% AR. In sterile samples M-02 was not determined for either concentrations.

Aclonifen will be rapidly degraded in surface water under aerobic conditions. Formation of carbon dioxide indicates the potential for a complete mineralization of aclonifen.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD Test Guideline No. 309 in a 'suspended sediment' test system representative of most surface waters according to OECD Test Guideline 309. Aclonifen was degraded rapidly in both test concentrations under the test conditions.

However the applicant considers that as aclonifen is very strongly adsorbed (K_{oc} > 5000) and any compound reaching aquatic bodies such as ditches, ponds or streams bordering fields in which

aclonifen is applied, will be degraded in the water phase and also partitioned from the water phase into sediment, where it will continue to be degraded albeit at a slower rate. Consequently, any aclonifen will be very quickly removed from the water column. Thus the 'suspended sediment' study is not considered the most appropriate test to assess the persistence of aclonifen, but rather the aerobic water sediment studies are more suitable to assess the behaviour of aclonifen in the aquatic environment.

Assessment and conclusion by RMS:

CA 7.2.2.3 Water/sediment study

The degradation and fate of aclonifen in aerobic water sediment systems has been investigated in two reliable studies in a total of four different aquatic systems.

Study KCA 7.2.2.3/01 was evaluated during the previous EU review and is still considered as reliable to assess the behaviour of aclonifen in water sediment systems. A statement about the levels of an unidentified minor metabolite from the water sediment study (called Metabolite E in the study) is provided in document KCA 7.2.2.3/05.

Studies KCA 7.2.2.3/06, a water sediment study with [phenoxy-UL-^{14}C]-aclonifen, and KCA 7.2.2.3/07, a study to assess the potential for remobilization of aclonifen once adsorbed to sediment, are provided as new data not yet reviewed under uniform principles.

Document KCA 7.2.2.3/03 is a position paper now superseded by KCA 7.2.2.3/06, which for procedural reasons has to be included in the current dossier.

Finally three kinetic evaluation reports (KCA 7.2.2.3/02, KCA 7.2.2.3/04 and KCA 7.2.2.3/08) are listed. For procedural reasons the two previously submitted kinetic evaluation reports also have to be included under Point KCA 7.2.2.3 in the current dossier (KCA 7.2.2.3/02 and KCA 7.2.2.3/04) but these reports are fully superseded by the latest kinetic evaluation report (KCA 7.2.2.3/08).

Report reference	Author, Year	Aniline Label	Phenoxy Label	Comment
KCA 7.2.2.3/01 M-199647-01-1	[redacted] P. [redacted] G., 2000	✓	×	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.2.2.3/02 M-198402-01-1	[redacted] 2000			Submitted for first approval of aclonifen, 2006. Reviewed under UP. Superseded by M-675507-01-1.
KCA 7.2.2.3/03 M-234959-01-1	[redacted] 2007	-	-	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Superseded by M-674479-01-1.
KCA 7.2.2.3/04 M-300702-01-1	[redacted] & [redacted] 2008	-	-	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Superseded by M-675507-01-1.
KCA 7.2.2.3/05 M-675436-01-1	[redacted] & [redacted] 2019	-	-	Statement addressing data from KCA 7.2.2.3/01. Not yet reviewed under UP.
KCA 7.2.2.3/06 M-674479-01-1	[redacted] P. & [redacted] D., 2019	×	✓	New data not yet reviewed under UP.
KCA 7.2.2.3/07 M-674034-01-1	[redacted] P. & [redacted] D., 2019	×	✓	New data not yet reviewed under UP.

KCA 7.2.2.3/08 M-675507-01-1	[redacted] & [redacted], 2019	-	-	New data not yet reviewed under UP.

In water/sediment systems aclonifen was readily degraded with total system DT_{50} values ranging from 5 to 44 days. The compound dissipated rapidly from the water phase with DT_{50} values of between <1 to 3 days. Once deposited in the sediment, parent continued to degrade over time with DT_{50} values of between 8 to 69 days. Aclonifen reached a maximum of 61.0% of applied radioactivity (AR) in the sediment at day 3 before declining to 4.1% at 100 days. The formation of unextractable bound residues in the sediment was found to be a major elimination process, with degradation of aclonifen to a number of minor metabolites also observed in water and sediment. Aclonifen was degraded by hydroxylation to form M-01 and hydrolysis to form M-02. Under reduced conditions the formation of M-04 was observed occasionally, possibly as a result of the reduction of M-02 as the reduced forms of aclonifen and M-01 were not observed. No major metabolites were observed in any of the water or sediment phases. The minor transformation products in water and sediment were all < 5.0% AR.

A new kinetic evaluation of the experimental data generated in aerobic water sediment studies, KCA 7.2.2.3/01 ([redacted] & [redacted], 2000) and KCA 7.2.2.3/06 ([redacted] & [redacted], 2019) has been conducted according to FOCUS kinetics guidance with the aim of deriving DT_{50} values for use as modelling and trigger endpoints (KCA 7.2.2.3/08, [redacted] & [redacted], 2020). Geometric mean modelling endpoint total system DT_{50} values for aclonifen are summarised in the table below.

Water sediment system	DT50 (days)		
	Total system	Water Phase	Sediment Phase
[redacted]	43.81	1.71	69.49
[redacted]	40.06	3.39	56.67
Anglersee	5.04	1.68	8.43
[redacted]	4.80	0.83	14.00
Geometric mean	**14.4**	**1.7**	**26.1**

Data Point:	KCA 7.2.2.3/01
Report Author:	[redacted]; [redacted]
Report Year:	2000
Report Title:	(14C)-Aclonifen degradation in two water/sediment systems
Report No:	C009991
Document No:	M-199647-01-1
Guideline(s) followed in study:	EU (=EEC): 95/36/EC
Deviations from current test guideline:	Current Guideline: EU (=EEC): 95/36/EC None
Previous evaluation:	Yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of [aniline-UL-^{14}C]-aclonifen were studied in two water/sediment systems, [redacted], [redacted] Farm, [redacted], Essex, UK (water pH 6.7, total organic carbon 7.8 mg/L, sediment texture sandy loam, pH 6.8) and [redacted], [redacted] Farm, [redacted], Essex, UK (water pH 7.5, total organic carbon 4.6 mg/L, sediment texture clay loam, pH 8.4) in the dark at 20°C for 180 days.

A nominal study application rate of 850 µg of [^{14}C]-aclonifen was based upon a field application rate of 3 kg/ha.

The test was performed in systems consisting of cylindrical metabolism flasks filled with a layer of sediment and overlying water at a ratio of 1/5.5 and 1/6.2 (w/w, based upon the dry weight of the sediment) for the [redacted] and [redacted] systems respectively.

Flasks from each system were analysed at time zero, 4 hours, 10 hours, 1, 2, 7, 14, 30, 61, 100 and 180 days. The aqueous phase was decanted off and immediately partitioned with dichloromethane for early time-point samples (0 hour, 4 hours, 10 hours and 1 day) to ensure a homogeneous solution. For later samples the water phases were measured directly. The sediments were extracted with acetonitrile, followed by acetonitrile/water (4/1, v/v) at ambient temperature. At later timepoints (2 days onwards) the sediments were further extracted by soxhlet extraction with acetonitrile/water (4/1, v/v). The radioactivity in the aqueous phase (and dichloromethane partition of early aqueous phases) and in each sediment extract was quantified by LSC. The individual components were identified by HPLC analysis in comparison to certified reference standards. The radioactivity remaining in the sediment was determined by combustion and LSC of the trapped combustion gases.

The overall mean recoveries of radioactivity were 97% and 94% for the [redacted] and [redacted] systems, respectively. Good recoveries of radioactivity (range 90 to 101.3%) were achieved throughout the study.

In both water / sediment systems there was an initial rapid transfer of radioactivity from the water phase to the underlying sediment, followed by a continuing steady transfer. In the [redacted] system, the level of radiolabelled material recovered in the aqueous phase decreased from 96% of applied radioactivity at time zero to 12% after 30 days and 2% after 180 days. In the [redacted] system, the level of radiolabelled material recovered in the aqueous phase decreased from 98% of applied radioactivity at time zero to 13% after 30 days and to 5% after 180 days.

Analysis of the sediment extracts indicated that there was initially a rapid transfer of aclonifen from the water to the sediment phase, reaching 53% for the [redacted] system at 7 days and 46% for the [redacted] system at 2 days. These levels steadily declined until aclonifen only accounted for 3 or 7% of applied radioactivity at the end of the study. In the [redacted] sediment extracts aclonifen was accompanied by a total of six minor metabolites, four of which had been seen in the water phase, including M-01 (called RPA 407074 in the report) and M-02 (called RPA 508285 in the report). In the [redacted] sediment extracts four minor metabolites were detected at various times, three of which corresponded to metabolites seen in the water phase, with M-01 again identified.

Of the nine metabolites observed in the [redacted] system, only two were detected at levels in excess of 5%. An unidentified metabolite D was detected at 7% in the water phase at Day 30 but was not observed in excess of 1% at any other timepoint. M-01 was observed in water and sediment throughout the incubation period but did not exceed 5% in either the water or sediment phases throughout the study and only exceeded 5% in the total system at one timepoint (Day 61). Of the eight metabolites observed in the [redacted] system, only three unidentified metabolites were originally reported at levels in excess of 5% (and after re-evaluation of the 180 day sediment extracts only two metabolites were > 5%). Metabolite B was observed in the water phase throughout the incubation period and occasionally in the sediment but did not exceed 5% in either the water or sediment phases throughout the study and only exceeded 5% in the total system at one timepoint (Day 100). Metabolite C was detected at 5% in the water phase at Day 7 before declining to ca. 3% by the end of the

incubation period. Metabolite E following re-evaluation of the sediment extracts from the final timepoint did not exceed 5% at any timepoint.

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014), resulted in best-fit DT_{50} values of 22.8, 1.5 and 69.5 days for the [redacted] total system, water and sediment compartments and DT_{50} values of 10.2, 3.4 and 56.7 days for the [redacted] total system, water and sediment compartments.

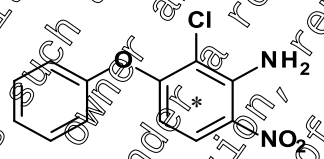
Water/Sediment System		Best Fit Kinetic Model	DT_{50} [days]	DT_{90} [days]	Chi^2 Error [%]
[redacted]	Total system	DFOP	22.81	179.5	5.7
	Water	HS	1.48	19.37	9.1
	Sediment	SFO	69.49	230.8	7.6
[redacted]	Total system	HS	10.19	133.0	5.8
	Water	SFO	3.39	11.3	15.3
	Sediment	SFO	56.67	188.3	9.4

DFOP: double first order in parallel, HS: hockey stick, SFO: simple first order

I. MATERIALS AND METHODS

A. MATERIALS

1. Test material: [U-^{14}C-aniline]-aclonifen



* Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC): 2-chloro-6-nitro-3-phenoxy-aniline
CA registry number: 74070-46-5
Lot or batch number: BTS 968
Specific activity: 3.32 MBq/mg (881.3 MBq/mmol)
Radiochemical purity: 99.3%
Stability of test compound: Shown to be stable under the conditions of the test

2. Water/Sediment: The water/sediment systems were freshly collected from [redacted] Farm, [redacted], Essex, UK and [redacted], [redacted] Farm, [redacted], Essex. The sediments were sieved through a 2 mm screen and the water was filtered through a 0.2 mm filter. The microbial biomass of both sediments was measured at the start and on completion of the study. The main characteristics of the two water / sediment systems are shown below.

Table 7.2.2.3- 2: Characteristics of sediments and associated water

Parameter	Units	[redacted]	[redacted]
Source location		[redacted] Farm, [redacted], Essex, UK	[redacted] Farm, [redacted], Essex, UK
Sediment			
Textural classification USDA		Sandy loam	Clay loam
Sand (50 µm – 2000 µm)	%	48.98	25.05
Silt (2 µm – 50 µm)	%	40.48	45.96
Clay (< 2 µm)	%	10.54	28.99
Organic Carbon	%	5.7	3.8
pH	in water	6.8	8.4
	in 1M KCl	5.9	7.7
	in 0.01M $CaCl_2$	6.2	7.7
Biomass Initial	µg C / g soil	244	71
Final		238	104
Cation Exchange Capacity	meq/100g	11.0	75.9
Total nitrogen	mg/kg	3248	2911
Total phosphorous	mg/kg	665	522
Water			
pH (at time of collection)		6.7	7.5
Oxygen content (at time of collection)	%	36	51
Redox potential (at time of collection)	mV	380	614
Total nitrogen	ppm	100	3.2
Total phosphorous	ppm	1.8	0.0
Total organic carbon	ppm	7.8	4.6
Water hardness	ppm as $CaCO_3$	311	301

B. STUDY DESIGN AND METHODS

1. In-life dates:

7 December 1999 – 4 September 2000

2. Experimental design

Cylindrical metabolism flasks of height 22 cm and 6 cm inner diameter were filled with a layer of sediment and overlying water at a ratio of 1/5.5 and 1/6.2 (w/w, based upon the dry weight of the sediment) for the [redacted] and [redacted] systems, respectively. The flasks were incubated in the dark at 20 ± 2°C, with a continuous supply of humidified air being drawn through the surface of the water phase so as not to disturb the sediment layer, and were allowed to acclimatise until phase separation was complete and parameters such as dissolved oxygen, pH and redox potential had stabilised. The air flows exiting the flasks were passed through a series of traps containing ethylene glycol (x1) and 2 M KOH (x2) to trap volatiles and CO_2.

The flasks were dosed by the application of 250 µL of an acetonitrile stock solution to the water surface. This contained 860 µg (target 850 µg) of [^{14}C]-aclonifen. The application rate was based upon a field application rate of 3 kg/ha and assuming direct overspray. No correction for the depth of the water body was considered. This resulted in an application equivalent to an initial concentration of approximately 2 mg/L to the [redacted] system and 2.4 mg/L to the [redacted] system. These values

are both significantly above the water solubility of aclonifen (approximately 1.4 mg/L). The samples were incubated in the dark at 20 ± 2°C.

Analytical procedures

Duplicate flasks from each system were analysed at time zero, 4 hours, 10 hours, 1, 2, 7, 14, 30, 61, 100 and 180 days. The aqueous phase was decanted off and immediately partitioned with dichloromethane for early time-point samples (0 hour, 4 hours, 10 hours and 1 day) to ensure a homogeneous solution. For later samples the water phases were measured directly. The sediments were extracted with acetonitrile, followed by acetonitrile/water (4/1, v/v) at ambient temperature. At later timepoints (2 days onwards) the sediments were further extracted by soxhlet extraction with acetonitrile/water (4/1, v/v). The radioactivity in the aqueous phase (and dichloromethane partition of early aqueous phases) and in each sediment extract was quantified by LSC. The individual components were identified by HPLC analysis in comparison to certified reference standards. The radioactivity remaining in the sediment was determined by combustion and LSC of the trapped combustion gases.

Unextractable radioactivity remaining in the sediment after extraction was further characterised by fractionation of the organic material. This analysis was conducted on the extracted sediment samples from the final time-point. This radioactive material was separated into three fractions:

- Soluble in 0.1 M sodium hydroxide solution and remaining in solution on acidification with hydrochloric acid (the fulvic acid fraction).
- Soluble in 0.1 M sodium hydroxide solution but insoluble on acidification with hydrochloric acid (the humic acid fraction).
- Insoluble in 0.1 M sodium hydroxide solution (the humin fraction).

Selected water samples and sediment extracts were analysed by LC-MS to confirm the identity of aclonifen.

Degradation kinetics

The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details are provided in Document KCA 7.2.2.3/08. A brief summary of the approach for trigger endpoints is provided below.

The data was best fit by the Double First Order in Parallel (DFOP) model in [redacted] total system with a $\chi 2$ error of 6% after removal of two outliers. For consistency the outliers were also removed from the fitting of the water and sediment compartments where best fits to the data were provided by the Hockey Stick (HS) model for the water column and the Simple First Order (SFO) model in the sediment with $\chi 2$ errors of 9 and 8%.

The data was best fit by the Hockey Stick (HS) model in [redacted] total system with a $\chi 2$ error of 6%. Best fits to the data were provided by the Simple First Order (SFO) model for both the water column and sediment with $\chi 2$ errors of 15 and 9%.

H. RESULTS AND DISCUSSION

The measurements of dissolved oxygen showed that the aqueous phase of both sediment/water systems was >49% throughout the 180 day incubation period and therefore aerobic. The redox measurements showed that an oxidising environment (redox potential of > +200 mV) was achieved in both aqueous phases and a reducing environment in both sediments (mean redox potentials -328 mV in the [redacted] and -331 mV in the [redacted] systems). The aqueous phases of the [redacted] system had a mean pH of 7.69 and the aqueous phases of the [redacted] system had a mean pH of 7.67.

A. Mass Balance

The overall mean recoveries of radioactivity were 97% and 94% for the [redacted] and [redacted] systems, respectively. Good recoveries of radioactivity (range 90 to 101.3%) were achieved throughout the study with the exception of the 14 day time-point for the [redacted] system where only 81% was recovered. A summary of the recoveries at each sampling time interval is provided in in Table 7.2.2.3- 4 and Table 7.2.2.3- 5

B. Findings

In both water / sediment systems there was an initial rapid transfer of radioactivity from the water phase to the underlying sediment, followed by a continuing steady transfer. In the [redacted] system, the level of radiolabelled material recovered in the aqueous phase decreased from 96% of applied radioactivity at time zero to 12% after 30 days and 2% after 180 days. In the [redacted] system, the level of radiolabelled material recovered in the aqueous phase decreased from 98% of applied radioactivity at time zero to 17% after 30 days and to 5% after 180 days.

The changing levels of radioactivity in the sediment reflected those for the water, with a rapid initial rise over the first 30 days being followed by the attainment of a relatively steady level. Thus for the [redacted] system, the total radioactivity in the sediment (extractable and non-extractable) rose rapidly to 84% at day 30 and reached 98% by day 100, before declining to 89% by day 180. In the [redacted] system, the total activity in the sediment reached 75% by day 30 and 86% by day 180. The majority of the radioactivity associated with the sediments, initially extractable as aclonifen, became unextractable with time. The portion of the sediment residue that remained unextracted, even after the soxhlet extraction, rose to 77% in the [redacted] system and to 66% in the [redacted] system. In both sediments the unextracted radioactivity was principally associated with the humin fraction (50% and 45%) with less than half those amounts (23% and 16%) associated with the humic acid fraction and the least (3% and 6%) associated with the fulvic acid fraction.

Negligible mineralisation of the compound occurred, as shown by the appearance of very low amounts of radioactivity in the potassium hydroxide traps. A maximum of 1% was evolved from the [redacted] system and 2% from the [redacted] system. No radioactivity was detected in the ethylene glycol traps.

Aclonifen was metabolised in both sediment-water systems at a moderate rate with DT_{50} values of 9.1 days and 11.0 days (DT_{90} values of 294 days and 147 days) for [redacted] and [redacted] systems, respectively. Aclonifen rapidly dissipated from the water phase after application by a combination of degradation and partitioning to the sediment. DT_{50} values of 1.6 days and 3.0 days and DT_{90} values of 9.4 days and 13.4 days were derived for the [redacted] and [redacted] systems, respectively.

Analysis of the water phase from the [redacted] system showed that aclonifen was the major radioactive component up to 7 days after treatment. Three minor metabolites were detected in the water at 2 days and a fourth was detected at 7 days. Three other minor metabolites (bringing the total number of metabolites detected to seven) were detected sporadically. At the end of the study none of the five metabolites still detectable accounted for as much as 1% of applied radioactivity and aclonifen itself had fallen to well below that figure. Three of the metabolites were identified as M-01 (RRT 0.81), M-02 (RRT 0.58) and M-04 (RRT 0.35).

The results from the [redacted] system water phases were similar to those from the [redacted] system. The transfer of radioactivity from the water phase to the sediment was slightly slower in the [redacted] system. Degradates (four) were first detected at 7 days. By the end of the study two were no longer detectable in the water (as was also the case for aclonifen) and one had fallen to <1% of applied radioactivity. The fourth accounted for 2 to 3%. Three other very minor metabolites (bringing the total to seven) were detected by the end of the study. Again, three of the seven compounds were identified as M-01, M-02 and M-04.

Analysis of the sediment extracts indicated that there was initially a rapid transfer of aclonifen from the water to the sediment phase, reaching 53% for the [redacted] system at 7 days and 46% for

the [redacted] system at 2 days. These levels steadily declined until aclonifen only accounted for 3 or 7% of applied radioactivity at the end of the study. In the [redacted] sediment extracts aclonifen was accompanied by a total of six minor metabolites, four of which had been seen in the water phase, including M-01 and M-02. In the [redacted] sediment extracts four minor metabolites were detected at timepoints up to 100 days, three of which corresponded to metabolites seen in the water phase, with M-01 again identified. At the final 180 day timepoint extensive degradation of aclonifen to multiple metabolites occurred in sediment, with a total of 23 minor metabolites detected, including M-01 (see KCA 7.2.2.3/05, [redacted], 2019; M-675436-01-1 for further details of the re-evaluation of [redacted] 180 day sediment extracts).

The following table summarises the maximum levels of these metabolites observed in total sediment-water systems, water and sediment fractions. Of the nine metabolites observed in the [redacted] system, only two were detected at levels in excess of 5%. An unidentified metabolite D was detected at 7% in the water phase at Day 30 but was not observed in excess of 1% at any other timepoint. M-01 was observed in water and sediment throughout the incubation period but did not exceed 5% in either the water or sediment phases throughout the study and only exceeded 5% in the total system at one timepoint (Day 61). Of the numerous metabolites observed in the [redacted] system, only two unidentified metabolites were detected at levels in excess of 5%. Metabolite B was observed in the water phase throughout the incubation period and occasionally in the sediment but did not exceed 5% in either the water or sediment phases throughout the study and only exceeded 5% in the total system at one timepoint (Day 100). Metabolite C was detected at 5% in the water phase at Day 7 before declining to *ca.* 3% by the end of the incubation period. Metabolite E following re-evaluation of the sediment extracts from the final timepoint did not exceed 5% at any timepoint.

Table 7.2.2.3- 3: Maximum Occurrences of Metabolites in Water Sediment Systems

	RRT	Maximum Level Reached (as % of applied radioactivity)					
		[redacted] System			[redacted] System		
		Water Phase	Sediment Phase	Whole System	Water Phase	Sediment Phase	Whole System
M-01 [A]	0.81	4.90	4.74	5.41	4.52	3.22	4.63
M-02 [B]	0.58	3.28	2.29	3.28	4.45	nd	4.45
M-04 [C]	0.35	0.35	nd	0.35	1.16	nd	1.16
Met A	0.66	4.10	nd	4.10	nd	nd	nd
Met B	0.71	nd	1.68	1.68	3.90	4.84	5.18
Met C	0.17	3.53	0.38	3.53	5.44	1.26	5.44
Met D	0.91	7.14	0.59	7.14	nd	nd	nd
Met E	1.06	nd	0.69	0.69	nd	4.42 [D]	4.42 [D]
Met F	0.49	0.14	nd	0.14	nd	nd	nd
Met G	0.51	nd	nd	nd	0.21	nd	0.21
Met H	0.13	nd	nd	nd	0.45	nd	0.45
Number		7	6	9	7	23 [D]	23 [D]

RRT = retention time relative to aclonifen
nd = not detected
[A] Called RPA 407074 in the report
[B] Called RPA 508285 in the report
[C] Called RPA 407288 in the report
[D] Day 180 [redacted] sediment extracts were re-evaluated (see KCA 7.2.2.3/05, [redacted], 2019; M-675436-01-1) as the region of radioactivity assigned as Metabolite E in the original report contained more than 1 peak.

The distribution and recovery of radioactivity in the two systems is shown in Table 7.2.2.3- 4 and Table 7.2.2.3- 5 and the composition of the recovered radioactivity in each compartment is shown in Table 7.2.2.3- 6 and Table 7.2.2.3- 7.

Table 7.2.2.3- 4: Distribution and Recovery of Applied Radioactivity: [redacted] system

Time (days)	DAT										
	0	0.17	0.42	1	2	7	14	30	61	100	180
Water Phase	95.71	90.68	79.73	75.29	45.07	31.00	14.63	11.63	5.03	3.37	2.17
Sediment Extracts	n.a.	7.06	19.24	24.45	50.2	53.39	20.31	26.09	32.95	29.12	12.27
Sediment Unextracted	na	0.07	0.11	0.54	0.98	13.54	55.11	57.57	62.90	68.51	76.53
$^{14}CO_2$	n.a.	n.a.	n.a.	n.a.	n.d.	0.03	0.08	0.20	0.25	0.31	0.75
Organic Volatiles	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Total	95.71	97.81	99.07	100.27	96.25	97.94	90.14	95.48	101.11	101.31	91.72
Mean 96.98%											

n.a. = not applicable, n.d. = not detected

Table 7.2.2.3- 5: Distribution and Recovery of Applied Radioactivity: [redacted] system

Time (days)	DAT										
	0	0.17	0.42	1	2	7	14	30	61	100	180
Water phase	98.47	77.96	68.41	80.80	49.91	41.41	20.74	16.93	10.92	6.59	4.64
Sediment Extracts	n.a.	21.35	30.73	18.43	45.51	36.14	31.71	29.87	28.33	22.78	19.98
Sediment Unextracted	na	0.23	0.11	0.30	1.46	13.39	28.15	45.54	52.15	60.24	65.63
$^{14}CO_2$	n.a.	n.a.	n.a.	n.a.	n.d.	0.05	0.29	1.25	0.97	2.07	1.32
Organic Volatiles	n.a.	n.a.	n.a.	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Total	98.47	99.55	99.24	99.54	96.89	90.99	80.89	93.59	92.37	91.69	91.57
Mean 94.07%											

n.a. = not applicable, n.d. = not detected

Table 7.2.2.3- 6: Characterisation of Radioactivity: [redacted] system

Time (days)	DAT											
	Phase	**0**	**0.17**	**0.42**	**1**	**2**	**7**	**14**	**30**	**61**	**100**	**180**
Aclonifen	Water	95.6	88.6	77.2	71.4	34.3	17.2	2.9	2.0	1.4	0.9	0.2
	Sediment	n.a.	4.6	17.4	21.4	50.2	53.4	17.5	19.9	28.9	25.1	6.9
	Total	95.6	93.2	94.6	92.8	84.5	70.6	20.4	21.9	30.3	26.0	7.1
M-01 [A]	Water	-	-	-	-	4.9	3.0	2.5	0.2	1.3	0.3	0.1
	Sediment	n.a.	-	-	-	-	-	1.1	3.9	4.1	4.0	2.6
	Total	-	-	-	-	4.9	3.0	3.6	4.1	5.4	4.3	2.7
M-02 [B]	Water	-	-	-	-	3.0	3.3	3.0	-	0.7	0.8	0.4
	Sediment	n.a.	-	-	-	-	-	-	2.3	-	-	-
	Total		-	-	-	3.0	3.3	3.0	2.3	0.7	0.8	0.4
M-04 [C]	Water	-	-	-	-	-	-	-	-	-	0.4	0.4
	Sediment	n.a.	-	-	-	-	-	-	-	-	-	-
	Total	-	-	-	-	-	-	-	-	-	-	0.4
Met A (rrt 0.66)	Water	-	-	-	-	2.9	4.0	4.1	0.4	0.7	0.1	0.2
	Sediment	n.a.	-	-	-	-	-	-	-	-	-	-
	Total	-	-	-	-	2.9	4.0	4.1	0.4	0.7	0.1	0.2
Met B (rrt 0.71)	Water	-	-	-	-	-	-	-	-	-	-	-
	Sediment	n.a.	-	-	-	-	-	1.7	-	-	-	0.9
	Total	-	-	-	-	-	-	1.7	-	-	-	0.9
Met C (rrt 0.17)	Water	-	-	-	-	-	3.5	2.2	1.9	0.8	0.8	0.9
	Sediment	n.a.	-	-	-	-	-	-	-	-	-	0.4
	Total	-	-	-	-	-	3.5	2.2	1.9	0.8	0.8	1.3
Met D (rrt 0.91)	Water	-	-	-	-	-	-	-	7.1	-	-	-
	Sediment	n.a.	-	-	-	-	-	-	-	-	-	0.9
	Total	-	-	-	-	-	-	-	7.1	-	-	0.9
Met E (rrt 1.06)	Water	-	-	-	-	-	-	-	-	-	-	-
	Sediment	n.a.	-	-	-	-	-	-	-	-	-	0.7
	Total	-	-	-	-	-	-	-	-	-	-	0.7
Met F (rrt 0.49)	Water	-	-	-	-	-	-	-	-	-	0.1	-
	Sediment	n.a.	-	-	-	-	-	-	-	-	-	-
	Total	-	-	-	-	-	-	-	-	-	0.1	-
Sum of Other Unidentified Metabolite	Water	-	-	-	-	-	-	-	-	-	-	-
	Sediment	na				-	-	-	-	-	-	-
	Total	-	-	-	-	-	-	-	-	-	-	-
Maximum single metabolite	Water	-	-	-	-	4.9	4.0	4.1	7.1	1.3	0.8	0.9
	Sediment	na	-	-	-	-	-	1.9	3.9	4.1	4.0	2.6
	Total	-	-	-	-	4.9	4.0	4.1	7.1	5.4	4.3	2.7

n.a. = not applicable "-" = not detected, rrt = retention time relative to aclonifen
[A] Called RPA 407074 in the report
[B] Called RPA 508285 in the report
[C] Called RPA 407288 in the report

Table 7.2.2.3- 7: Characterisation of Radioactivity: [redacted] system

Time (days)	DAT											
	Phase	0	0.17	0.42	1	2	7	14	30	61	100	180 [D]
Aclonifen	Water	98.3	76.9	65.1	78.2	49.9	23.3	6.6	3.7	4.2	1.3	-
	Sediment	na	16.5	29.2	17.2	45.5	36.1	31.7	29.9	24.4	11.1	2.8 [D]
	Total	98.3	93.5	94.3	95.4	95.4	59.4	38.3	33.6	28.6	12.4	2.8 [D]
M-01 [A]	Water	-	-	-	-	-	4.5	2.3	3.0	1.9	1.4	-
	Sediment	na	-	-	-	-	-	-	-	-	3.2	0.4 [D]
	Total	-	-	-	-	-	4.5	2.3	3.0	1.9	4.6	0.4 [D]
M-02 [B]	Water	-	-	-	-	-	4.4	3.4	2.0	1.7	1.6	0.5
	Sediment	na	-	-	-	-	-	-	-	-	-	-
	Total	-	-	-	-	-	4.4	3.4	2.0	1.7	1.6	0.5
M-04 [C]	Water	-	-	-	-	-	-	-	-	-	-	1.2
	Sediment	na	-	-	-	-	-	-	-	-	-	-
	Total	-	-	-	-	-	-	-	-	-	-	1.2
Met B (rrt 0.71)	Water	-	-	-	-	-	3.7	3.9	3.4	1.5	1.1	-
	Sediment	na	-	-	-	-	-	-	-	-	4.1	2.3 [D]
	Total	-	-	-	-	-	3.7	3.9	3.4	1.5	5.2	2.3 [D]
Met C (rrt 0.17)	Water	-	-	-	-	-	5.4	4.6	4.8	1.7	1.8	2.3
	Sediment	na	-	-	-	-	-	-	-	-	1.3	0.3 [D]
	Total	-	-	-	-	-	5.4	4.6	4.8	1.7	3.1	2.6 [D]
Met E (rrt 1.06)	Water	-	-	-	-	-	-	-	-	-	-	-
	Sediment	na	-	-	-	-	-	-	-	3.9	4.4	1.9 [D]
	Total	-	-	-	-	-	-	-	-	3.9	4.4	1.9 [D]
Sum of Other Unidentified Metabolite	Water	-	-	-	-	-	-	-	-	-	-	0.7
	Sediment	na	-	-	-	-	-	-	-	-	-	12.3 [D]
	Total	-	-	-	-	-	-	-	-	-	-	13.0 [D]
Maximum single metabolite	Water	-	-	-	-	-	5.4	4.6	4.8	1.9	1.8	2.3
	Sediment	na	-	-	-	-	-	-	-	3.9	4.4	2.3
	Total	-	-	-	-	-	5.4	4.6	4.8	3.9	5.2	2.6

n.a. = not applicable "-" = not detected, rrt = retention time relative to aclonifen
[A] Called RPA 407074 in the report
[B] Called RPA 508285 in the report
[C] Called RPA 407288 in the report
[D] Day 180 [redacted] sediment extracts were re-evaluated (see KCA 7.2.2.3/05, [redacted], 2019; M-675436-01-1) regions of radioactivity assigned in the original report contained more than 1 peak.

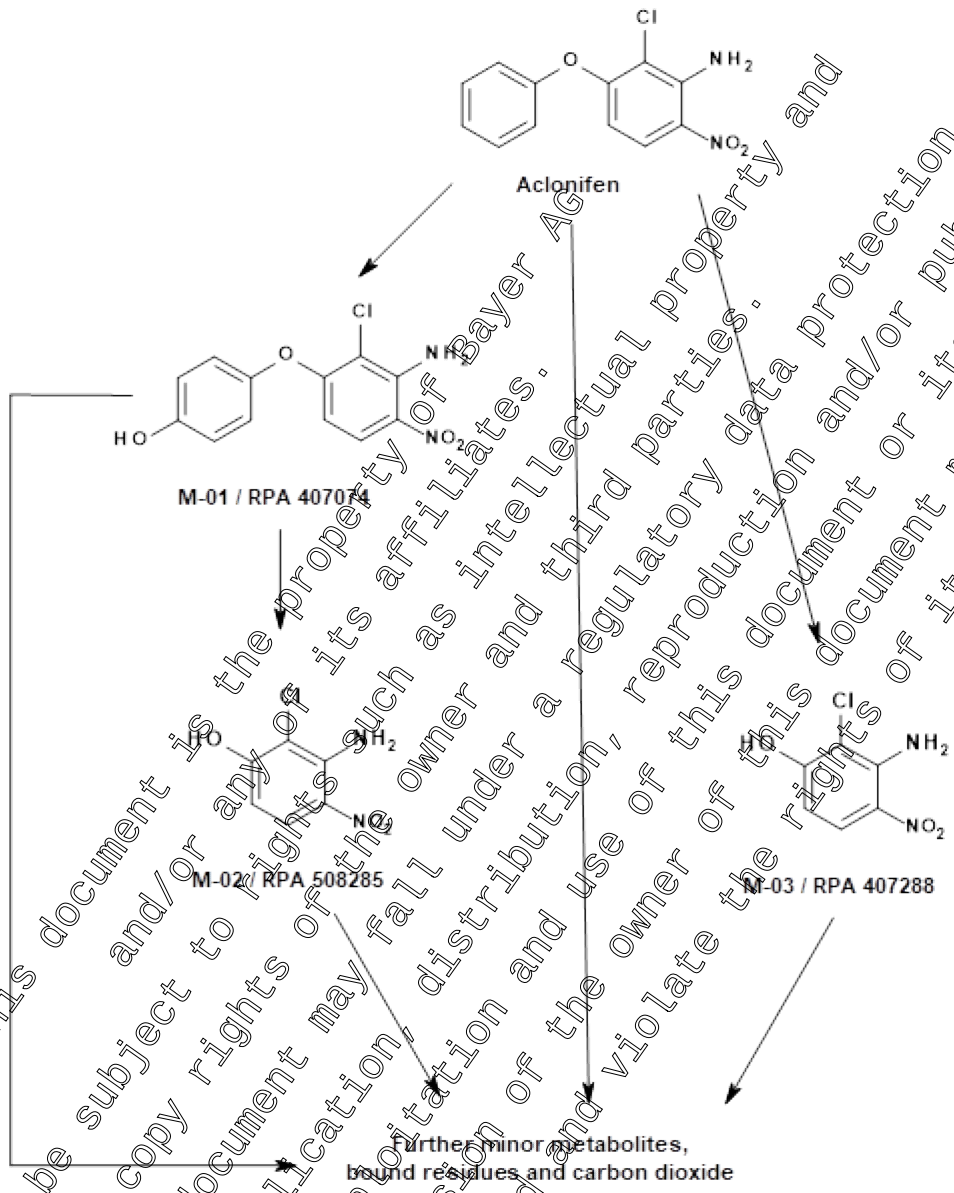
The levels of aclonifen present in the aqueous phase and in the total sediment/water system at each sampling occasion were reported for the multi compartmental model (KIM v 1.0) to determine DT_{50} and DT_{90} values. The degradation kinetics determined in the report were conducted prior to the issuing of the FOCUS guidance document on degradation kinetics and are no longer considered valid. DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.2.2.3/08. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.2.2.3- 8.

Table 7.2.2.3- 8: Trigger endpoint DT_{50} values of aclonifen in aquatic sediment systems determined at 20 °C

System	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted] Total system Outliers excluded	DFOP	95.71	k1 0.147580 k2 0.010100 g 0.387266	5.74	0.1526 0.0271	n.r n.r -0.076303	n.r n.r 0.851	22.81	179.5
[redacted] Water Outliers excluded	HS	95.71	k1 0.47003 k2 0.04400 tb 3.40377	9.069	2.49e-06 0.171917	- 1.86559	- 4.942	1.475	19.37
[redacted] Sediment Outliers excluded	SFO	55.16	k = 0.009975	7.626	0.00172	n.r.	n.r.	69.49	230.8
[redacted] Total system	HS	98.47	k1 6.804e-02 k2 1.165e-02 tb 1.335e+01	5.8371	1.44e-06 3.60e-05 2.231e-05	5.026e-02 7.581e-03 8.840e+00	0.086 0.016 17.866	10.19	133.0
[redacted] Water	SFO	98.47	k = 0.20460	15.29	7.27e-07	0.15055	0.259	3.388	11.25
[redacted] Sediment	SFO	51.31	k = 0.012231	9.366	5.31e-06	-	-	56.67	188.3

The metabolic pathway for [U-^{14}C-aniline] labelled aclonifen in water sediment systems is presented in Figure 7.2.2.3- 1.

Figure 7.2.2.3- 1: Metabolic pathway for [U-^{14}C-aniline] labelled aclonifen in water sediment systems



III. CONCLUSION

Aclonifen dissipated rapidly from the water in water/sediment systems under aerobic conditions in the laboratory in the dark. The calculated best fit DT_{50} values for the dissipation of aclonifen from water were 1.5 and 3.4 days in the water sediment systems. In the total water sediment systems, aclonifen was degraded rapidly. The calculated best fit DT_{50} values for the total systems were 22.8 and 10.2 days.

These results indicate in the environment, any aclonifen reaching water/sediment systems will move to the sediment at an initially rapid rate and then at a steady rate, ultimately leaving very little in the water at all. The formation of unextractable bound residues in the sediment was found to be a major elimination process for aclonifen residues, with degradation of the compound to a number of minor metabolites also observed in both the water and sediment. Aclonifen was degraded by hydroxylation to form M-01 and hydrolysis (of aclonifen or M-01) to form M-02. Under reduced conditions the formation of M-04 was observed on two occasions in the [redacted] system and once in the [redacted] system, possibly as a result of the reduction of M-02 as the reduced forms of aclonifen and M-01 were

not observed. No metabolites were observed in either the water or sediment phase which require further consideration.

Assessment and conclusion by applicant:

The study was conducted according to SETAC (1995) guidelines. The study is considered valid to assess the aerobic aquatic degradation of [aniline-UL-^{14}C] aclonifen in water sediment systems.

Assessment and conclusion by RMS:

Data Point:	KCA 7.2.2.3/02
Report Author:	[redacted]
Report Year:	2000
Report Title:	Aclonifen: Kinetic evaluation of a water/sediment study using TopFit 2.0 model
Report No:	C010084
Document No:	M-198402-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	Current Guideline: FOCUS Degradation Kinetics (2006, 2014) Does Current Guideline: Not meet guideline recommendations.
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

In the previous submission (Addendum to DAR, 2008), this modelling report was evaluated and accepted as valid although not used for risk assessment purposes. However additional studies have been conducted and the requirements of kinetic evaluations according to FOCUS kinetics have changed, thus the report is no longer considered as valid. It has been superseded by KCA 7.2.2.3/08 ([redacted] & [redacted], 2019, M-675507-01-1) and hence a summary is not presented in this dossier.

Data Point:	KCA 7.2.2.3/03
Report Author:	[redacted]
Report Year:	2004
Report Title:	Aclonifen Metabolic fate in the environment: Cleavage of the diphenyl ether bond
Report No:	C034458
Document No:	M-234959-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Not applicable
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

KCA 7.2.2.3/03 is a position paper (M-234959-01-1, [redacted], 2004) prepared for the first inclusion of aclonifen into Annex I of Council Directive 91/414/EEC. It was evaluated, accepted and included in the Aclonifen Draft Assessment Report, Volume 3, B8 Environmental fate and behaviour data, November 2006. The document is a review of the environmental fate studies that have been conducted on aclonifen, focusing on the position of the radiolabel in the test material and the fate and behaviour in the environment of potential metabolites arising from cleavage of the diphenyl ether linkage. For procedural reasons it has to be included in the current dossier however it is now superseded as a water sediment study with [phenoxy-UL-^{14}C]-aclonifen has been conducted.

Data Point:	KCA 7.2.2.3/04
Report Author:	[redacted]
Report Year:	2008
Report Title:	Aclonifen: Kinetic modelling analysis of data from a water sediment study
Report No:	VC/08/016B
Document No:	M-300702-01-1
Guideline(s) followed in study:	EU Council Directive 91/414/EEC as amended by Commission Directive 95/36/EC of July 1995, Section 5, Sub-section 7.2.1.3
Deviations from current test guideline:	Current guideline: FOCUS Degradation Kinetics (2006, 2014) Does not meet guideline recommendation - impact: the study is superseded
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Now is no longer acceptable

In the previous submission (Addendum to DAR, 2008), this modelling report was evaluated and accepted as valid for risk assessment purposes. However additional studies have been conducted and the requirements of kinetic evaluations according to FOCUS kinetics have changed, thus the report is

no longer considered as valid. It has been superseded by KCA 7.2.2.3/08 ([redacted] & [redacted], 2019, M-675507-01-1) and hence a summary is not presented in this dossier.

Data Point:	KCA 7.2.2.3/05
Report Author:	[redacted]; [redacted]
Report Year:	2019
Report Title:	Aclonifen - Statement on the levels of water sediment metabolite E from M-199647-01-1 ([redacted] and [redacted], 2000)
Report No:	VC/19/025A
Document No:	M-675436-01-1
Guideline(s) followed in study:	None
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary

This document summarizes strategies undertaken to address an unidentified metabolite formed in a water sediment study treated with [aniline-UL-^{14}C]-aclonifen (KCA 7.2.2.3/01, M-199647-01-1, [redacted] P. & [redacted] E., 2000). The study was conducted in 2000 at which time there was no need to identify metabolites formed at <10% AR and an unidentified metabolite (Metabolite E) was reported at levels above 5% AR.

Attempts to generate and identify Metabolite E were not successful. However following re-examination of the study raw data it was possible to confirm the reported levels of Metabolite E at the final timepoint in the [redacted] sediment extracts represented a region of radioactivity which contained more than one peak. Re-integration of the extracts concerned has been conducted and is reported in this statement. A total of 24 peaks were quantified in the Day 180 sediment extracts.

Metabolite E following re-evaluation of the final timepoint data did not exceed 5% at any timepoint. Consequently none of the metabolite reported in this study requires further consideration in aclonifen risk assessments.

I. MATERIALS AND METHODS

An unidentified metabolite was reported in [redacted] water sediment system treated with [aniline-UL-^{14}C]-aclonifen (KCA 7.2.2.3/01, M-199647-01-1). Metabolite E was detected eluting ahead of parent aclonifen in sediment extracts when analysed by HPLC, with a relative retention time (RRT) of 1.06. The metabolite was reported in the [redacted] system at the final three timepoints at mean concentrations of 3.95% AR at Day 65, 4.42% AR at Day 100 and 6.97% AR at Day 180, indicating that the maximum formation of Metabolite E may not have been reached in the [redacted] system by the final timepoint.

In extracts of [redacted] sediment the levels were much lower and Metabolite E was detected at the final timepoint only at mean concentrations of 0.69% AR at Day 180.

The metabolite was not detected in the water phase of either system.

New Water Sediment Study (KCA 7.2.2.3/06, M-674479-01-1)

A new water sediment study with [phenoxy-UL-^{14}C]-aclonifen was conducted with two different water sediment systems, Anglersee and [redacted] (KCA 7.2.2.3/06, M-674479-01-1, [redacted] & [redacted], 2019). Additional test systems of system Anglersee were treated with [aniline-UL-^{14}C]-aclonifen, and analysed 104 days after treatment in an attempt to generate and identify Metabolite E.

However attempts to generate the metabolite were not successful.

Re-integration of HPLC data from M-199647-01-1

At Day 61 and Day 100 a single radioactive peak was detected at a RRT of 1.06 in sediment extracts from the [redacted] system but by Day 180 (the final timepoint) the region of radioactivity quantified as Metabolite E contained more than one peak.

The HPLC data from the final timepoint was re-evaluated. A total of 24 peaks were detected in the Day 180 sediment extracts. The environment in the sediments was reductive as shown by the redox potential of *ca.* -330 mV measured throughout the study. Extensive degradation of aclonifen in this manner to multiple minor metabolites is also seen in the anaerobic soil metabolism study (KCA 7.1.1.2/02, M-404038-01-1, Stupp & [redacted], 2011) once the redox potential of the soil becomes reductive.

II. RESULTS AND DISCUSSION

The following table summarizes the maximum levels of all metabolites observed in total sediment water systems, water and sediment fractions throughout the study after Day 180 sediment extracts were re-integrated.

Table 7.2.2.3- 9: Maximum reported levels of metabolites in water sediment systems after re-evaluation

Metabolite	RRT	Maximum Level Reached (as % of applied radioactivity)					
		[redacted] System			[redacted] System		
		Water Phase	Sediment Phase	Whole System	Water Phase	Sediment Phase	Whole System
M-01 [A]	0.81	4.90	4.74	5.41	4.52	3.22	4.63
M-02 [B]	0.58	3.28	2.29	3.28	4.45	nd	4.45
M-04 [C]	0.35	0.35	nd	0.35	1.16	nd	1.16
Met A	0.66	4.10	nd	4.10	nd	nd	nd
Met B	0.71	nd	1.68	1.68	3.90	4.07	5.18
Met C	0.17	3.53	0.38	3.53	5.44	1.26	5.44
Met D	0.91	7.14	0.59	7.14	nd	nd	nd
Met E	1.06	nd	0.69	0.69	nd	4.42	4.42
Met F	0.49	0.14	nd	0.14	nd	nd	nd
Met G	0.51	nd	nd	nd	0.21	nd	0.21
Met H	0.13	nd	nd	nd	0.45	nd	0.45
Number		7	6	9	7	23	23

RRT = retention time relative to aclonifen
nd = not detected
[A] Called RPA 407074 in the report
[B] Called RPA 508285 in the report
[C] Called RPA 407288 in the report

Of the metabolites observed in the [redacted] system, only two were reported at levels in excess of 5%. An unidentified metabolite D was reported at 7% in the water phase at Day 30 but was not observed in excess of 1% at any other timepoint. M-01 was observed in water and sediment throughout the incubation period but was not reported to exceed 5% in either the water or sediment phases throughout the study and only exceeded 5% in the total system at one timepoint (Day 61).

Of the metabolites observed in the [redacted] system, only three unidentified metabolites were originally reported at levels in excess of 5%. Metabolite B was observed in the water phase throughout the incubation period and occasionally in the sediment but was not reported to exceed 5% in either the water or sediment phases throughout the study and was only reported to exceed 5% in the total system at one timepoint (Day 100). Metabolite C was reported at 5% in the water phase at Day 7 before declining to *ca.* 3% by the end of the incubation period. Metabolite E following re-evaluation of the final timepoint did not exceed 5% at any timepoint.

III. CONCLUSION

Following re-integration of HPLC radiochromatograms from KCA 7.2.2.3/01 (M-199647-01-1, [redacted] P. & [redacted] E., 2000) it was possible to establish the levels of an unidentified metabolite (Metabolite E) did not exceed 5% of applied radioactivity at any timepoint in a water sediment study conducted with [aniline-UL-^{14}C]-aclonifen. Consequently none of the metabolite reported in this study requires further consideration in aclonifen risk assessments.

Assessment and conclusion by applicant:

The position paper is considered valid to aid assessment of the route and rate of biological degradation of [aniline-UL-^{14}C]-aclonifen in water sediment systems.

Assessment and conclusion by RMS:

Data Point:	KCA 7.2.2.3/06
Report Author:	[redacted]
Report Year:	2019
Report Title:	Aclonifen: Aerobic aquatic metabolism
Report No:	EnSa-19-0253
Document No:	M-674479-01-1
Guideline(s) followed in study:	OECD Test Guideline No. 308 (2002); Commission Regulation (EU) No 283/2013 in accordance with Regulation (EC) No 1107/2009 (2013); US EPA OCSPP Test Guideline No. 835.4300 / 835.4400 (2008)
Deviations from current test guideline:	Current guideline: OECD 308 (2002) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of [phenoxy-UL-^{14}C]-aclonifen were studied in two water/sediment systems, Anglersee, [redacted], Germany (water pH 7.2, total organic carbon <2.0 mg/L, sediment texture sand, pH 7.1) and [redacted], [redacted] / [redacted], Germany (water pH 7.4, total organic carbon 2.0 mg/L, sediment texture loam, pH 5.2).

A nominal study application rate of 93.5 µg/test system was applied based on a single field application rate of aclonifen of 1800 g/ha.

The test was performed in systems consisting of cylindrical glass containers containing a water-to-sediment volume ratio of 3/1 (v/v) and equipped with traps (permeable for oxygen) for the collection of carbon dioxide and volatile organic compounds. During incubation, the water was in smooth motion. For comparison, sterile test systems were prepared to investigate abiotic degradation of aclonifen.

Duplicate samples were analysed at 0, 0.17, 1, 3, 21, 7, 14, 29, 50, 70 and 100 days of incubation. Additionally, sterile test systems were processed and analysed 100 days after treatment. At each sampling interval, the water was separated from the sediment by decantation and centrifugation. The sediment was extracted three times at ambient temperature using acetonitrile/water 4/1 (v/v). Furthermore, two microwave-assisted extraction steps were performed using acetonitrile/water 4/1 (v/v) at 70 °C and methanol/water 1/1 (v/v) at 50 °C. The amounts of test item and degradation products in water and sediment extracts were determined by liquid scintillation counting (LSC) and by HPLC/radiodetection analysis. The amounts of volatiles and non-extractable residues were determined by LSC and combustion/LSC respectively.

Mean material balances were 98.6% AR for system Anglersee (range from 92.9 to 104.2% AR) and 97.2% AR for system [redacted] Wjctcz (range from 93.0 to 101.5% AR). For sterile samples, mean material balances were 102.2% AR for system Anglersee and 102.3% AR for system.

The amount of radiolabelled material in the water layer in the Anglersee system increased form day 0 to day 1 from 25.7 to 40.8% and then decreased to 1.1% AR at day 100. In the [redacted] system the residues in the water also increased from day 0 to day 0.17 from 5.1 to 39.8% AR and then decreased to <0.1% AR at day 100. The extractable radioactivity in the sediment layer decreased from day 0 to day 100 from 53.5 to 7.3% in the Anglersee system. In the [redacted] system the extractable residues in the sediment increased from 25.8% AR at day 0 to 61.0% AR at day 3 before decreasing to 6.9% AR at day 100. Non-extractable residues (NER) increased in the Anglersee system from day 0 to day 14 from 20.8 to 80.0% before decreasing to 67.3% AR at 100 days. In the [redacted] system NER increased form day 0 to day 29 from 69.2 to 81.7% AR before falling back slightly to 80.7% at 100 days. The maximum amount of carbon dioxide was 16.4 and 8.1% AR at the end of the study in the Anglersee and [redacted] systems, respectively. Formation of volatile organic molecules was insignificant with values of ≤ 0.8% AR.

In the sterile samples taken at 100 days the residues in the water amounted to 7.7% AR in the Anglersee system and 2.3% AR in the [redacted] system. In the sterile samples exactable radioactivity amounted to 84.0% and 86.4% AR in the Anglersee and [redacted] systems, respectively. NER in the sterile flasks reached 10.4% and 13.4% AR at 100 days in the Anglersee and [redacted] systems, respectively.

The amount of aclonifen in the water gradually decreased from 25.7% AR at day 0 to non-detectable amounts from day 14 onwards in the Anglersee system and from 5.1% at day 0 to non-detectable amounts from day 14 onwards in the [redacted] system. The amount of aclonifen in the sediment extracts of the Anglersee system gradually decreased from day 0 to day 100 from 53.5 to 7.3% AR. In the [redacted] system the amount of aclonifen in the sediment extracts gradually increased from day 0 to day 3 from 25.8 to 61.0% AR but then decreased to 4.1% AR by day 100.

The amount of aclonifen in the total system gradually decreased from day 0 to day 100 from 79.2 to 7.3% AR in the Anglersee system. The amount of aclonifen in the total system of the [redacted]

system increased from 30.8% at day 0 to 69.0% AR at day 3 but then decreased to 4.1% AR by day 100.

The amount of aclonifen in the sterile water at day 100 was 7.7% AR in system Anglersee and 2.3% AR in system [redacted]. The amount of aclonifen in the sterile sediment at day 100 was 84.0% AR in system Anglersee and 86.4% AR in system [redacted]. In sterile samples (day 100), the amount of aclonifen in the total system amounted to 91.6% AR in system Anglersee and 88.7% AR in system [redacted].

A re-evaluation of the degradation kinetics in accordance with FOCUS guidance document on degradation kinetics (2014) resulted in best-fit DT_{50} values of 5.0, 0.01 and 8.4 days for the Anglersee total system, water and sediment compartments and DT_{50} values of 4.6, 0.5 and 3.7 days for the [redacted] total system, water and sediment compartments.

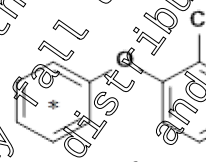
Water/Sediment System		Best Fit Kinetic Model	DT_{50} [days]	DT_{90} [days]	Chi^2 Error [%]
Anglersee	Total system	SFO	5.04	16.75	8.6
	Water	DFOP	0.01	5.58	13.3
	Sediment	SFO	8.43	27.99	19.1
[redacted]	Total system	HS	4.57	30.55	6.8
	Water	FOMC	0.46	2.54	0.6
	Sediment	DFOP	3.70	56.71	3.0

DFOP: double first order in parallel, HS: hockey-stick, SFO: simple first order, FOMC: first order multiple compartment

II. MATERIALS AND METHODS

A. MATERIALS

1. Test material: [phenoxy-UL-^{14}C]-aclonifen



*Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC): 2-chloro-6-nitro-3-phenoxy-aniline
CA registry number: 74070-46-5
Lot or batch number: KML 10278
Specific activity: 6.79 MBq/mg
Radiochemical purity: > 99% (HPLC with radioactivity detector)
Stability of test compound: Shown to be stable under the conditions of the test

2. Water/Sediment: The water/sediment systems were freshly collected from [redacted] and [redacted], two sites in North Rhine-Westphalia, Germany in June 2017. Prior to use, each test water and sediment were sieved through a 0.063 mm and 2 mm sieve respectively. The sediment and water were stored for one day prior to the start of the equilibration.

Table 7.2.2.3- 10: Physicochemical Parameters of the Water/Sediment Systems

Parameter	Anglersee	[redacted]
Geographic Location	[redacted] / North Rhine-Westphalia / Germany	[redacted] / North Rhine-Westphalia / Germany
Batch	20170627	20170627

Parameter		Anglersee	[redacted]
GPS Coordinates		[redacted]	[redacted]
Properties of Sediment			
Texture Class (USDA)		sand	loam
Sand [%]	[50 µm – 2 mm]	95	41
Silt [%]	[2 µm – 50 µm]	2	48
Clay [%]	[< 2 µm]	3	11
pH (sediment:0.01 M $CaCl_2$ 1:2)		7.1	5.2
pH (sediment:water 1:1)		7.4	4.9
% Organic Carbon		0.32	6.2
CEC (meq/100 g)		2.9	7.8
TOC [g/kg]		5.9 / 5.4 / 3.7	77.4 / 73.7 / 77.0
Redox Potential EH [mV] [1]		226.1	-58.9
Moisture [g H2O ad 100 g dry weight]		31.1	202.0
Soil Biomass DAT -1 (BIO-)[3] DAT -102 (BIO- / BIO+)[4, 5]		2.38 3.0 / 3.6	107.6 73.7 / 86.2
Water Parameter			
Temperature (°C)[1]		21.9	19.3
pH[1]		7.21	7.39
Redox potential E_H (mV)[1]		240.9	-5.0
Oxygen saturation [%][1]		106.8	103.4
Total Organic Carbon (TOC) [mg/L][2]		2 / 2 / 12	2 / 2 / 15

[1] Determined on-site immediately after sampling
[2] Measured at start of equilibration / DAT-0 / DAT-100
[3] BIO- samples were left untreated
[4] BIO+ samples were applied with solvent of application solution (200 µL ethanol)
[5] Expressed as mg microbial carbon dioxide per hour per kg of sediment dry weight

B. STUDY DESIGN

In-life dates: 27 June 2017 – 30 October 2019

Experimental conditions

Parameter		Description
Duration of test		100 days
Water and sediment conditions		Fresh water and sediment samples
Concentration in test system	g test item per ha	Nominal: 1800
	µg test item/test system	Nominal: 93.8 (actual:106 for System Anglersee and 111 for [redacted] System), corresponding to 180 µg/L
Control conditions (if used)		Samples for determination of microbial activity: native sediment with and without application solvent
Number of replications	Treatments	Duplicate samples for each sampling interval
	Controls	Duplicate sterile samples for the last sampling interval (DAT-100)
Test apparatus		Cylindrical glass containers
Sediment sample weight [$V_{sediment}$ and $m_{sediment\ DW}$]		175 mL wet sediment per replicate corresponded to Anglersee: approx. 247.2 g dry weight [redacted]: approx. 68.9 g dry weight.

Parameter		Description
Water sample Volume [V_{Water}]		520 mL per replicate
Test material application	Identity of solvent	Ethanol
	Volume of application solution	200 µL per test system
	Application method	Dropwise application using an adjustable pipette onto the water surface
Traps for CO_2 and organic volatiles		The traps were filled with soda lime and polyurethane foam plug. The traps were permeable for oxygen.
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	19.4°C
	Lighting	Dark

Sampling

Parameter		Details
Sampling intervals for the parent/transformation products		Duplicate samples were processed and analysed at 0, 0.17, 1, 3, 21, 7, 14, 29, 50, 70 and 100 after treatment (DAT). Additional sterile test samples were processed and analysed 100 days after treatment.
Sampling procedure		See below
Collection of CO_2 and other volatiles		Soda lime for absorption of carbon dioxide and polyurethane foam for adsorption of volatile organic compounds.
Measurement of sediment water parameters	Moisture content	N/A
	Redox potential	At each sampling interval
	Sterility checks	At last sampling interval for sterile samples
	Oxygen Saturation, pH	At each sampling interval
	Other	The amounts of total organic carbon in water and sediment were determined at the start of equilibration, DAT-0 and DAT-100 for untreated water and sediment. Sediment microbial activity was determined for untreated sediment at start (DAT-1) and at the end of the study (DAT-102), as well as for sediment treated with application solvent at DAT-102.
Sample storage before analysis		Water and sediment were processed immediately after sampling; first HPLC/radiodetection analysis of water and sediment extracts was performed within three days.

Description of analytical procedures

At each sampling interval an aliquot of water was initially removed, and the concentration of any dissolved carbon dioxide determined. The radioactive content of the water was then determined by LSC. Once the physical measurements had been determined the water phase was decanted form the sediment. The sediment was extracted three times at ambient temperature using acetonitrile/water 4/1 (v/v). Furthermore, two microwave assisted extraction steps were performed using acetonitrile/water 4/1 (v/v) at 70 °C and methanol/aqueous 0.1 N HCl 1/1 (v/v) at 50 °C.

Following extraction, sediment samples were lyophilised and homogenised, and the remaining unextracted radioactivity quantified by combustion.

The amounts of test item and degradation products in the water and the sediment extracts were determined by liquid scintillation counting (LSC) and by HPLC/radiodetection analysis. The limit of detection for the primary chromatographic method was determined to be 0.9% AR.

Bound and Extractable Residues

From the 100 day timepoint, sediment samples post extraction were subjected to soil organic matter fractionation into humic acids, fulvic acids and humin fractions. The results indicated that the majority of the non-extractable radioactivity was associated with the humic acid fraction. The recoveries and distribution of radioactivity from humic substance fractionation are shown in Table 7.2.2.3- 13.

Degradation kinetics

The degradation kinetics determined in the report were evaluated according to the FOCUS guidance document on degradation kinetics using the software KinGUI 2. Additionally, DT_{50} and DT_{90} values for the degradation of aclonifen have been re-calculated from the reported data following the recommendations of the FOCUS work group using the software KinGUI (version 2.1) along with all other water sediment data relied on. Full details are provided in Document KCA 7.2.2.3/08. A brief summary of the approach for trigger endpoints is provided below.

The data was best fit by the Simple First Order (SFO) model in Anglersee total system with a χ^2 error of 9%. Best fits to the data were provided by the Double First Order in Parallel (DFOP) model and the Simple First Order (SFO) model for the water column and sediment, respectively, with χ^2 errors of 13 and 19%.

The data was best fit by the Hockey Stick (HS) model in [redacted] total system with a χ^2 error of 7% after removal of two outliers. The best fits to the data were provided by the First Order Multiple Compartment (FOMC) model for the water column and the Double First Order in Parallel (DFOP) model for the sediment with χ^2 errors of 0.6 and 3%, respectively.

II. RESULTS AND DISCUSSION

A. Mass Balance

Mean material balances were 98.6% AR for system Anglersee (range from 92.9 to 104.2% AR) and 97.2% AR for system [redacted] (range from 93.0 to 101.5% AR). For sterile samples, mean material balances were 102.2% AR for system Anglersee and 102.3% AR for system [redacted].

A summary of the recoveries at each sampling time interval is provided in Table 7.2.2.3- 11 and Table 7.2.2.3- 12.

B. Findings

The amount of radiolabelled material in the water layer of the Anglersee system increased from day 0 to day 1 from 25.7% to 40.8% AR before decreasing to 1.1% AR by day 100. In the [redacted] system the amount of radiolabelled material increased from day 0 to day 0.17 from 5.1% to 39.8% AR before decreasing to <0.1% AR by day 100. For the sterile samples, mean material balances were 102.2% AR for Anglersee system and 102.3% AR for the [redacted] system.

The extractable radioactivity in the sediment was approximately 53% AR at day 0 for the Anglersee system, but then gradually decreased to around 7% over the course of the study. In the [redacted] system approximately 26% AR was extractable at day 0, this increased to 61% by day 3 before decreasing to around 6% AR by the end of the study. Non-extractable residues gradually increased, reaching a maximum of 80% AR in the Anglersee system at 14 days before decreasing to 67.3% AR by the end of the study. In the [redacted] system the non-extractables reached a maximum of 82% AR at day 29 where it remained until the end of the study. The maximum amount of carbon dioxide was 16.4 and 8.1% AR at the end of the study in the Anglersee system and [redacted] system, respectively. Formation of volatile organic compounds (VOC) was insignificant as demonstrated by values of ≤ 0.8% AR at all sampling intervals for both water/sediment systems.

In sterile samples taken at day 100 the residues in the water amounted to 7.7% AR in the Anglersee system and 2.3% AR in the [redacted] system. The amounts of carbon dioxide and VOC formed after 100 days in sterile samples of both water/sediment systems were ≤ 0.2% AR and < 0.1% AR, respectively. In sterile samples at day 100 the extractable residues in the sediment amounted to 84.0% AR in the Anglersee system and 86.4% AR and [redacted] system. In the sterile test system, non-extractable residues (NER) at day 100 accounted for 10.4% AR in the Anglersee system and 13.4% AR in the [redacted] system.

The distribution and recovery of radioactivity in the two systems is shown in Table 7.2.2.3- 11 and Table 7.2.2.3- 12.

Aclonifen dissipated from the water due to degradation and translocation into the sediment. The amount of aclonifen in the water gradually decreased from 25.3% AR at day 0 to non-detectable amounts from day 14 onwards in the Anglersee system and from 5.1% AR at day 0 to non-detectable amounts from day 14 onwards in the [redacted] system. The amount of aclonifen in the sterile water at day 100 was 7.7% AR in the Anglersee system and 2.3% AR in the [redacted] system.

No degradation products of aclonifen were identified in the water. The total unidentified residues in the water amounted to a maximum of 3.8% AR at any sampling interval for both water/sediment systems.

The amount of aclonifen in the sediment extracts of the Anglersee system gradually decreased from day 0 to day 100 from 53.5 to 7.3% AR. In the [redacted] system the amount of aclonifen in the sediment extracts increased from day 0 to day 3 from 25.8 to 61.0% AR but then decreased to 4.1% AR by day 100. The amount of aclonifen in the sterile sediment at day 100 was 84.0% AR in the Anglersee system and 86.4% AR in the [redacted] system.

No degradation products of aclonifen were identified in the sediment. The total unidentified residues in the sediment extracts amounted to a maximum of 6.9% AR at any sampling interval for both water/sediment systems and no single compound exceeded 2.3% AR.

The amount of aclonifen in the total system gradually decreased from day 0 to day 100 from 79.2 to 7.3% AR in the Anglersee system. The amount of aclonifen in the total system of the [redacted] system increased from 30.8% AR at day 0 to 69.0% AR at day 3 but then decreased to 4.1% AR by day 100. In sterile samples (day 100), the amount of aclonifen in the total system amounted to 91.6% AR in the Anglersee system and 88.7% AR in the [redacted] system.

No degradation products of aclonifen were identified in the total system. The total unidentified residues amounted to a maximum of 10.7% AR and no single component exceeded 2.3% AR at any sampling interval in both water/sediment systems.

In sterile samples, no degradation products of aclonifen were observed in the water or sediment.

The composition of the radioactivity is shown in Table 7.2.2.3- 14 and Table 7.2.2.3- 15.

Table 7.2.2.3- 11: Material balance of radioactivity in system Anglersee under aerobic conditions expressed as percentage of applied radioactivity

	Repl. No.	DAT										
		0	0.17	1	3	7	14	29[1]	50	70	100[1]	100 sterile
Volatiles												
Carbon Dioxide	A	n.a	<0.1	0.4	1.1	1.2	3.3	6.0	11.8	13.3	16.4	0.2
	B	n.a	<0.1	0.3	1.1	0.4	2.9	[A]	11.8	14.6	[A]	0.2
	Mean	n.a	<0.1	0.3	1.1	0.8	3.1	6.0	11.8	14.0	16.4	0.2
Volatile Organics	A	n.a	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.8	<0.1
	B	n.a	<0.1	<0.1	<0.1	<0.1	<0.1	[A]	<0.1	<0.1	[A]	<0.1
	Mean	n.a	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.8	<0.1
Total Volatiles	A	n.a	<0.1	0.4	1.2	1.2	3.4	6.0	11.9	13.3	17.3	0.2
	B	n.a	0.1	0.3	1.2	0.5	2.9	[A]	11.8	14.6	[A]	0.2
	Mean	n.a	<0.1	0.3	1.2	0.8	3.1	6.0	11.9	14.0	17.3	0.2
Water and Sediment Extractable Residues												
Water	A	26.2	36.5	36.2	14.6	7.0	2.9	3.2	1.4	0.3	1.1	7.6
	B	25.2	33.5	45.3	15.3	7.9	4.8	[A]	2.3	0.2	[A]	7.7
	Mean	25.7	35.0	40.8	15.0	7.4	3.8	3.2	1.9	0.2	1.1	7.7
Sediment Extractable Residues												
Ambient Extract 1	A	42.5	40.6	33.4	43.2	25.8	13.2	7.5	6.2	4.9	4.3	77.3
	B	45.1	41.5	32.4	43.0	26.6	15.4	[A]	6.5	5.2	[A]	82.8
	Mean	43.8	41.0	32.9	43.3	26.0	14.3	7.5	6.4	5.1	4.3	80.0
Microwave Extract 2	A	9.3	5.8	6.2	2.4	3.4	1.5	2.0	2.2	1.0	3.0	3.6
	B	10.1	5.9	5.3	2.8	3.8	3.2	[A]	4.5	2.0	[A]	4.2
	Mean	9.7	5.9	5.8	2.6	3.6	2.8	2.0	3.3	1.5	3.0	3.9
Total Sediment Extractable Residues	A	51.7	46.4	39.7	45.6	28.8	14.7	9.5	8.4	5.8	7.3	80.9
	B	55.2	47.4	37.7	45.8	30.5	19.6	[A]	11.1	7.2	[A]	87.0
	Mean	53.5	46.9	38.7	45.8	29.6	17.2	9.5	9.7	6.5	7.3	84.0
Total Extractable Residues	A	78.0	82.9	75.9	60.5	35.7	17.6	12.7	9.8	6.1	8.4	88.5
	B	80.4	80.9	83.0	61.1	38.3	24.4	[A]	13.4	7.4	[A]	94.7
	Mean	79.2	81.9	79.5	60.8	37.0	21.0	12.7	11.6	6.7	8.4	91.6
Non-Extractable Residues	A	20.5	17.1	26.7	42.7	58.5	84.2	76.0	78.7	75.7	67.3	11.4
	B	21.2	15.3	27.4	41.5	52.3	75.8	[A]	76.0	71.8	[A]	9.5
	Mean	20.8	16.2	24.0	42.1	55.4	80.0	76.0	77.4	73.8	67.3	10.4
Material Balance	A	98.5	100.0	103.4	104.3	95.4	105.2	94.7	100.4	95.2	92.9	100.1
	B	101.5	96.3	104.7	103.8	91.1	103.1	[A]	101.2	93.8	[A]	104.4
	Mean	100.0	98.2	103.9	104.1	93.3	104.2	94.7	100.8	94.5	92.9	102.2
Overall Mean 98.6 ± 4.4 (excludes sterile samples)												

n.a.: not analysed, DAT: days after treatment

[A] Replicate 2 of DAT-29 and DAT-100 is an outlier due to low amounts of NER determined. Therefore, these replicates were excluded from the evaluation.

Table 7.2.2.3- 12: Material balance of radioactivity in system [redacted] under aerobic conditions expressed as percentage of applied radioactivity

	Repl. No.	DAT										
		0	0.17	1	3	7	14	29	50	70	100	100 sterile
Volatiles												
Carbon Dioxide	A	n.a	0.1	0.1	0.4	1.3	2.5	4.7	7.3	7.6	9.7	0.1
	B	n.a	<0.1	0.1	0.3	1.2	3.0	7.4	4.9	6.1	6.6	0.1
	Mean	**n.a**	**<0.1**	**0.1**	**0.4**	**1.3**	**2.8**	**6.1**	**6.1**	**6.8**	**8.1**	**0.1**
Volatile Organic Compounds	A	n.a	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	B	n.a	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
	Mean	**n.a**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**	**<0.1**
Total Volatiles	A	n.a	0.1	0.1	0.4	1.3	2.6	4.7	7.3	7.6	9.7	0.2
	B	n.a	0.1	0.1	0.4	1.2	3.0	7.4	4.9	6.2	6.6	0.2
	Mean	**n.a**	**0.1**	**0.1**	**0.4**	**1.3**	**2.8**	**6.1**	**6.1**	**6.9**	**8.1**	**0.2**
Water and Sediment Extractable Residues												
Water	A	5.3	53.6	14.9	7.4	1.9	1.0	0.3	<0.1	<0.1	0.1	2.3
	B	4.8	26.0	21.7	8.6	3.1	0.8	0.3	<0.1	<0.1	<0.1	2.3
	Mean	**5.1**	**39.8**	**18.3**	**8.0**	**2.5**	**0.9**	**0.3**	**<0.1**	**<0.1**	**<0.1**	**2.3**
Sediment Extractable Residues												
Ambient Extract 1	A	2.4	6.4	8.5	54.7	29.0	13.8	14.8	8.0	7.1	6.0	81.0
	B	15.5	8.4	8.1	60.4	25.1	16.4	9.4	8.0	6.7	5.9	80.5
	Mean	**18.5**	**7.4**	**8.3**	**57.6**	**27.1**	**15.1**	**12.1**	**8.0**	**6.9**	**5.9**	**80.7**
Microwave Extract 2	A	7.2	1.5	4.3	3.3	2.2	1.5	1.2	1.3	0.8	0.9	6.3
	B	7.4	2.3	4.2	3.6	1.9	1.6	1.0	1.3	1.0	1.0	5.1
	Mean	**7.3**	**1.9**	**4.3**	**3.5**	**2.0**	**1.6**	**1.1**	**1.3**	**0.9**	**1.0**	**5.7**
Total Sediment Extractable Residues	A	28.6	7.9	12.9	58.0	31.2	15.3	16.0	9.3	7.9	6.9	87.3
	B	22.9	10.7	12.3	64.0	27.0	18.0	10.3	9.4	7.7	6.9	85.6
	Mean	**25.8**	**9.3**	**12.6**	**61.0**	**29.1**	**16.7**	**13.2**	**9.3**	**7.8**	**6.9**	**86.4**
Total Extractable Residues	A	33.9	61.5	27.7	65.5	33.0	16.3	16.3	9.3	7.4	7.0	89.6
	B	27.8	36.8	34.0	72.6	30.1	18.9	10.6	9.4	7.4	6.9	87.9
	Mean	**30.8**	**49.1**	**30.9**	**69.0**	**31.6**	**17.6**	**13.4**	**9.4**	**7.4**	**7.0**	**88.7**
Non-Extractable Residues	A	65.9	30.9	66.3	35.4	63.6	77.9	79.1	82.8	78.0	75.1	12.8
	B	72.4	56.8	59.9	28.7	66.5	78.0	84.2	79.8	79.8	86.2	14.1
	Mean	**69.2**	**43.8**	**63.1**	**32.0**	**65.0**	**78.0**	**81.7**	**81.3**	**78.9**	**80.7**	**13.4**
Material Balance	A	99.8	92.5	94.0	101.3	97.9	96.8	100.1	99.4	93.0	91.8	102.5
	B	100.2	93.6	94.0	101.7	97.9	99.9	102.3	94.1	93.4	99.7	102.1
	Mean	**100.0**	**93.0**	**94.1**	**101.5**	**97.9**	**98.4**	**101.2**	**96.8**	**93.2**	**95.7**	**102.3**
Overall Mean 97.2 ± 3.1												

n.a.: not analysed, DAT: days after treatment

Table 7.2.2.3- 13: Humic substance fractionation (as % applied radioactivity)

Water/Sediment System	Humin fraction [% AR]	Fulvic acid fraction [% AR]	Humic acid fraction [% AR]	Total [% AR]
Anglersee	6.2	9.3	27.0	42.6
[redacted]	9.3	2.2	40.7	52.1

Table 7.2.2.3- 14: Degradation of aclonifen in system Anglersee under aerobic conditions expressed as percentage of applied radioactivity

Compound	Source	Repl No.	DAT (Mean % AR ± SD)										
			0	0.17	1	3	7	14	29[5]	50	70	100[5]	100 sterile
Aclonifen	Water	Mean	25.7	35.0	40.8	15.6	7.4	1.4	n.d.	n.d.	n.d.	n.d.	7.7
		SD	± 0.8	± 1.5	± 4.5	± 0.4	± 0.5	± 0.0					± 0.1
	Sediment	Mean	53.5	46.9	38.7	45.8	29.6	9.1	3.5	5.3	3.5	7.3	84.0
		SD	± 1.7	± 0.5	± 1.0	± 0.0	± 0.9	± 1.9		± 0.5	± 0.3		± 3.0
	Entire System	Mean[4]	79.2	81.9	79.5	60.8	37.0	10.6	3.5	5.3	3.5	7.3	91.6
		SD	± 1.2	± 1.0	± 3.5	± 0.3	± 1.3	± 0.5		± 0.5	± 0.2		± 3.1
Sum of Unid./Diff. Residues[1]	Water	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	3.8	3.1	1.9	<LOD	1.5	n.d.
		SD						± 1.0	± 0.1	± 0.4		± 0.4	
	Sediment	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	6.9	2.4	4.1	2.3	2.0	n.d
		SD						± 0.2		± 1.5	± 0.4		
	Entire System	Mean[4]	n.d.	n.d.	n.d.	n.d.	n.d.	10.7	5.5	6.0	2.3	3.5	n.d
		SD						± 1.1		±1.9	± 0.4		
Total Extractable Residues[2]	Water	Mean	25.7	35.0	40.8	15.6	7.4	3.8	3.2	1.9	<LOD	1.1	7.7
		SD	± 0.8	± 1.5	± 4.5	± 0.4	± 0.5	± 1.0		± 0.4			± 0.1
	Sediment	Mean	53.5	46.9	38.7	45.8	29.6	16.1	6.6	9.3	5.7	7.3	84.0
		SD	± 1.7	± 0.5	± 1.0	± 0.0	± 0.9	± 2.1		± 1.0	± 0.6		± 3.0
	Entire System	Mean	79.2	81.9	79.5	60.8	37.0	19.9	9.8	11.2	5.7	8.4	91.6
		SD	± 0.8	± 1.5	± 4.5	± 0.4	± 0.5	± 0.0		± 1.4	± 0.6		± 3.1
Carbon Dioxide[3]		Mean	n.a	<0.1	0.3	1.1	0.8	3.1	6.0	11.8	14.0	16.4	0.2
		SD		± 0.0	± 0.0	± 0.0	± 0.4	± 0.2	± 0.8	± 0.0	±0.7		± 0.0
Volatile Organic Compounds[3]		Mean	n.a	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.8	<0.1
		SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0		± 0.1	± 0.0		± 0.0
Non-Extractable Residues[3]		Mean	20.8	16.2	24.0	42.1	55.4	80.0	76.0	77.4	73.8	67.3	10.4
		SD	± 0.3	± 0.9	± 2.6	± 0.6	± 3.1	± 4.2		± 1.3	± 2.0		± 0.9
Total Recovery[2]		Mean	100.0	98.1	103.8	104.1	93.2	103.1	91.8	100.4	93.5	92.9	102.2
		SD	± 1.5	± 1.9	± 0.9	± 0.2	± 2.1	± 1.4		± 0.0	± 0.7		± 0.1

n.d.: not detected, n.a.: not analyzed, DAT: days after treatment, SD: standard deviation

[1] Minor components are summed up to sum of unidentified / diffuse residues.

[2] Difference to Material Balance, values due to rounding errors as well as clean up and chromatographic losses.

[3] Values taken from Material Balance.

[4] Mean values of the entire system could be unequal compared to the sum of the mean values of water and sediment, because mean values of the entire system were calculated from the single replicate values of the entire system and not by summation of the mean values of water and sediment.

[5] Replicate 2 of DAT-29 and DAT-100 is an outlier due to low amounts of NER determined. Therefore these replicates were excluded from the evaluation.

Table 7.2.2.3- 15: Degradation of aclonifen in system [redacted] under aerobic conditions expressed as percentage of applied radioactivity

Compound	Source	Repl No.	DAT (Mean % AR ± SD)										
			0	0.17	1	3	7	14	29	50	70	100	100 sterile
Aclonifen	Water	Mean	5.1	39.8	18.3	8.0	2.5	n.d.	n.d.	n.d.	n.d.	n.d.	2.3
		SD	± 0.2	± 13.8	± 3.4	± 0.6	± 0.6						± 0.0
	Sediment	Mean	25.8	9.3	12.6	61.0	29.1	13.8	10.0	5.8	5.4	4.1	86.4
		SD	± 0.2	± 1.4	± 0.3	± 3.0	± 2.1	± 1.0	± 2.7	± 0.2	± 0.1	± 0.4	± 0.8
	Entire System	Mean[4]	30.8	49.1	30.9	69.0	31.6	13.8	10.0	5.8	5.4	4.1	88.7
		SD	± 3.1	± 12.4	± 3.1	± 3.6	± 1.5	± 1.0	± 2.7	± 0.2	± 0.1	± 0.4	± 0.8
Sum of Unid./Diff. Residues[1]	Water	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	LOD	<LOD	<LOD	<LOD	<LOD	n.d.
		SD											
	Sediment	Mean	n.d.	n.d.	n.d.	n.d.	n.d.	1.7	1.6	3.0	2.0	<LOD	n.d
		SD						± 0.6	± 0.4	± 0.8	± 0.7		
	Entire System	Mean[4]	n.d.	n.d.	n.d.	n.d.	n.d.	2.2	1.6	3.0	2.0	<LOD	n.d
		SD						± 0.1	± 0.4	± 0.8	± 0.7		
Total Extractable Residues[2]	Water	Mean	5.1	39.8	18.3	8.0	2.5	LOD	<LOD	<LOD	<LOD	<LOD	2.3
		SD	± 0.2	± 13.8	± 3.4	± 0.6	± 0.6						± 0.0
	Sediment	Mean[4]	25.8	9.3	12.6	61.0	29.1	15.6	11.6	8.8	7.3	4.9	86.4
		SD	± 2.8	± 1.4	± 0.3	± 3.0	± 2.1	± 1.6	± 2.3	± 0.5	± 0.6	± 0.3	± 0.8
	Entire System	Mean	30.8	49.1	30.9	69.0	31.6	16.0	11.6	8.8	7.3	4.9	86.4
		SD	± 3.1	± 12.4	± 3.1	± 3.6	± 1.5	± 1.1	± 2.3	± 0.5	± 0.6	± 0.3	± 0.8
Carbon Dioxide[3]		Mean	n.a	<0.1	0.1	0.4	1.3	2.8	6.1	6.1	6.8	8.1	0.1
		SD		±0.0	± 0.0	± 0.0	± 0.0	± 0.3	± 1.3	± 1.2	±0.7	± 1.6	± 0.0
Volatile Organic Compounds[3]		Mean	n.a	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
		SD		± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0	± 0.0
Non-Extractable Residues[3]		Mean	69.2	43.8	63.1	32.0	65.0	78.0	81.7	81.3	78.9	80.7	13.4
		SD	± 3.2	± 12.9	± 3.2	± 3.3	± 1.5	± 0.1	± 2.5	± 1.5	± 0.9	± 5.6	± 0.6
Total Recovery[2]		Mean	100.0	93.0	94.1	101.0	97.9	96.8	99.4	96.2	93.1	93.7	102.2
		SD	± 0.2	± 0.5	± 0.1	± 0.2	± 0.0	± 1.4	± 1.6	± 3.2	± 0.4	± 3.7	± 0.2

n.d.: not detected, n.a.: not analyzed, DAT: days after treatment, SD: standard deviation
[1] Minor components are summed up to sum of unidentified / diffuse residues.
[2] Difference to Material Balance values due to rounding errors as well as clean up and chromatographic losses.
[3] Values taken from Material Balance.
[4] Mean values of the entire system could be unequal compared to the sum of the mean values of water and sediment, because mean values of the entire system were calculated from the single replicate values of the entire system and not by summation of the mean values of water and sediment.

The reported DT_{50} values for the dissipation of aclonifen from the water column were 0.01 and 0.12 days and for the degradation of aclonifen in the total system were 4.8 and 6.6 days in Anglersee and [redacted] systems, respectively. The experimental data has been re-evaluated according to the FOCUS guidance document on degradation kinetics (FOCUS, 2014) using the software KinGUI (version 2.1). Full details of the evaluation are provided in the summary for KCA 7.2.2.3/08. The resulting best-fit DT_{50} values for trigger endpoints are summarised below in Table 7.2.2.3- 16.

Table 7.2.2.3- 16: Trigger endpoint DT_{50} values of aclonifen in aquatic sediment systems determined at 20 °C

System	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Anglersee Total system	SFO	101.5	k = 0.13744	8.603	4.21e-09	-	-	5.043	16.75
Anglersee Water	DFOP	101.5	k1 2.248e+02 k2 2.553e-01 g 5.840e-01	13.29	<2e-16 0.00174	- - 4.992e-01	- - 0.669	0.008566	5.584
Anglersee Sediment	SFO	47.4	k = 0.08228	19.1	2.78e-05		-	8.425	27.99
[redacted] Total system Outliers excluded	HS	100.2	k1 1.517e-01 k2 1.815e-02 tb 1.309e+01	6.833	3.75e-10 0.00532 3.00e-07	1.345e-01 6.373e-03 1.040e+01	0.169 0.030 15.784	4.57	30.55
[redacted] Water Outliers excluded	FOMC	100.2	α 1.7753 β 0.9555	0.3774	- -	1.1300 0.2785	2.421 1.633	0.4564	2.54
[redacted] Sediment	DFOP	64.0	k1 0.264793 k2 0.013592 g 0.783847	3.011	1.60e-05 0.00913 -	- - 0.693732	- - 0.874	3.698	56.71

III CONCLUSION

Aclonifen dissipated rapidly from the water in water sediment systems under aerobic conditions in the laboratory in the dark. The calculated best fit DT_{50} values for the dissipation of aclonifen from water were 0.01 and 0.46 days in the water sediment systems. In the total water sediment systems, aclonifen was degraded rapidly. The calculated best fit DT_{50} values for the total systems were 4.6 and 3.7 days.

Formation of carbon dioxide was significant (up to 16.4% AR) at study end indicated the potential for complete mineralization of aclonifen and its degradation products.

Besides carbon dioxide, no degradation products of aclonifen were identified.

Formation of non-extractable residues (NER) was up to 80.7% AR at study end, which is an indication for biotic degradation of aclonifen. In sterile samples, NER accounted to a maximum of 13.4% AR at day 100, indicating that formation of NER is enhanced by microbial activity and thus considered as transformation and detoxification of aclonifen.

Assessment and conclusion by applicant:

The study was conducted in accordance with OECD 308 (2002) and is considered valid to assess the aerobic aquatic degradation of [phenoxy-UL-^{14}C] aclonifen in water sediment systems.

Assessment and conclusion by RMS:

Data Point:	KCA 7.2.2.3/07
Report Author:	[redacted]; [redacted]
Report Year:	2019
Report Title:	Aclonifen: Mobilization from sediment
Report No:	EnSa-19-0254
Document No:	M-674034-01-1
Guideline(s) followed in study:	Related to OECD Test Guideline No. 308 (2002); Commission Regulation (EU) No 283/2013 in accordance with Regulation (EC) No 1107/2009 (2013); Related to US EPA OCSPP Test Guideline No. 835.4300 / 835.4400 (2008)
Deviations from current test guideline:	None guideline study.
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The mobilisation behaviour of [phenoxy-UL-^{14}C]-aclonifen was studied in two water/sediment systems, Anglersee, [redacted], Germany (water pH 7.2, total organic carbon <2.0 mg/L, sediment texture sand, pH 7.1) and [redacted], Germany (water pH 7.4, total organic carbon 2.0 mg/L, sediment texture loam, pH 5.2) under aerobic conditions in the dark at 20 ± 2 °C for 36 days following water exchange after 7 days of incubation.

A nominal application rate of 93.5 µg aclonifen/test system was applied based on a maximum single field application rate of aclonifen of 1800 g/ha.

The test was performed in sterile systems consisting of cylindrical glass containers containing a water-to-sediment volume ratio of 3/1 (v/v) and equipped with traps (permeable for oxygen) for the collection of carbon dioxide and volatile organic compounds.

After 7 days of incubation, the water phase was removed and replaced by fresh, sterile water from the respective water/sediment system. Aliquots of the water from duplicate samples were analysed 7, 9, 11, 17, 22, 28, 32 and 43 days after treatment (DAT), corresponding to 0, 2, 4, 10, 15, 21, 25 and 36 days after water exchange (DAE). At the terminal sampling interval (DAE-36) the test systems were processed completely to establish a material balance.

At each sampling interval, amounts of test item and degradation products in aliquots of the water were determined by liquid scintillation counting (LSC) and by HPLC/radiodetection analysis. At the terminal sampling interval, the water was separated from the sediment by decantation and centrifugation. Then, the sediment was extracted, and water and sediment extracts were analysed by liquid scintillation counting (LSC) and by HPLC/radiodetection analysis. The amounts of volatiles and non-extractable residues were determined by LSC and combustion/LSC, respectively.

After 7 days of incubation the water phase was removed and replaced by untreated, sterilized water from the respective water/sediment system (day of water exchange, DAE-0).

Amounts of aclonifen removed with the spiked water phase at DAE-0 were 25.2% AR in system Anglersee and 8.9% AR in system [redacted]. Amounts of aclonifen related to the sediment fraction (adsorbed and NER) were determined by calculation and amounted to 72.4% AR in system Anglersee and 88.1% AR in system [redacted], respectively.

Test systems were processed completely at the terminal sampling interval (DAE-36). Aclonifen was the only compound (100% ROI) determined in water and sediment extracts. Thus, stability of the test item in sterile test systems was proven for the duration of study and radioactivity determined in water and sediment extracts is equivalent to the amount of aclonifen determined in these compartments.

Total mean material balances at DAE-36 (calculated by summation of the relative radioactivity contents detected in the respective compartments at DAE-36, in the removed water phase at DAE-0 and in the removed water aliquots at all previous sampling dates) amounted to 77.1% AR for system Anglersee and 99.4% AR for system [redacted]. Although mass balance for system Anglersee is below 90% AR, results are usable to evaluate the remobilization potential of aclonifen, since remobilization was determined relatively against the combined adsorbed/NER fraction.

The amounts of carbon dioxide and volatile organic compounds (VOC) were determined at the terminal sampling interval (DAE-36) only and were < 0.1% AR in both water/sediment systems.

Residues in water (equal to aclonifen as it was the only compound detected in the chromatograms with 100% ROI) at DAE-36 were 5.0% AR in system Anglersee and < LOD in system Wiehltalsperr.

Extractable residues in sediment (equal to aclonifen as it was the only compound detected in the chromatograms with 100% ROI) at DAE-36 were 44.4% AR in system Anglersee and 70.0% AR in system [redacted].

Non-extractable residues (NER) were determined at the terminal sampling interval (DAE-36) only and amounted to 2.1% AR in system Anglersee and 19.1% AR in system [redacted].

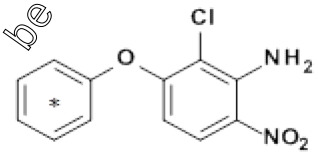
After removal of the treated water phase and replacement with fresh untreated water, the amount of aclonifen in the Anglersee water phase remained almost constant at 7.1% at DAE-2 (5.0% AR) and 7.1% of the determined combined adsorbed/NER fraction at DAE-36 (3.5% AR). It is assumed that the amount of aclonifen determined in the water phase from DAE-2 to DAE-36 is caused by inaccuracy in the calculations as no mass balances were available at these sampling dates. However, decreasing amounts of aclonifen in the water phase from DAE-2 to DAE-36 confirm that aclonifen was not remobilized from Anglersee sediment. In the [redacted] system, amounts of aclonifen in the water phase remained almost constant, amounting to -1.0% of the determined combined adsorbed/NER fraction at DAE-2 (-0.9% AR) and -1.6% at DAE-36 (-1.4% AR), respectively. Slightly negative values could be caused by inaccuracy of calculations, since amounts of aclonifen related to, e.g. the residual water and sediment adsorbed/NER fractions, were determined by calculations, since no mass balances were established for each sampling date. Overall, levels of aclonifen in the water phase were close to zero throughout the study duration from DAE-2 to DAE-36 confirm that aclonifen was not remobilized from the [redacted] sediment.

It is concluded that there is little potential for remobilisation of aclonifen once absorbed to the sediment.

I MATERIALS AND METHODS

A. MATERIALS

1. Test material: [Phenoxy-UL-^{14}C]-aclonifen



* Denotes position of [^{14}C]-radiolabel

Chemical name (IUPAC) 2-chloro-6-nitro-3-phenoxy-aniline

CA registry number: 74070-46-5

Lot or batch number: KML 10278

Specific activity: 6.79 MBq/mg

Radiochemical purity: 99%

Stability of test compound: Shown to be stable under the conditions of the test

2. Water/Sediment: The water/sediment systems were freshly collected from [redacted] and [redacted], two sites in North Rhine-Westphalia, Germany in June 2017. Prior to use, each test water and sediment were sieved through a 0.063 mm and 2 mm sieve respectively. The sediment and water were stored for one day prior to the start of the equilibration.

Table 7.2.2.3- 17: Physicochemical Parameters of the Water/Sediment Systems

Parameter		Anglersee	[redacted]
Geographic Location		[redacted] / North Rhine-Westphalia / Germany	[redacted] / North Rhine-Westphalia / Germany
GPS Coordinates		[redacted]	[redacted]
Properties of Sediment			
Texture Class (USDA)		sand	loam
Sand [%]	[50 µm – 2 mm]	95	41
Silt [%]	[2 µm – 50 µm]	2	48
Clay [%]	[< 2 µm]	3	11
pH (sediment:0.01 M $CaCl_2$ 1:2)		7.1	5.2
pH (sediment:water 1:2)		7.4	4.9
% Organic Carbon		0.32	6.2
CEC (meq/100 g)		2.9	7.8
TOC [g/kg]		5.4	73.7
Redox Potential EH [mV][1]		226.1	-58.9
Moisture [g H2O ad 100 g dry weight]		31.1	202.0
Water Parameter			
Temperature (°C)[1]		21.9	19.3
Ph[1]		7.21	7.39
Redox potential E_H (mV)[1]		240.9	-5.0
Oxygen saturation [%][1]		106.8	103.4
Total Organic Carbon (TOC) [mg/L][2]		2	2

[1] Determined on-site immediately after sampling
[2] Measured at start of equilibration / DAT-0 / DAT-100

B. STUDY DESIGN

In-life dates: 14 July 2017 – 14 November 2019

Experimental conditions

Parameter	Description
Duration of test	43 days
Water and sediment conditions	Fresh sterilized water and sediment samples

Parameter		Description
Concentration in test system	g test item per ha	Nominal: 1800
	µg test item/test system	Nominal: 93.5 (actual:81.1 for System Anglersee and 84.6 for [redacted] System), corresponding to 180 µg/L
Control conditions (if used)		N/A
Number of replications	Treatments	Duplicate samples per water/sediment system
	Controls	N/A
Test apparatus		Cylindrical glass containers
Sediment sample weight [$V_{Sediment}$ and $m_{Sediment\ DW}$]		175 mL wet sediment per replicate corresponded to Anglersee: approx. 247.2 g dry weight [redacted]: approx. 68.9 g dry weight
Water sample Volume [V_{Water}]		520 mL per replicate
Test material application	Identity of solvent	Ethanol
	Volume of application solution	200 µL per test system
	Application method	Dropwise application using an adjustable pipette onto the water surface
Traps for CO_2 and organic volatiles		The traps were filled with soda lime and polyurethane foam plug. The traps were permeable for oxygen.
Is there any indication of the test material absorbing to the walls of the test apparatus?		No
Experimental conditions	Temperature	19.5°C
	Lighting	Dark

Sampling

Parameter		Details
Sampling intervals for the parent/transformation products		After 7 days of incubation, the water phase was removed and replaced by fresh, sterile water from the respective water/sediment system. Aliquots of the water from duplicate samples were analysed 7, 9, 11, 17, 22, 28, 32 and 43 days after treatment (DAT), corresponding to 0, 2, 4, 10, 15, 21, 25 and 36 days after water exchange (DAE). At the terminal sampling interval (DAE-36) the test systems were processed completely to establish a material balance.
Sampling procedure		See below
Collection of CO_2 and other volatiles		Soda lime for absorption of carbon dioxide and polyurethane foam for adsorption of volatile organic compounds.
Measurement of sediment water parameters	Moisture content	N/A
	Redox potential	At terminal sampling interval
	Sterility checks	At terminal sampling interval
	Oxygen Saturation, pH	At terminal sampling interval
	Other	The amounts of total organic carbon in water and sediment were determined at DAT-0 for untreated water and sediment.
Sample storage before analysis		Water and sediment were processed immediately after sampling; first HPLC/radiodetection analysis of water and sediment extracts was performed within three days.

Description of analytical procedures

At the terminal sampling an aliquot of water was initially removed, and the concentration of any dissolved carbon dioxide determined. The radioactive content of the water was then determined by LSC. Once the physical measurements had been determined the water phase was decanted from the sediment. The sediment was extracted three times at ambient temperature using acetonitrile/water 4/1 (v/v). Furthermore, two microwave assisted extraction steps were performed using acetonitrile/water 4/1 (v/v) at 70 °C and methanol/aqueous 0.1 N HCl 1/1 (v/v) at 50 °C.

Following extraction, sediment samples were lyophilised and homogenised, and the remaining unextracted radioactivity quantified by combustion.

The amounts of test item and degradation products in the water and the sediment extracts were determined by liquid scintillation counting (LSC) and by HPLC/radiodetection analysis. The limit of detection for the primary chromatographic method was determined to be 0.9% AR.

II. RESULTS AND DISCUSSION

A. Mass Balance

Total mean material balances at DAE-36 (calculated by summation of the relative radioactivity contents detected in the respective compartments at DAE-36, in the removed water phase at DAE-0 and in the removed water aliquots at all previous sampling dates) amounted to 77.1% AR for system Anglersee and 99.4% AR for system [redacted]. Although mass balance for system Anglersee is below 90% AR, results are usable to evaluate the remobilisation potential of aclonifen, since remobilisation was determined relatively against the combined adsorbed/NER fraction.

A summary of the recoveries is provided in Table 7.2.2.3- 18 and Table 7.2.2.3- 19.

B. Findings

After 7 days of incubation the water was removed and replaced by untreated, sterilised water from the respective water/sediment systems (day of water exchange, DAE-0).

The concentration of aclonifen removed in the spiked water at DAE-0 was 25.2% AR in the Anglersee system and 8.9% AR in the [redacted] system. The concentration of aclonifen in the sediment fraction (adsorbed and NER) was determined by calculation and amounted to 72.4% AR in the Anglersee system and 88.1% AR in the [redacted], system respectively.

The amounts of carbon dioxide and volatile organic compounds (VOC) were determined at the terminal sampling interval (DAE-36) only and were < 0.1% AR in both water/sediment systems.

The concentration of aclonifen in the water at the end of the study was 5.0% AR in the Anglersee system and < LOD in the [redacted] system respectively.

The concentration of aclonifen in the sediment at the end of the study was 44.4% AR in the Anglersee system and 70.0% AR in the [redacted] system respectively. Non-extractable residues (NER) were determined at the end of the study (DAE-36) only and accounted for 2.1% AR in the Anglersee system and 19.1% AR in [redacted] system.

A summary of the degradation of aclonifen in the systems is provided in Table 7.2.2.3- 20 and Table 7.2.2.3- 21.

After removal of the treated water and replacement with untreated water, the concentration of aclonifen in the water in the Anglersee system remained roughly constant 7.1% at DAE-2 (5.0% AR) and 7.1% in the combined adsorbed/NER fraction at DAE-36 (3.5% AR). It is assumed that substantial amounts of aclonifen that are present in the water at DAE-2 to DAE-36 are significant, however this could be the result of inaccuracies introduced in the calculations, as a complete mass balances could not be established for these samplings. The concentration of aclonifen gradually decreased in the water from DAE-2 to DAE-36 showing that aclonifen was not remobilized from the sediment in the

Anglersee system. In the [redacted] system, the concentration of aclonifen in the water also remained roughly constant, amounting to -1.0% of the determined combined adsorbed/NER fraction at DAE-2 (-0.9% AR) and -1.6% at DAE-36 (-1.4% AR), respectively. The negative values are probably the result of inaccuracies introduced into the calculations, as the amount of aclonifen related to, e.g. the residual water and sediment adsorbed/NER fractions, was determined by calculation, as a complete mass balance could not be established for each sampling. Overall as the concentration of aclonifen in the water remained close to zero percent for the duration of the study (DAE-2 to DAE-36) the results show that aclonifen was not remobilized from the sediment in the [redacted] system see Table 7.2.2.3- 22 and Table 7.2.2.3- 23.

Table 7.2.2.3- 18: Material balance of radioactivity in system Anglersee under aerobic conditions expressed as percentage of applied radioactivity

	Repl. No.	DAT							
		0[1]	2	4	10	15	21	25	36
Volatiles									
Carbon Dioxide	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**<0.1**
Volatile Organic Compounds	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**<0.1**
Total Volatiles	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**<0.1**
Water and Sediment Extractable Residues									
Water	A	23.8	6.6	26.8	6.8	6.3	6.0	5.7	5.4
	B	26.7	7.0	7.4	6.6	6.2	5.9	5.3	4.6
	Mean	**25.2**	**6.8**	**7.1**	**6.7**	**6.3**	**5.9**	**5.5**	**5.0**
Sediment Extractable Residues									
Ambient Extract 1	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	48.6
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	37.9
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**43.2**
Microwave Extract 2	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1.1
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	1.2
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**1.1**
Total Sediment Extractable Residues	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	49.6
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	39.1
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**44.4**
Total Extractable Residues	A	23.8	6.6	6.8	6.8	6.3	6.0	5.7	55.0
	B	26.7	7.0	7.4	6.6	6.2	5.9	5.3	43.7
	Mean	**25.2**	**6.8**	**7.1**	**6.7**	**6.3**	**5.9**	**5.5**	**49.4**
Non-Extractable Residues	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	2.0
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	2.2
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**2.1**
Material Balance [2]	A	23.8	6.6	6.8	6.8	6.3	6.0	5.7	57.1
	B	26.7	7.0	7.4	6.6	6.2	5.9	5.3	45.9
	Mean	**25.2**	**6.8**	**7.1**	**6.7**	**6.3**	**5.9**	**5.5**	**51.5**
Mean 77.1									

n.a.: not analysed, DAE: days after water exchange

[1] At day 0, the radioactivity in the removed water phase was determined.

[2] A complete mass balance was only established for DAE-36 since samplings at DAE-0 to DAE-25 were non-destructive.

Table 7.2.2.3- 19: Material balance of radioactivity in system [redacted] under aerobic conditions expressed expressed as percentage of applied radioactivity

	Repl. No.	DAT							
		0[1]	2	4	10	15	21	25	36
Volatiles									
Carbon Dioxide	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**<0.1**
Volatile Organic Compounds	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**<0.1**
Total Volatiles	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	0.1
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**<0.1**
Water and Sediment Extractable Residues									
Water	A	10.0	1.9	1.7	1.5	2.2	1.6	1.7	1.2
	B	7.8	1.2	1.1	1.1	1.5	1.8	1.4	0.7
	Mean	**8.9**	**1.6**	**1.4**	**1.3**	**1.9**	**1.7**	**1.5**	**1.0**
Sediment Extractable Residues									
Ambient Extract 1	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	67.5
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	64.8
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**66.2**
Microwave Extract 2	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	3.7
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	3.9
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**3.8**
Total Sediment Extractable Residues	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	71.2
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	68.7
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**70.0**
Total Extractable Residues	A	10.0	1.9	1.7	1.5	2.2	1.6	1.7	72.4
	B	7.8	1.2	1.1	1.1	1.5	1.8	1.4	69.5
	Mean	**8.9**	**1.6**	**1.4**	**1.3**	**1.9**	**1.7**	**1.5**	**70.9**
Non-Extractable Residues	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	17.6
	B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	20.5
	Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**19.1**
Material Balance [2]	A	10.0	1.9	1.7	1.5	2.2	1.6	1.7	90.0
	B	7.8	1.2	1.1	1.1	1.5	1.8	1.4	90.0
	Mean	**8.9**	**1.6**	**1.4**	**1.3**	**1.9**	**1.7**	**1.5**	**90.0**
Mean 99.4									

n.a.: not analysed, DAE: days after water exchange
[1] At day 0, the radioactivity in the removed water phase was determined.
[2] A complete mass balance was only established for DAE-36 since samplings at DAE-0 to DAE-25 were non-destructive.

Table 7.2.2.3- 20: Degradation of aclonifen in system Anglersee under aerobic conditions expressed as percentage of applied radioactivity

Compound	Source	Repl No.	DAE							
			0 [1]	2	4	10	15	21	25	36
Aclonifen	Water	A	23.8	6.6	6.8	6.8	6.3	6.0	5.7	5.4
		B	26.7	7.0	7.4	6.6	6.2	5.9	5.3	4.6
		Mean	**25.2**	**6.8**	**7.1**	**6.7**	**6.3**	**5.9**	**5.5**	**5.0**
	Sediment	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	49.6
		B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	39.1
		Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**44.4**
	Entire System	A	23.8	6.6	6.8	6.8	6.3	6.0	5.7	55.0
		B	26.7	7.0	7.4	6.6	6.2	5.9	5.3	43.7
		Mean	**25.2**	**6.8**	**7.1**	**6.7**	**6.3**	**5.9**	**5.5**	**49.4**
Diffuse Residues	Water	A	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
		B	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
		Mean	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
Total Extractable Residues[2]	Water	A	23.8	6.6	6.8	6.8	6.3	6.0	5.7	5.4
		B	26.7	7.0	7.4	6.6	6.2	5.9	5.3	4.6
		Mean	**25.2**	**6.8**	**7.1**	**6.7**	**6.3**	**5.9**	**5.5**	**5.0**
	Sediment	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	49.6
		B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	39.1
		Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**44.4**
	Entire System	A	23.8	6.6	6.8	6.8	6.3	6.0	5.7	55.0
		B	26.7	7.0	7.4	6.6	6.2	5.9	5.3	43.7
		Mean	**25.2**	**6.8**	**7.1**	**6.7**	**6.3**	**5.9**	**5.5**	44.4
Carbon Dioxide[3]		A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	<0.1
		B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	<0.1
		Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**<0.1**
Volatile Organic Compounds[3]		A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	<0.1
		B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	<0.1
		Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**<0.1**
Non-Extractable Residues[2]		A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	2.0
		B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	2.2
		Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**2.1**
Total Recovery[2,3]		A	23.8	6.6	6.8	6.8	6.3	6.0	5.7	57.1
		B	26.7	7.0	7.4	6.6	6.2	5.9	5.3	45.9
		Mean	**25.2**	**6.8**	**7.1**	**6.7**	**6.3**	**5.9**	**5.5**	**51.5**

n.d.: not detected, n.a.: not analyzed, DAE: days after water exchange
[1] At day 0, the total removed water phase was analysed by HPLC
[2] Values taken from Material Balance.
[3] A complete mass balance was only established for DAE-36 since samplings at DAE-0 to DAE-25 were non-destructive..

Table 7.2.2.3- 21: Degradation of aclonifen in system [redacted] under aerobic conditions expressed as percentage of applied radioactivity

Compound	Source	Repl No.	DAE							
			0 [1]	2	4	10	15	21	25	36
Aclonifen	Water	A	10.0	1.9	1.7	1.5	2.2	1.6	1.7	1.2
		B	7.8	1.2	1.1	1.1	1.5	1.8	1.4	< LOD
		Mean	**8.9**	**1.6**	**1.4**	**1.3**	**1.9**	**1.7**	**1.5**	**< LOD**
	Sediment	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	71.3
		B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	68.7
		Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**70.0**
	Entire System	A	10.0	1.9	1.7	1.5	2.2	1.6	1.7	72.4
		B	7.8	1.2	1.1	1.1	1.5	1.8	1.4	68.7
		Mean	**8.9**	**1.6**	**1.4**	**1.3**	**1.9**	**1.7**	**1.5**	**70.0**
Diffuse Residues	Water	A	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
		B	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
		Mean	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**	**n.d.**
Total Extractable Residues[2]	Water	A	10.0	1.9	1.7	1.5	2.2	1.6	1.7	1.2
		B	7.8	1.2	1.1	1.1	1.5	1.8	1.4	< LOD
		Mean	**8.9**	**1.6**	**1.4**	**1.3**	**1.9**	**1.7**	**1.5**	**< LOD**
	Sediment	A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	71.2
		B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	68.7
		Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**70.0**
	Entire System	A	10.0	1.9	1.7	1.5	2.2	1.6	1.7	72.4
		B	7.8	1.2	1.1	1.1	1.5	1.8	1.4	69.5
		Mean	**8.9**	**1.6**	**1.4**	**1.3**	**1.9**	**1.7**	**1.5**	**70.9**
Carbon Dioxide[3]		A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	<0.1
		B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	<0.1
		Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**<0.1**
Volatile Organic Compounds[3]		A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	<0.1
		B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	<0.1
		Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**<0.1**
Non-Extractable Residues[2]		A	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	17.6
		B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	20.5
		Mean	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**n.a.**	**19.1**
Total Recovery[2,3]		A	10.0	1.9	1.7	1.5	2.2	1.6	1.7	90.0
		B	7.8	1.2	1.1	1.1	1.5	1.8	1.4	90.0
		Mean	**8.9**	**1.6**	**1.4**	**1.3**	**1.9**	**1.7**	**1.5**	**90.0**

n.d.: not detected, n.a.: not analyzed, DAE: days after water exchange
[1] At day 0, the total removed water phase was analysed by HPLC
[2] Values taken from Material Balance.
[3] A complete mass balance was only established for DAE-36 since samplings at DAE-0 to DAE-25 were non-destructive..

Table 7.2.2.3- 22: Summary of the remobilization of aclonifen from sediment to the water phase of water/sediment system Anglersee under aerobic conditions

DAT	Sample ID	Dissolved in water (from adsorbed + NER) [%]	Dissolved in water (from applied) [%]
7 (DAE-0)	A	n.a.	n.a.
	B	n.a.	n.a.
	Mean	**n.a.**	**n.a.**
9 (DAE-2)	A	6.9	5.0
	B	7.2	5.1
	Mean	**7.1**	**5.0**
11 (DAE-4)	A	7.4	5.3
	B	7.9	5.6
	Mean	**7.6**	**5.4**
17 (DAE-10)	A	7.3	5.3
	B	6.7	4.8
	Mean	**7.0**	**5.0**
22 (DAE-15)	A	6.8	4.9
	B	6.3	4.5
	Mean	**6.5**	**4.7**
28 (DAE-21)	A	6.3	4.5
	B	5.9	4.2
	Mean	**6.1**	**4.4**
32 (DAE-25)	A	6.0	4.3
	B	5.0	3.6
	Mean	**5.5**	**3.9**
43 (DAE-36)	A	7.5	4.1
	B	6.8	3.0
	Mean	**7.1**	**3.5**

n.a.: not applicable; DAT: Days After Treatment; DAE: Days After Exchange (water phase)

Table 7.2.2.3- 23: Summary of the remobilization of aclonifen from sediment to the water phase of water/sediment system [redacted] under aerobic conditions

DAT	Sample ID	Dissolved in water (from adsorbed + NER) [%]	Dissolved in water (from applied) [%]
7 (DAE-0)	A	n.a.	n.a.
	B	n.a.	n.a.
	Mean	**n.a.**	**n.a.**
9 (DAE-2)	A	-1.0	-0.8
	B	-1.1	-0.9
	Mean	**-1.0**	**-0.9**
11 (DAE-4)	A	-1.3	-1.1
	B	-1.2	-1.1
	Mean	**-1.2**	**-1.1**
17 (DAE-10)	A	-1.5	-1.3
	B	-1.2	-1.1
	Mean	**-1.4**	**-1.2**
22 (DAE-15)	A	-0.3	-0.3
	B	-0.5	-0.5
	Mean	**-0.4**	**-0.4**
28 (DAE-21)	A	-1.2	-1.0
	B	-0.1	-0.1
	Mean	**-0.7**	**-0.6**
32 (DAE-25)	A	-1.9	-0.8
	B	-0.6	-0.5
	Mean	**-0.8**	**-0.7**
43 (DAE-36)	A	-1.6	-1.4
	B	-1.5	-1.4
	Mean	**-1.6**	**-1.4**

n.a.: not applicable; DAT: Days After Treatment; DAE: Days After Exchange (water phase)

III. CONCLUSION

Remobilization of aclonifen adsorbed to sediment into the water was between 5.5 to 7.1% in the Anglersee system and between -1.6 and -0.4% in the [redacted] system during a period of 36 days after water exchange.

It is concluded that there is little potential for remobilization of aclonifen once adsorbed or incorporated into the sediment.

Assessment and conclusion by applicant:

This non-guideline study is considered valid to assess the potential for remobilization of aclonifen once adsorbed or incorporated into sediment.

Assessment and conclusion by RMS:

Data Point:	KCA 7.2.2.3/08
Report Author:	[redacted]; [redacted]
Report Year:	2019
Report Title:	Aclonifen: Kinetic evaluation of the degradation in water sediment systems
Report No:	VC/19/025E
Document No:	M-675507-01-1
Guideline(s) followed in study:	none
Deviations from current test guideline:	Current Guideline: FOCUS Degradation Kinetics (2006, 2014) No deviation
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The route and rate of degradation of aclonifen applied to water sediment systems has been studied in the laboratory in two studies, on four aquatic/sediment systems.

The model fit as well as the statistical evaluation of the results were carried out with the software KinGUI, version 2.1. The selection of the most appropriate kinetic model was based on a detailed statistical analysis including visual assessment, χ^2 err. statistics, randomness of residuals, and t-test significance following the FOCUS guidance (2006, 2014).

The resulting DT_{50} values of aclonifen are given below:-

Modelling endpoint DT_{50} values of Aclonifen in aquatic/sediment systems

Phase	Sediment system	Model	St. (χ^2err) (%)	DT_{50} (days)
Total	[redacted]	SFO	8.79	43.81
Total	[redacted]	HS DT_{90}/3.32	5.84	40.06
Total	Anglersee	SFO	8.60	5.04
Total	[redacted]	SFO	12.98	4.80
		Geometric mean		**14.4**
Water Column	[redacted]	SFO	10.61	1.71
Water Column	[redacted]	SFO	15.29	3.39
Water Column	Anglersee	DFOP DT_{90}/3.32	13.29	1.68
Water Column	[redacted]	SFO	3.35	0.83
		Geometric mean		**1.7**
Sediment	[redacted]	SFO	7.63	69.49
Sediment	[redacted]	SFO	9.37	56.67
Sediment	Anglersee	SFO	19.10	8.43
Sediment	[redacted]	FOMC DT_{90}/3.32	4.78	14.00
		Geometric mean		**26.1**

Trigger endpoint DT_{50} values of Aclonifen in aquatic sediment

Phase	Sediment system	Best-fit kinetic	DT_{50} (days)	DT_{90} (days)
Total	[redacted]	DFOP	22.81	179.5
Total	[redacted]	HS	10.19	133.0
Total	Anglersee	SFO	5.04	16.75
Total	[redacted]	HS	4.57	30.55
Water Column	[redacted]	HS	1.48	19.37
Water Column	[redacted]	SFO	3.39	11.25
Water Column	Anglersee	DFOP	0.01	5.58
Water Column	[redacted]	FOMC	0.46	2.54
Sediment	[redacted]	SFO	69.49	230.8
Sediment	[redacted]	SFO	56.67	188.3
Sediment	Anglersee	SFO	8.43	27.99
Sediment	[redacted]	DFOP	3.70	56.71

The calculated DT_{50} values are suitable for use as modelling and trigger endpoints for additional work.

I. MATERIALS AND METHODS

The experimental data generated in the aerobic aquatic–sediment studies [summarised under CA 7.2.2.3/01 and CA 7.2.2.3/06] were evaluated following the guidance in FOCUS Kinetics (2006, 2014a) at level P-I and M-I using the software KinGUI, version 2.1.

The aim of this evaluation was to conduct a kinetic modelling analysis of aclonifen data from aquatic sediment studies in order to derive DT_{50} values for use in subsequent exposure assessments.

The M0 values for each system were determined through free optimisation of parameters in KinGUI, version 2.1. The first timepoint with residues declining below LOD was set to ½ LOD according to FOCUS Kinetics approaches (FOCUS, 2006 2014). Subsequent values below LOD were not included. Values below LOD occurring when previous and subsequent samples included detection at significant levels were excluded as outliers.

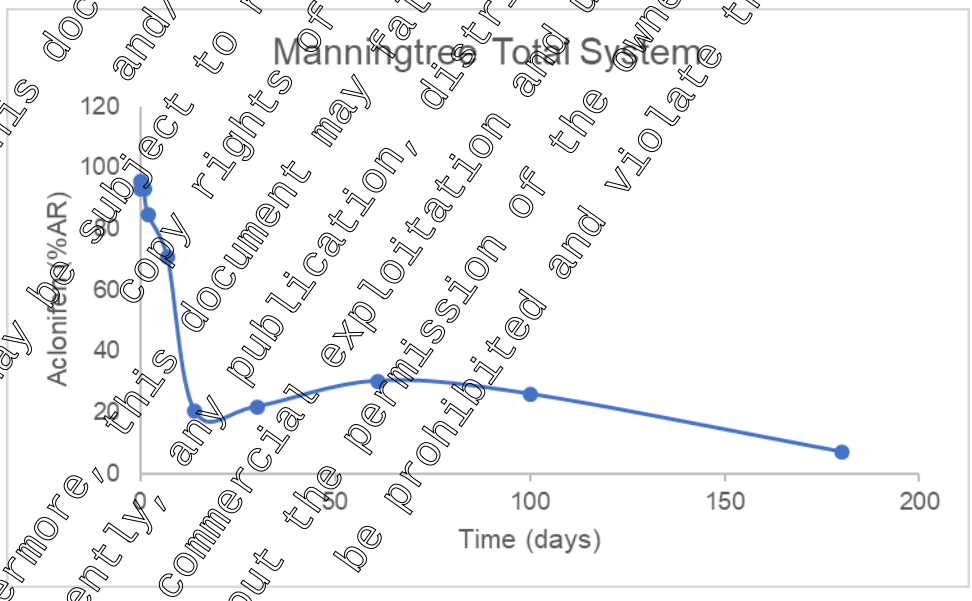
Confidence in the resulting parameters has been assessed visually and from the confidence intervals for the α and β parameters of the first order multicompartment (FOMC) model or probability values for a t-test of the rate parameters for the single first order (SFO), dual first order in parallel (DFOP) and hockey stick (HS) models. Where the parameters for a particular model are not significantly different from zero at the 95th or 90th significance level, it has been concluded that the model is not appropriate to represent the degradation behaviour in that soil. The χ^2 error% parameter has been used to determine goodness of fit and where two models are an appropriate to fit the data, the choice of best fit has been based on the lowest value of this parameter.

All datasets were evaluated against FOCUS Kinetics criteria based on visual assessment, minimum chi^2 error of <15% and t-test parameter significance >=95%. All studies were conducted at 20 °C and normalisation of endpoints was, therefore not required.

II. RESULTS AND DISCUSSION

Results of the kinetic evaluation of two different water sediment studies on aclonifen aquatic system degradation, are given below. In study [redacted] and [redacted], 2000 for derivation of modelling endpoints, the kinetic evaluation of the total system was started by comparing SFO with FOMC and evaluated according the FOCUS, 2006 & 2014 decision tree. The SFO, FOMC, DFOP and HS models were fitted, with their statistics.

The [redacted] system afforded good model fits to the data whilst the [redacted] system resulted in poor visual fits. Evaluation of the data indicates that aclonifen residues dropped rapidly to DAT14 and DAT30 but then rose again significantly, as illustrated below.



The [redacted] system was therefore re-fitted with DAT14 and DAT30 removed as outliers. This resulted in a significant visual improvement for the fits whilst yielding more conservative DT_{50} estimates. The Total System DT_{50} of 43.8 days is considered acceptable as the modelling endpoint value. For consistency, DAT14 and DAT30 data were similarly excluded from the fitting of the water and sediment compartments to derive modelling and trigger endpoints.

Table 7.2.2.3- 24 summarises the calculated DT_{50} values for modelling and trigger endpoints along with their associated statistical and visual assessments.

Table 7.2.2.3- 24: Aclonifen ([redacted] and [redacted], 2000): kinetic and statistical results of the modelling curve fits

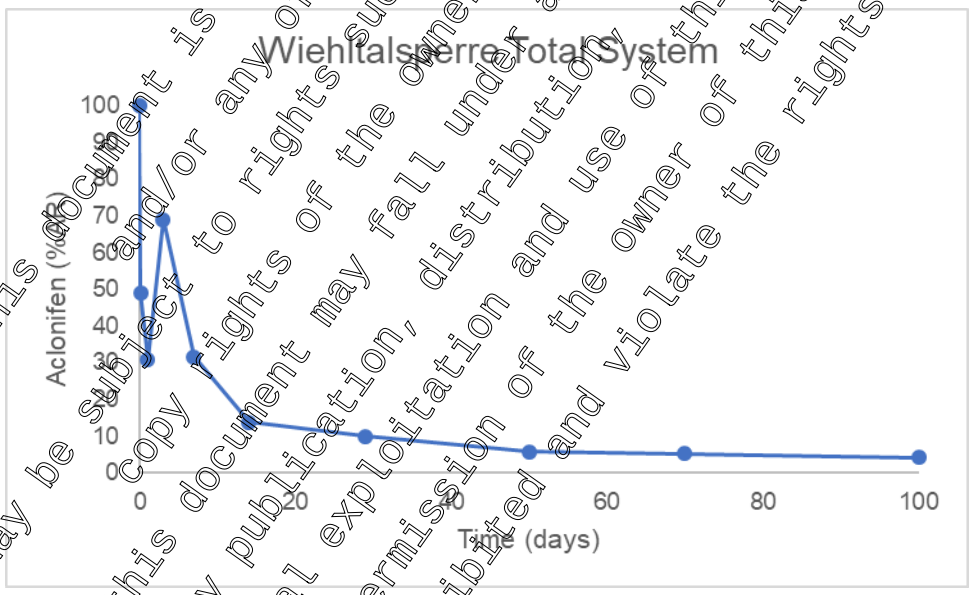
Kinetic model	DT_{50} (d)	DT_{90} (d)	VA	χ^2err (%)	k_1 / α (1/d / -)	k_2 / β (1/d / -)	t_b / g (d / -)	t-test k_1 / k_2 (-)	MS
[redacted] *Total System*									
SFO	*10.59*	*35.19*	-	*19.17*	*0.06544*	-	-	*0.000682*	
FOMC	*9.142*	*193.6*	-	*14.75*	*0.5963*	*4.1600*	-	-	
DFOP	*8.441*	*285.9*	-	*13.16*	*0.159001*	*0.003301*	*0.743028*	*0.0124/ 0.2529*	
HS	*8.419*	*237.9*	-	*11.26*	*0.082327*	*0.004930*	*14.598422*	*3.38e-05/ 0.11796*	
[redacted] **Total System (outliers excluded)**									
SFO	43.81	145.50	o	8.78	0.015822	-	-	1.60e-05	M
FOMC	21.25	320.70	o	9.27	0.7044	12.6884	-	-	
DFOP	22.81	179.50	+	5.74	0.147580	0.010100	0.387266	0.1526/ 0.0271	T
HS	33.56	159.50	+	5.95	0.074029	0.012784	4.211613	0.0719/ 0.0001	
[redacted] **Total System**									
SFO	18.06	59.98	-	13.41	0.038389	-	-	1.53e-05	
FOMC	11.73	149.60	-	8.34	0.7771	8.1486	-	-	
DFOP	10.51	134.30	+	7.36	1.476e-01	1.073e-02	5.774e+01	0.00285/ 0.00361	
HS	10.19	133.00	+	5.84	6.804e-02	1.165e-02	1.335e+01	1.44e-06/ 3.60e-05	M/T
[redacted] *Water Column*									
SFO	*1.72*	*5.71*	o	*11.86*	*0.4032*	-	-	*3.66e-07*	
FOMC	*1.56*	*8.33*	o	*11.39*	*1.9024*	*3.5369*	-	-	
DFOP	*1.50*	*9.14*	o	*11.50*	*0.64893*	*0.10610*	*0.74157*	*0.01230/ 0.15336*	
HS	*1.48*	*9.39*	o	*9.94*	*0.46754*	*0.18511*	*2.00000*	*5.07e-05/ 0.00167*	
[redacted] **Water Column (outliers excluded)**									
SFO	1.71	5.68	o	10.61	0.40521	-	-	8.30e-06	M
FOMC	1.55	9.05	o	10.46	1.6427	2.9555	-	-	
DFOP	1.48	17.03	+	10.22	0.61647	0.03456	0.81993	0.000627/0.172902	
HS	1.48	19.37	+	9.07	0.47003	0.04400	3.40377	2.49e-06/ 0.171917	T
[redacted] **Water Column**									
SFO	3.89	11.25	o	15.29	0.20460	-	-	7.27e-07	M/T
FOMC	2.70	15.38	o	15.06	1.7016	5.3588	-	-	
DFOP	3.00	13.61	+	15.95	0.26361	0.01712	0.90539	0.000736/ 0.274373	
HS	2.96	13.08	o	16.01	0.27018	0.15911	2.00001	0.00723/ 0.01355	

Kinetic model	DT_{50} (d)	DT_{90} (d)	VA	χ^2err (%)	k_1 / α (1/d / -)	k_2 / β (1/d / -)	t_b / g (d / -)	t-test k_1 / k_2 (-)	MS
[redacted] *Sediment*									
SFO	*100.1*	*332.5*	-	*35.65*	*0.006925*	-	-	*0.063771*	
FOMC	*0.08738*	*> 1000*	-	*23.51*	*4.965e-02*	*7.559e-08*	-	-	
[redacted] **Sediment (outliers excluded)**									
SFO	69.49	230.8	o	0.86	0.009975	-	-	0.00172	MT
FOMC	69.49	230.8	o	0.86	1.112e+04	1.115e+06	-	-	
[redacted] **Sediment**									
SFO	56.67	188.3	o	9.37	0.012231	-	-	5.31e-06	MT
FOMC	56.70	188.3	o	10.12	5.041e+05	4.123e+07	-	-	

MS: Model selected (T: for Trigger evaluation; M: for modelling evaluation)

In study [redacted] and [redacted], 2019 for derivation of modelling endpoints, the kinetic evaluation of the total system was again started by comparing SFO with FOMC and evaluated according the FOCUS, 2006 & 2014 decision tree. The SFO, FOMC, DFOP and HS models were fitted, with their statistics.

For the [redacted] system, aclonifen residues dropped very rapidly at DAT0.17 and DAT1 days before increasing significantly again at DAT3, as illustrated below:



The [redacted] system was therefore re-fitted with DAT0.17 and DAT1 removed as outliers. This resulted in a significant visual improvement for the fits whilst yielding more conservative DT_{50} estimates. The Total System DT_{50} of 43.8 days is considered acceptable as the modelling endpoint value. For consistency, DAT0.17 and DAT1 data were similarly excluded from the fitting of the water and sediment compartments to derive modelling and trigger endpoints.

Table 7.2.2.3- 25 summarises the calculated DT_{50} values for modelling and trigger endpoints along with their associated statistical and visual assessments.

Table 7.2.2.3- 25: Aclonifen ([redacted] and [redacted], 2019): kinetic and statistical results of the modelling curve fits

Kinetic model	DT_{50} (d)	DT_{90} (d)	VA	χ^2err (%)	k_1 / α (1/d / -)	k_2 / β (1/d / -)	t_b / g (d / -)	t-test k_1 / k_2 (-)	MS
Anglersee Total System									
SFO	5.04	16.75	o	8.603	0.13744	-	-	4.21e-09	M/T
FOMC	5.043	16.76	o	9.093	7.339e+03	5.339e+04	-	-	
[redacted] ***Total System***									
SFO	*7.33*	*24.35*	-	*41.54*	*0.09455*	-	-	*0.00846*	
FOMC	*0.33*	*271.60*	-	*37.78*	*0.24209*	*0.02010*	-	-	
DFOP	*6.62*	*38.10*	*o*	*45.27*	*0.124229*	*0.005933*	*0.884397*	*0.1279/ 0.4579*	
HS	*6.77*	*52.12*	*o*	*45.18*	*0.10241*	*0.01409*	*17.75676*	*0.0138/ 0.3859*	
[redacted] **Total System (outliers excluded)**									
SFO	4.8	15.95	o	12.98	0.14441	-	-	5.82e-09	M
FOMC	4.4	22.58	o	11.89	2.0714	11.0717	-	-	
DFOP	4.486	19.85	+	8.447	0.173455	0.005740	0.920921	1.85e-07/ 0.262	
HS	4.57	30.55	+	6.883	1.517e-01	1.815e-02	1.309e+01	3.75e-10/ 0.00532	T
Anglersee Water Column									
SFO	0.9436	3.134	o	40.6	0.73460	-	-	0.0246	
FOMC	0.08869	13	o	22.98	0.33140	0.01250	-	-	
DFOP	0.01	5.58	+	13.29	2.248e+02	2.553e-01	5.840e-01	<2e-16/ 0.00174	M/T
HS	0.6877	6.243	o	49.05	1.00791	0.20361	1.28256	0.0455/ 0.3406	
[redacted] ***Water Column***									
SFO	*0.1337*	*0.444*	-	*22.68*	*5.186*	-	-	*0.000684*	
FOMC	*0.1077*	*2.023*	-	*5.734*	*0.65010*	*0.05374*	-	-	
DFOP	*0.1166*	*2.563*	*o*	*1.301*	*9.3817*	*0.3812*	*0.7344*	*0.018590/ 0.099213*	
[redacted] **Water Column (outliers excluded)**									
SFO	0.83	2.76	o	3.35	0.83433	-	-	4.24e-07	M
FOMC	0.46	2.54	o	0.58	1.7753	0.9555	-	-	T
DFOP	0.62	2.53	+	*NaN	1.30116	0.22936	0.87597	0.060630/ 0.075663	
HS	0.82	2.74	-	*NaN	0.84191	0.22992	3.39784	6.56e-07/ 0.07225	
Anglersee Sediment									
SFO	8.43	27.99	o	19.10	0.08228	-	-	2.78e-05	M/T
FOMC	8.43	28	o	20.19	2.560e+03	3.111e+04	-	-	
DFOP	8.099	37.85	+	19.02	9.614e-02	2.337e-14	9.243e-01	0.007717/ 0.500000	
HS	8.217	27.3	+	17.64	8.436e-02	2.337e-14	2.748e+01	4.42e-05/ 0.5000	
[redacted] **Sediment**									

Kinetic model	DT_{50}	DT_{90}	VA	χ^2err	k_1 / α	k_2 / β	t_b / g	t-test k_1 / k_2	MS
	(d)			(%)	(1/d / -)	(1/d / -)	(d / -)	(-)	
SFO	4.822	16.02	-	21.63	0.14374	-	-	2.96e-05	
FOMC	3.34	46.49	+	4.78	0.7372	2.1405	-	-	M
DFOP	3.70	56.71	+	3.01	0.264793	0.013593	0.788847	1.60e-05/ 0.00913	T
HS	3.746	55.96	+	3.71	0.185034	0.017377	7.933497	1.41e-07/ 0.000956	

MS: Model selected (T: for Trigger evaluation; M: for modelling evaluation)

Model selection was described in the preceeding section. Modelling and trigger endpoints for aclonifen are summarised in Table 7.2.2.3- 26 to Table 7.2.2.3- 28. The standard EFSA template can be seen in Table 7.2.2.3- 29 and graphical representations of the best fit models in Table 7.2.2.3- 30.

Table 7.2.2.3- 26: Aquatic system (Total) degradation DT_{50} values of aclonifen for modelling and trigger purposes

Study	System	Model fitted	DT_{50} modelling (d)	DT_{50} persistence (d)	DT_{90} persistence (d)
[redacted] and [redacted] (2000)	[redacted]	SFO/DFOP	42.81	22.81	179.50
[redacted] and [redacted] (2000)	[redacted]	HS	40.06	10.19	133.1
[redacted] and [redacted] (2019)	Anglersee	SFO	5.04	5.04	16.75
[redacted] and [redacted] (2019)	[redacted]	SFO/HS	4.80	4.57	30.55
		Geomean	**14.4**		

Table 7.2.2.3- 27: Aquatic system (Water Column) dissipation DT_{50} values of aclonifen for modelling and trigger purposes

Study	System	Model fitted	DT_{50} modelling (d)	DT_{50} persistence (d)	DT_{90} persistence (d)
[redacted] and [redacted] (2000)	[redacted]	SFO/HS	1.71	1.48	9.94
[redacted] and [redacted] (2000)	[redacted]	SFO	3.39	3.39	11.25
[redacted] and [redacted] (2019)	Anglersee	DFOP	1.68	0.01	5.58
[redacted] and [redacted] (2019)	[redacted]	SFO/FOMC	0.83	0.46	2.54
		Geomean	**1.7**		

Table 7.2.2.3- 28: Aquatic system (Sediment) degradation DT_{50} values of aclonifen for modelling and trigger purposes

Study	System	Model fitted	DT_{50} modelling (d)	DT_{50} persistence (d)	DT_{90} persistence (d)
[redacted] and [redacted] (2000)	[redacted]	SFO	69.49	69.49	230.8
[redacted] and [redacted] (2000)	[redacted]	SFO	56.67	56.67	188.3
[redacted] and [redacted] (2019)	Anglersee	SFO	8.43	8.43	27.99
[redacted] and [redacted] (2019)	[redacted]	FOMC/DFOP	14.00	3.70	56.71
		Geomean	**26.1**		

Table 7.2.2.3- 29: Standard EFSA template for kinetic fitting

Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
Total system	SFO	95.71	k = 0.06544	19.17	0.000682	82.47934	113.802	10.59	35.19
	FOMC	95.71	α 0.5963 β 4.1600	14.75	n.r. n.r.	0.1374 -3.3490	1.055 11.669	9.142	193.6
	DFOP	95.71	k1 0.129001 k2 0.003301 g 0.743028	13.16	0.0124 0.2529 n.r.	n.r n.r 0.518864	n.r n.r 0.967	8.441	285.9
	HS	95.71	k1 0.082327 k2 0.004930 tb 14.598422	14.26	3.38e-05 0.11796	- - 6.112364	- - 23.084	8.419	237.9
Total system Outliers excluded	SFO	95.71	k = 0.015822	8.778	1.603-05	83.485821	98.272	43.81	145.5
	FOMC	95.71	α 0.7044 β 12.6884	6.273	n.r. n.r.	0.2061 -6.8515	1.203 32.228	21.25	320.7
	DFOP	95.71	k1 0.147580 k2 0.010100 g 0.387266	5.74	0.1526 0.0271	n.r n.r -0.076303	n.r n.r 0.851	22.81	179.5
	HS	95.71	k1 0.074029 k2 0.012784 tb 4.311613	5.951	0.0719 0.0001	- - 1.699969	- - 10.323	33.56	159.5
Water	SFO	95.71	k = 0.4032	11.86	3.66e-07	88.6438	105.501	1.719	5.711
	FOMC	95.71	α 1.9024 β 3.5369	11.39	n.r. n.r.	0.1913 -0.8700	3.614 7.944	1.555	8.328
	DFOP	95.71	k1 0.64893 k2 0.10610 g 0.74157	11.5	0.01230 0.15336	n.r n.r 0.24158	n.r n.r 1.242	1.503	9.136
	HS	95.71	k1 0.46754 k2 0.18514 tb 2.00600	9.939	5.07e-05 0.00167	- - -0.37496	- - 4.375	1.483	9.387
Water Outliers excluded	SFO	95.71	k = 0.40521	10.61	8.30e-06	-	-	1.711	5.682
	FOMC	95.71	α 1.6427 β 2.9555	10.46	n.r n.r	-0.3286 -1.9869	3.614 7.898	1.552	9.051
	DFOP	95.71	k1 0.61047 k2 0.03456 g 0.81993	10.22	0.000627 0172902 -	- - 0.65046	- - 0.989	1.482	17.03
	HS	95.71	k1 0.47003 k2 0.04400 tb 3.40377	9.069	2.49e-06 0.171917	- - 1.86559	- - 4.942	1.475	19.37
Sediment	SFO	55.16	k = 0.10	35.65	0.063771	n.r.	n.r.	100.1	332.5
	FOMC	55.16	α 4.965e-02 β 7.559e-08	23.51	n.r n.r	-2.383e-01 -8.724e-06	0.338 0.000	0.08738	>1000

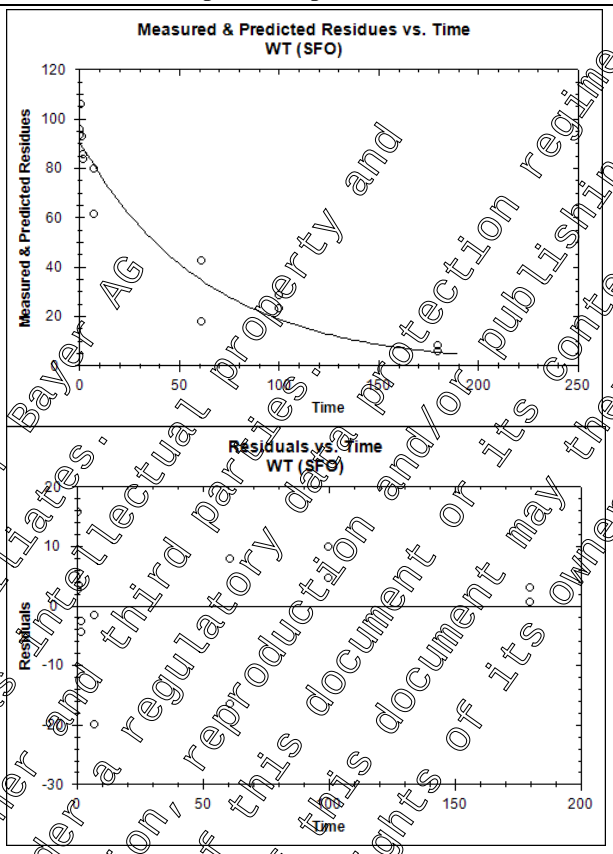
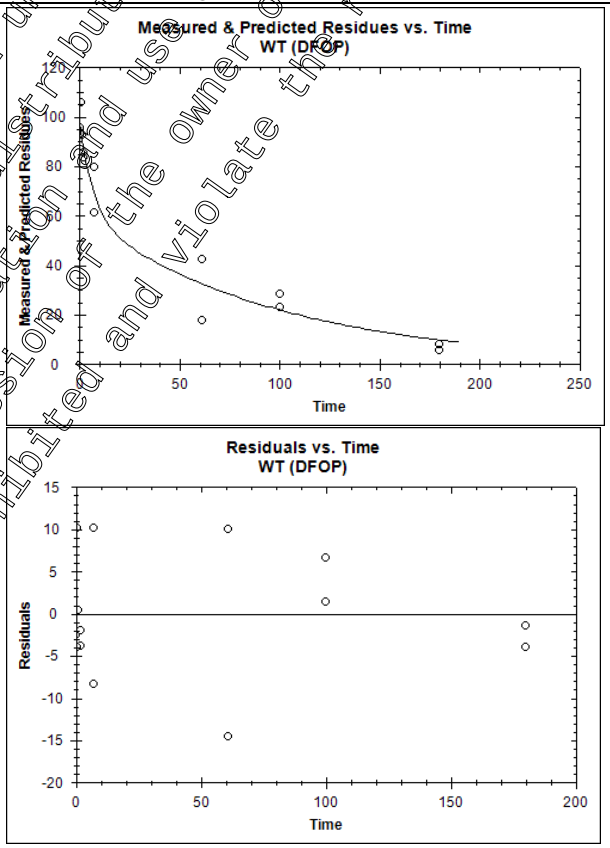
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted] Sediment Outliers excluded	SFO	55.16	k = 0.009975	7.626	0.00172	n.r.	n.r.	69.49	230.8
	FOMC	55.16	α 1.112e+04 β 1.115e+06	9.524	n.r n.r	6.010e+03 1.115e+06	1.624e+04 1.115e+06	69.49	230.8
[redacted] Total system	SFO	98.47	k = 0.038389	13.41	1.53e-05	0.025272	0.052	18.06	59.98
	FOMC	98.47	α 0.7771 β 8.1486	8.342	n.r n.r	0.4034 0.4111	1.151 15.886	11.73	149.6
	DFOP	98.47	k1 1.476e-01 k2 1.0743e-02 g 5.773e-01	7.358	0.00285 0.00361 5.61e-06	5.884e-02 4.034e-03 4.069e-01	0.236 0.017 0.748	10.51	134.3
	HS	98.47	k1 6.804e-02 k2 1.165e-02 tb 1.335e+01	5.8371	1.44e-06 3.60e-05 231e-05	5.026e-02 7.531e-03 8.840e+00	0.086 0.016 17.866	10.19	133
[redacted] Water	SFO	98.47	k = 0.20460	15.29	7.27e-07	0.15055	0.259	3.388	11.25
	FOMC	98.47	α 1.7016 β 5.3588	15.06	n.r n.r	0.1040 -1.9924	3.299 12.710	2.695	15.38
	DFOP	98.47	k1 0.26361 k2 0.01712 g 0.90539	15.95	0.000738 0.274323 1.19e-07	0.13252 -0.03748 0.71389	0.395 0.072 1.097	3.004	13.61
	HS	98.47	k1 0.27018 k2 0.15911 tb 2.00001	16.01	0.00723 0.01355 -	- - -6.84129	- - 10.841	2.96	13.08
[redacted] Sediment	SFO	51.31	k = 0.012231	9.366	5.31e-06	-	-	56.67	188.3
	FOMC	51.31	α 5.041e+05 β 4.123e+07	10.12	n.r n.r	5.041e+05 4.123e+07	5.041e+05 4.123e+07	56.7	188.3
Anglersee Total system	SFO	101.5	k = 0.13744	8.603	4.21e-09	-	-	5.043	16.75
	FOMC	101.5	α 7.339e+03 β 5.339e+04	9.093	n.r n.r	-2.902e+07 -2.112e+08	2.904e+07 2.113e+08	5.043	16.76
Anglersee Water	SFO	101.5	k = 0.73460	40.6	0.0246	-	-	0.9436	3.134
	FOMC	101.5	α 0.33140 β 0.01250	22.98	n.r n.r	0.11037 -0.02190	0.552 0.047	0.08869	13
	DFOP	101.5	k1 2.248e+02 k2 2.553e-01 g 5.840e-01	13.29	<2e-16 0.00174	- - 4.992e-01	- - 0.669	0.008566	5.584
	HS	98.50	k1 1.00791 k2 0.20361 tb 1.28256	49.05	0.0455 0.3406 0.2660	-0.02078 -0.73291 -2.56715	2.037 1.140 5.132	0.6877	6.243
Anglersee Sediment	SFO	47.4	k = 0.08228	19.1	2.78e-05	-	-	8.425	27.99
	FOMC	47.4	α 2.560e+03 β 3.111e+04	20.19	n.r n.r	-6.120e+06 -7.439e+07	6.125e+06 7.446e+07	8.425	28
	DFOP	47.4	k1 9.614e-02 k2 2.337e-14 g 9.243e-01	19.02	0.007717 0.500000 0.000131	2.934e-02 -6.707e-02 5.691e-01	0.163 0.067 1.279	8.099	37.85
	HS	47.4	k1 8.436e-02 k2 2.337e-14 tb 2.748e+01	17.64	4.42e-05 0.5000 0.0114	5.572e-02 -3.906e-02 6.833e+00	1.461e-02 1.993e-02 1.054e+01	8.217	27.3
[redacted] Total system	SFO	100.2	k = 0.09455	41.54	0.00846	-	-	7.331	24.35
	FOMC	100.2	α 0.24209 β 0.02010	37.78	n.r n.r	0.09566 -0.04145	0.389 0.082	0.3321	271.6
	DFOP	100.2	k1 0.124229 k2 0.005933 g 0.884397	45.27	0.1279 0.4579 -	- - 0.087595	- - 1.681	6.615	38.1
	HS	100.2	k1 0.10241 k2 0.01409 tb 17.75676	45.18	0.0138 0.3859 -	- - -22.59439	- - 58.108	6.768	52.12

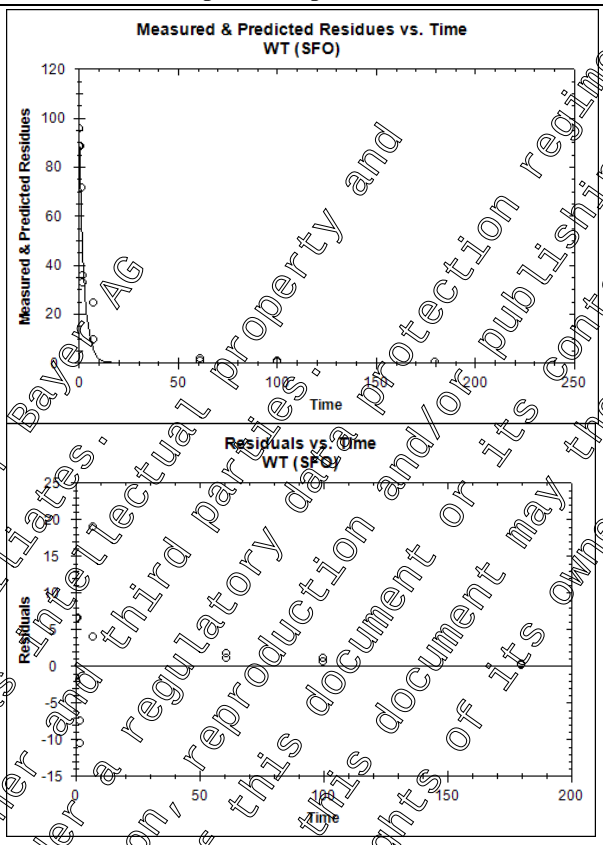
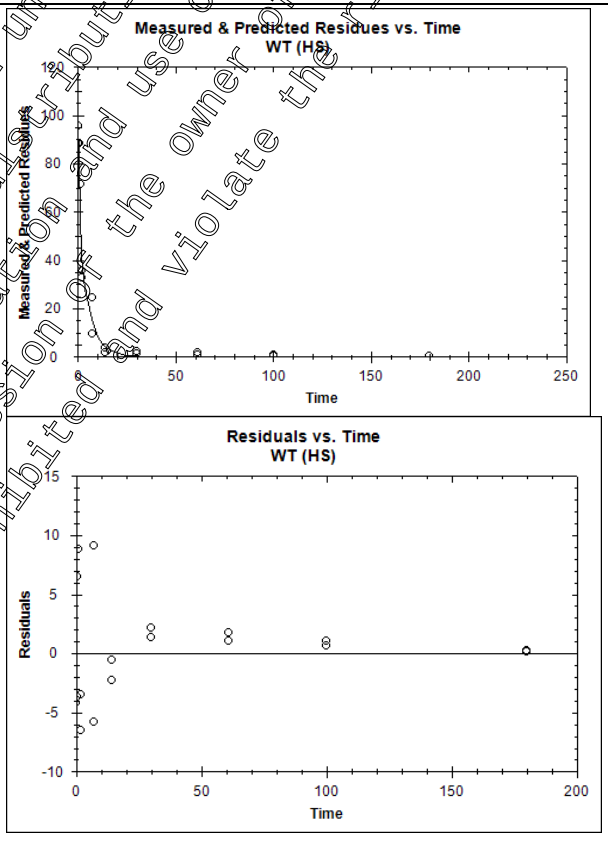
Soil	Kinetic model	M_0	Parameter (k, k1, k2, g, tb, a, β)	X^2, %-error	Prob >t	Lower CI	Upper CI	DT_{50} [days]	DT_{90} [days]
[redacted] Water	SFO	100.2	k = 5.186	22.68	0.000684	-	-	0.1337	0.444
	FOMC	100.2	α 0.63010 β 0.05374	5.734	n.r n.r	0.27406 -0.02025	0.986 0.128	0.1077	2.023
	DFOP	100.2	k1 9.3817 k2 0.3812 g 0.7344	1.304	0.018590 0.099213 -	- - 0.4944	- - 0.974	0.1466	2.363
[redacted] Sediment	SFO	64.0	k = 0.14374	21.63	2.96e-05	-	-	4.822	16.02
	FOMC	64.0	α 0.7372 β 2.1405	4.782	n.r n.r	0.5612 0.8071	0.943 3.474	3.34	46.49
	DFOP	64.0	k1 0.264793 k2 0.013593 g 0.783847	3.011	1.60e-05 0.00913 -	- - 0.693732	- - 0.874	3.698	56.71
	HS	64.0	k1 0.185034 k2 0.017377 tb 7.933497	3.71	2.41E-07 0.000956 4.22E-06	0.154935 0.009213 6.061671	0.215 0.026 9.805	3.746	55.96
[redacted] Total system Outliers excluded	SFO	100.2	k = 0.14441	12.98	5.82E-09	0.12041	0.168	4.8	15.95
	FOMC	100.2	α 2.0704 β 11.0717	11.89	0.0108 0.0375	0.5171 -0.1375	3.626 22.281	4.4	22.58
	DFOP	100.2	k1 0.173453 k2 0.005740 g 0.920921	8.147	1.85e-07 0.262 1.58e-01	0.139358 -0.011378 0.840426	0.208 0.023 1.000	4.486	19.85
	HS	100.2	k1 1.517e-01 k2 1.815e-02 tb 1.309e+01	6.833	3.75e-10 0.00532 3.00e-07	1.345e-01 6.373e-03 1.040e+01	0.169 0.030 15.784	4.57	30.55
[redacted] Water Outliers excluded	SFO	100.2	k = 0.83433	3.345	4.24e-07	-		0.8308	2.76
	FOMC	100.2	α 1.7753 β 0.9555	0.5774	- -	1.1300 0.2785	2.421 1.633	0.4564	2.54
	DFOP	100.2	k1 1.30416 k2 0.22936 g 0.87597	*NaN	0.060630 0.075663 -	- - 0.63868	- - 1.113	0.6173	2.565
	HS	100.2	k1 0.84191 k2 0.22992 tb 3.39784	6.833	6.56e-07 0.07225 -	- - 1.71899	- - 5.077	0.8233	30.55

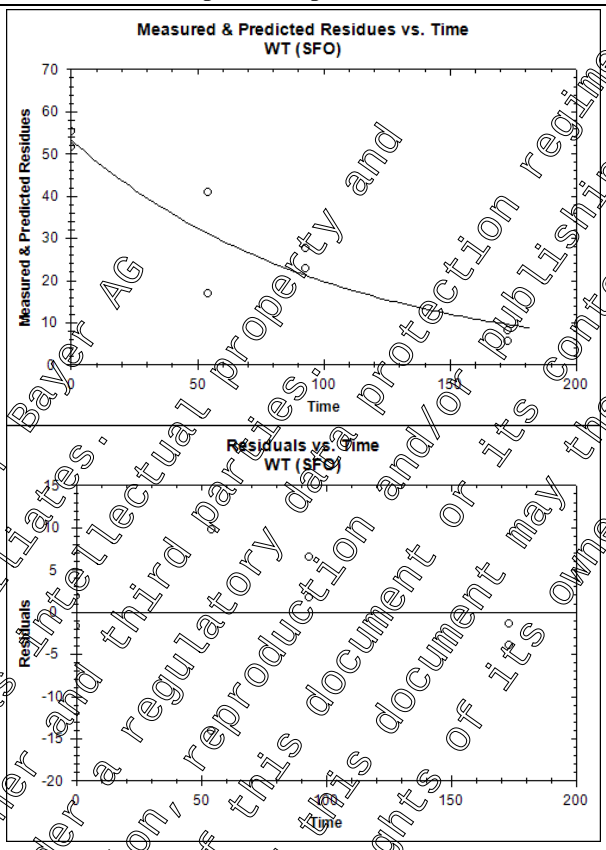
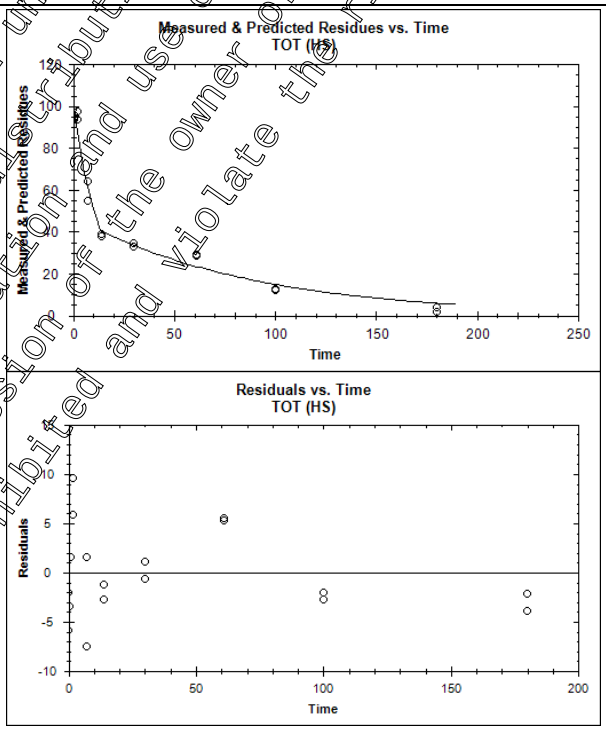
* Degrees of freedom <=0, therefore no Chi2 error can be calculated

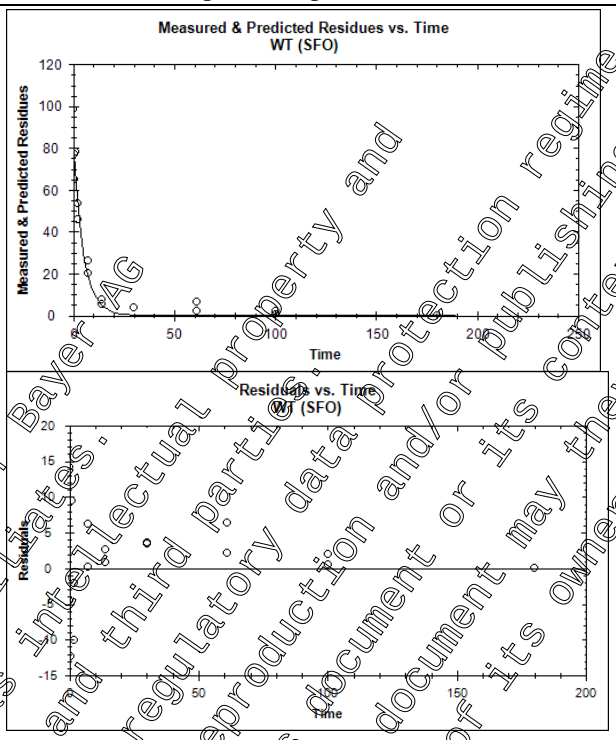
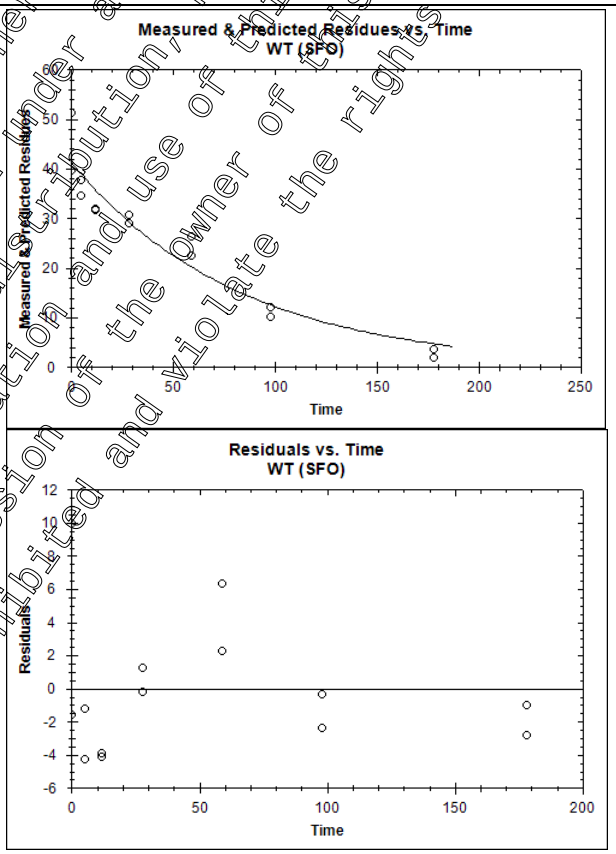
Table 7.2.2.3- 30: Graphical representations of the best fit model in aquatic systems

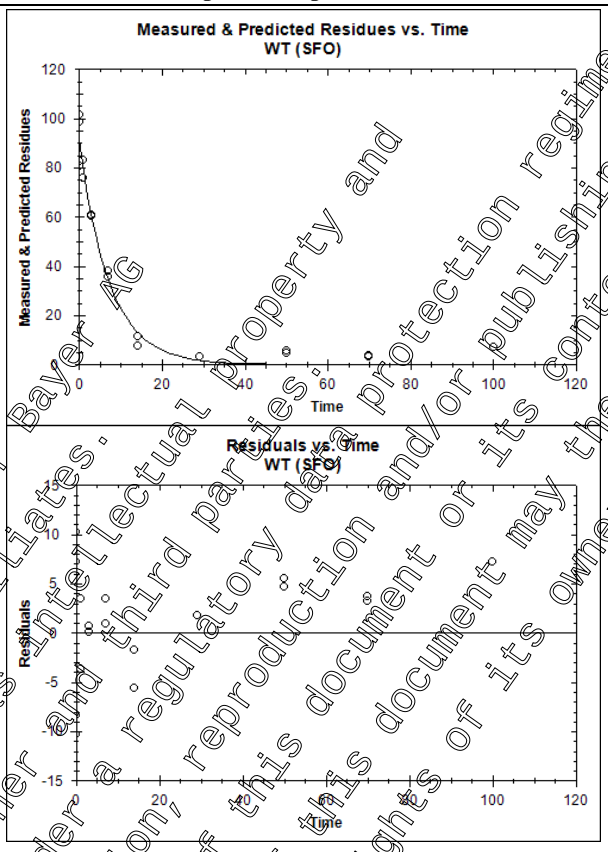
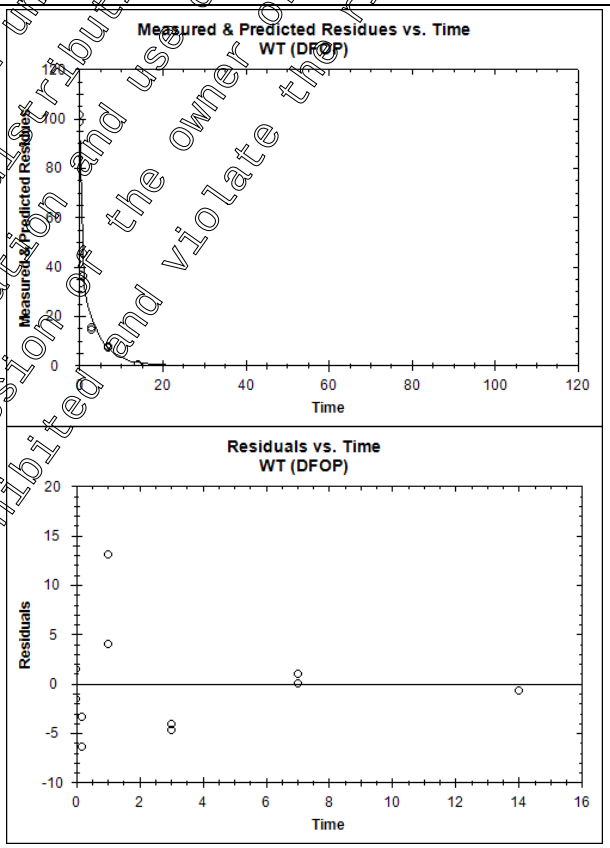
Trial / Best Fit Model	Graphical Representations

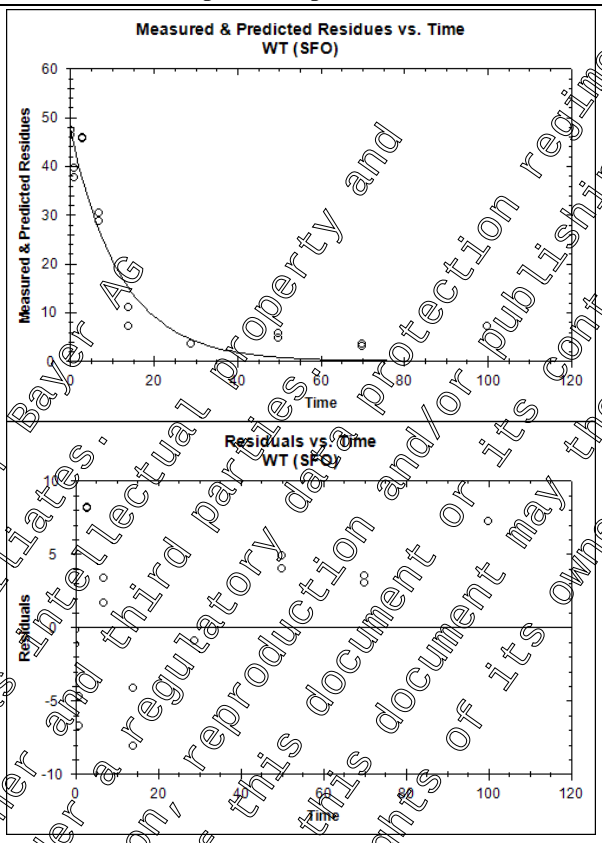
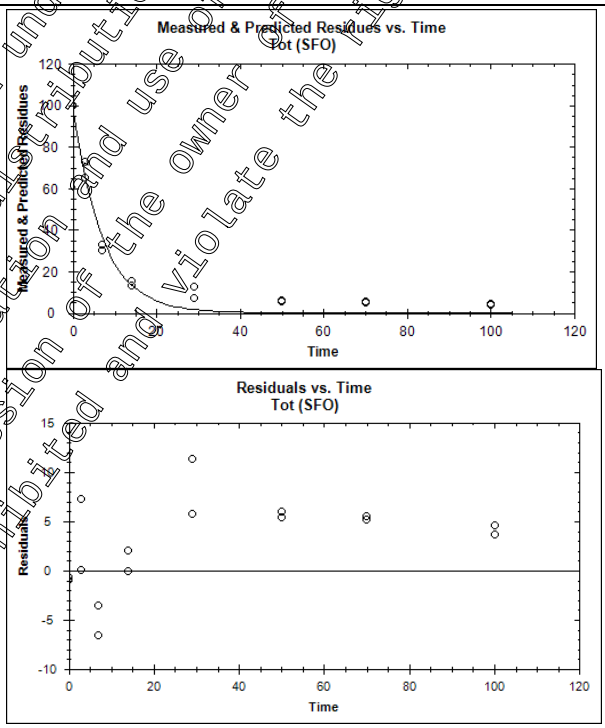
Trial / Best Fit Model	Graphical Representations
[redacted] SFO Modelling endpoint Total system	
[redacted] DFOP Persistence endpoints Total system	

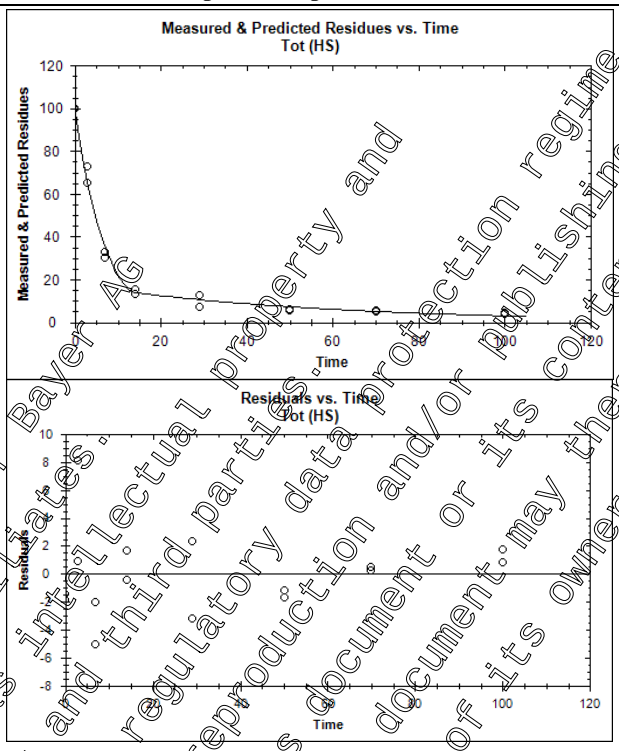
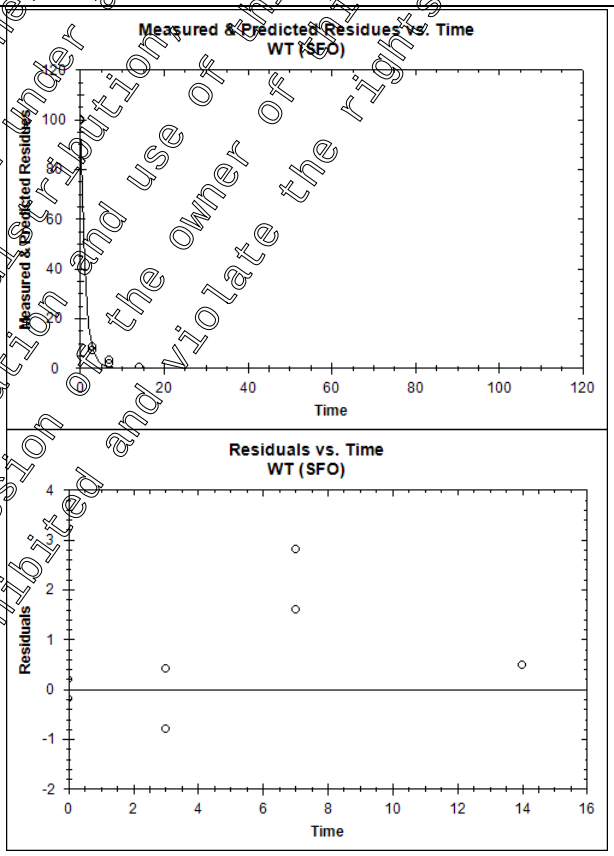
Trial / Best Fit Model	Graphical Representations
[redacted] SFO Modelling endpoint Water	
[redacted] HS Persistence endpoints Water	

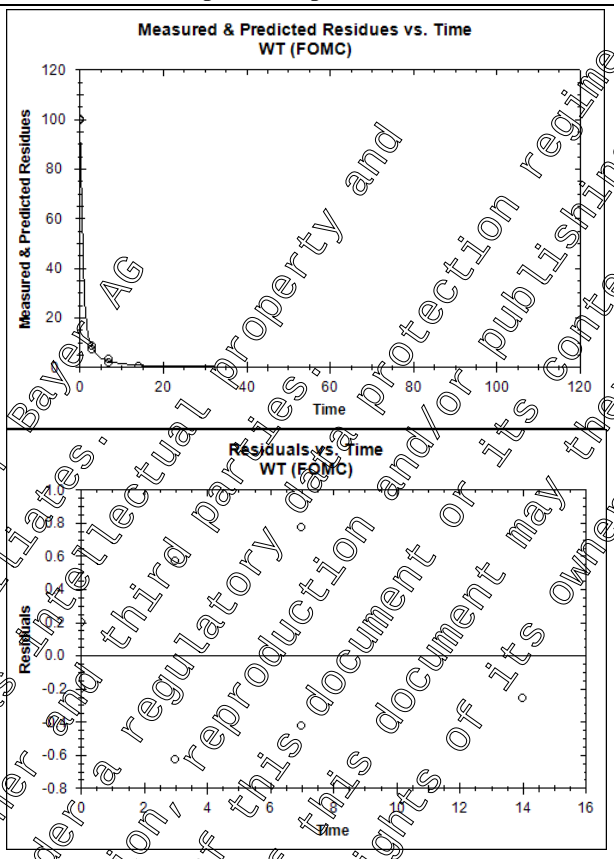
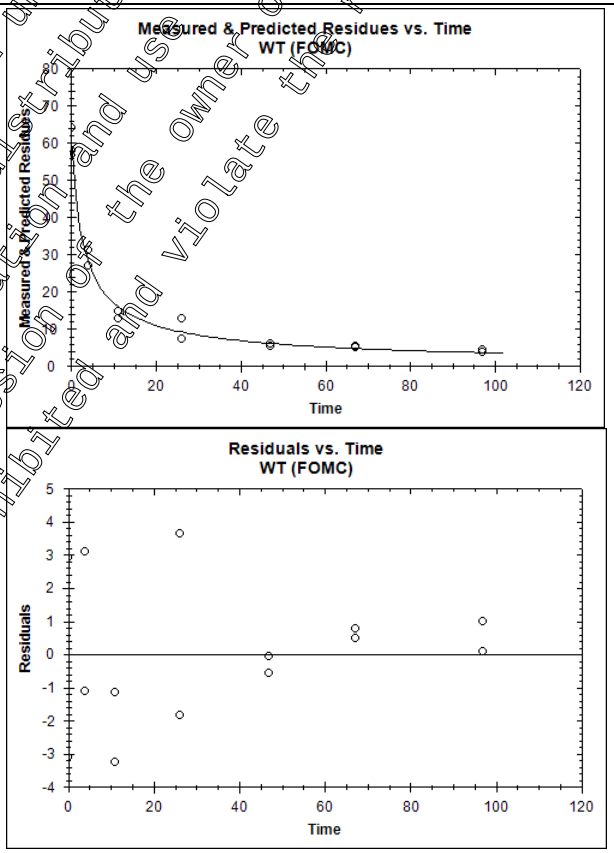
Trial / Best Fit Model	Graphical Representations
[redacted] SFO Modelling endpoint & Persistence endpoints Sediment	
[redacted] HS Modelling endpoint & Persistence endpoints Total system	

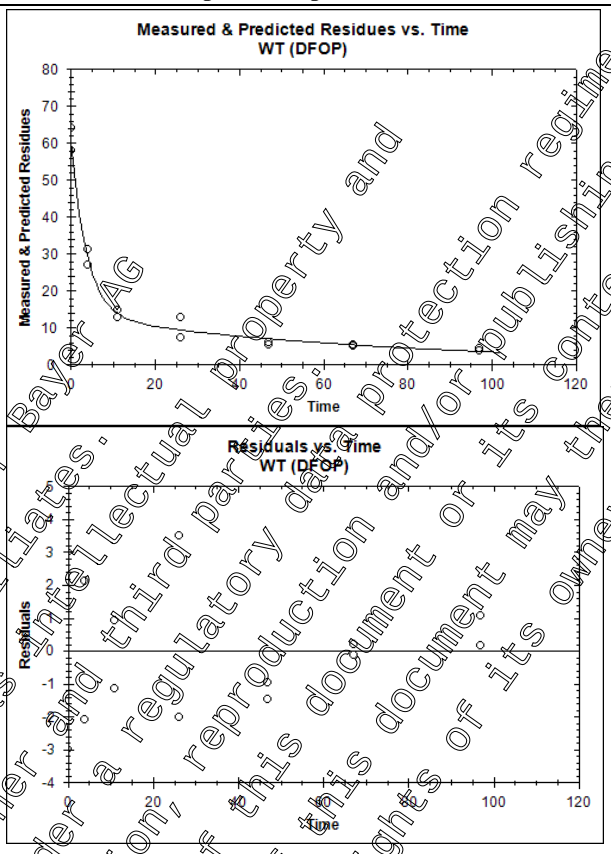
Trial / Best Fit Model	Graphical Representations
[redacted] SFO Modelling endpoint & Persistence endpoints Water	
[redacted] SFO Modelling endpoint & Persistence endpoints Sediment	

Trial / Best Fit Model	Graphical Representations
Anglersee SFO Modelling endpoint & Persistence endpoints Total system	
Anglersee DFOP Modelling endpoint & Persistence endpoints Water	

Trial / Best Fit Model	Graphical Representations
Anglersee SFO Modelling endpoint & Persistence endpoints Sediment	
[redacted] SFO Modelling endpoint Total system	

Trial / Best Fit Model	Graphical Representations
[redacted] HS Persistence endpoint Total system	
[redacted] SFO Modelling endpoint Water	

Trial / Best Fit Model	Graphical Representations
[redacted] FMOC Persistence endpoint Water	
[redacted] FOMC Modelling endpoint Sediment	

Trial / Best Fit Model	Graphical Representations
[redacted] DFOP Persistence endpoints Sediment	

III. CONCLUSION

Modelling and trigger endpoints for aclonifen were calculated with kinetic models according to FOCUS guidance (2006, 2014) in 4 European water sediment systems.

Modelling endpoint DT_{50} values for aclonifen in total water sediment systems ranged from 4.8 days to 43.81 days with a geometric mean of 14.4 days. Best fit trigger endpoint DT_{50} values for aclonifen in total water sediment systems ranged from 4.6 to 22.8 days.

Assessment and conclusion by applicant:

The modelling report was conducted according to FOCUS Degradation Kinetics (2006, 2014) and is considered valid to assess best fit and modelling DT_{50} values for aclonifen in water sediment systems.

Assessment and conclusion by RMS:

CA 7.2.2.4 Irradiated water/sediment study

An irradiated water sediment study is an optional higher tier study which is not required for aclonifen.

CA 7.2.3 Degradation in the saturated zone

Aclonifen is immobile in soils and the risk of groundwater contamination is therefore unlikely. The compound is strongly adsorbed in soils and is retained in the uppermost soil layers.

Additional information is available from a soil study summarised under point KCA 7.1.1.2 (Anaerobic degradation). Treated soil samples were incubated first under aerobic conditions and thereafter converted to anaerobic conditions. During the aerobic phase aclonifen metabolised at a moderate rate (DT_{50} value of 54 -71 days), similar to results obtained in aerobic soil degradation studies. After flooding the soil, aclonifen degraded more rapidly in soil (DT_{50} value of 18-25 days under anaerobic conditions). Anaerobic metabolism of aclonifen led to the formation of a number of unidentified minor degradation products, none of which exceeded 4%, and non-extractable soil residues, which reached a maximum of 93% after 122 days incubation. ~~89% after 91 days and remained at this level until the end of the incubation period.~~

~~CA 7.2.4 Effect of water treatment processes on the nature of residues~~

~~Data Point:~~	~~KCA 7.2.4/01~~
~~Report Author:~~	[redacted]
~~Report Year:~~	~~2019~~
~~Report Title:~~	~~Aclonifen - Effect of water treatment processes on the nature of residues present in groundwater and surface water~~
~~Report No:~~	~~EnSa 19-0595~~
~~Document No:~~	~~M-676096-01-1~~
~~Guideline(s) followed in study:~~	~~–~~
~~Deviations from current test guideline:~~	~~–~~
~~Previous evaluation:~~	
~~GLP/Officially recognised testing facilities:~~	~~No, not conducted under GLP/Officially recognised testing facilities~~
~~Acceptability/Reliability:~~	

~~**Executive Summary**~~

~~An assessment of the potential impact of drinking water treatment, considering both the exposure aspects and transformation chemistry, has been prepared for aclonifen.~~

~~The definition of residue relevant for assessment for aclonifen in groundwater and surface water is parent active substance only. No exposure of groundwater is predicted following application of aclonifen. The worst case assumption for potential exposure of surface water at the field scale for aclonifen would be the regulatory acceptable concentration (RAC) of 1.36 µg/L. However raw water for drinking water purposes will usually be abstracted from dedicated groundwater and surface water sources where attenuation factors from conservative field scale predictions to actual exposure situation at drinking water abstraction points will be significant. Before being subjected to chemical oxidative treatment steps, raw water sourced from a surface water body would typically undergo filtration steps. As aclonifen is a highly adsorptive compound (K_{oc} = 5727, Log P = 4.37), it would be expected that any contact with organic material in these pre-treatment steps would further significantly reduce any aclonifen presence, via simple physical adsorption processes. Consequently it is not expected any~~

residues of the aclonifen would be present at the chemico-oxidative stages of drinking water processing in excess of 0.1 µg/L.

An evaluation of public monitoring data has shown findings of aclonifen are extremely rare with exceedance above the regulatory groundwater trigger of 0.1 µg/L at only 0.007% (12 out of 184651 analyses) and above the surface water Tier 1 RAC SW also at 0.007% (22 out of 311942 analyses).

The diphenylether moiety is not known to raise any concerns for critical transformation reactions with chlorine or ozone based water treatment. Literature searches did not identify any relevant publications on water treatment of aclonifen. One publication observed no reactivity of four diphenylether herbicides with chlorine or ozone. The structure of aclonifen does not match any of the previously known precursors of N-nitrosodimethylamine (NDMA), nor does it include any substructure moieties of concern.

Aclonifen residues is not expected to be presence in raw water abstracted for drinking water production. In the highly unlikely case that residues are present, there are no indications of any risk of forming N-nitrosamine type byproducts upon oxidative water processing from aclonifen residues.

I. MATERIALS AND METHODS

The first step in the assessment was a review of the predicted exposure concentrations at the field level for groundwater and surface water. This was refined by a review of the predicted exposure situation at raw water abstraction points and finally the impact of mechanical, physical, biological steps conducted prior to the eventual chemical steps on the predicted exposure situation at the chemical stages of raw water processing was assessed.

The next step in the assessment was an assessment of the behaviour of aclonifen residues upon chemical-oxidative water treatment. For the production of public drinking water, raw water is sourced from groundwater and/or surface water and is processed for purification and disinfection typically by a sequence of mechanical, physical, biological, and eventually chemical treatment steps. The chemical steps can be summarised as an exposure of pre-cleaned raw water to chemical oxidants, aiming for a transformation or mineralization of unwanted matrix components and micropollutants (e.g. to remove colour, odour, toxicity, and/or to meet other quality parameters), and for the inactivation of microbial life (i.e. control of waterborne diseases). In practice, two major groups of chemical reagents are in use; ozone based treatment and chlorine based treatment. An evaluation of public monitoring data for findings of aclonifen and public literature searches have been conducted to identify any relevant publications on water treatment of aclonifen.

Different classes of compounds have been assayed for their potential to form nitrosodimethylamine (NDMA) upon ozone treatment. Three types of structural moieties appear to have relevance as eventual NDMA precursors which can serve as structure alerts for screening purposes; N,N-dimethylhydrazine (UDMH) derivatives, N,N-dimethylsulfamide (DMS) derivatives and N,N-dimethylaminocarbamate (DMAC) derivatives. The structure of aclonifen was checked for exact matches, matching substructures or homologues that might form UDMH, DMS or DMAC.

II. RESULTS AND DISCUSSION

Predicted exposure concentrations at the field level

The definition of residue relevant for assessment for aclonifen in groundwater and surface water is parent active substance only. The first step was a review of the groundwater and surface water exposure situation at the field scale level for aclonifen based on the PEC_{gw} and PEC_{sw} values predicted for the most critical 'representative use' for EU Annex I inclusion. These values are provided in full in both Documents MCP Section 9.

The predicted environmental concentrations (PEC) for groundwater are presented below for the highest use rate.

Table 7.2.1- 4: FOCUS PEARL, PELMO and MACRO, PECgw results of aclonifen in legumes at 600 g/ha

Crop	Scenario	80th percentile PECgw at 1 m soil depth (µg/L)	
		Aclonifen	
		PEARL	PELMO
Beans (field) Peas (Animal)	[redacted]	<0.001 <0.001 <0.001 <0.001 <0.001 <0.001 <0.001 <0.001	<0.001 <0.001 <0.001 <0.001 <0.001 <0.001 <0.001 <0.001
MACRO	[redacted]	<0.001	

No exposure of groundwater is predicted following application of aclonifen.

The PEC values for surface water for FOCUS Step 4 are presented below for the highest use rate for cereals and peas.

Table 7.2.1- 5: Winter cereals: Single application FOCUS Step 4 PEC_{sw} results for aclonifen, use rate 350 g/ha

PEC_{sw} (µg/L)	Scenario	Step 4 Aclonifen							
Nozzle reduction	Vegetated strip (m)	None	None	None	None	None	10 m	10 m	20 m
	No spray buffer (m)	0 m	5 m	10 m	15 m	20 m	10 m	15 m	20 m
None	D1 Ditch	2.22	0.602	0.319	0.218	0.166	0.319	0.218	0.166
50 %		1.11	0.301	0.159	0.109	0.083	0.159	0.109	0.083
75 %		0.555	0.150	0.080	0.054	0.041	0.080	0.054	0.041
90 %		0.222	0.060	0.032	0.022	0.017	0.032	0.022	0.017
None	D1 Stream	1.95	0.710	0.376	0.257	0.195	0.376	0.257	0.195
50 %		0.972	0.355	0.188	0.128	0.098	0.188	0.128	0.098
75 %		0.485	0.177	0.094	0.064	0.049	0.094	0.064	0.049
90 %		0.194	0.071	0.038	0.026	0.020	0.038	0.026	0.020
None	D2 Ditch	2.23	0.603	0.319	0.218	0.166	0.319	0.218	0.166
50 %		1.11	0.301	0.160	0.109	0.083	0.160	0.109	0.083
75 %		0.556	0.150	0.080	0.054	0.041	0.080	0.054	0.041
90 %		0.222	0.060	0.032	0.022	0.017	0.032	0.022	0.017
None	D2 Stream	1.98	0.723	0.383	0.262	0.199	0.383	0.262	0.199
50 %		0.990	0.361	0.191	0.131	0.099	0.191	0.131	0.099
75 %		0.494	0.180	0.096	0.065	0.050	0.096	0.065	0.050
90 %		0.198	0.072	0.038	0.026	0.020	0.038	0.026	0.020
None	D3 Ditch	2.19	0.594	0.315	0.215	0.163	0.315	0.215	0.163
50 %		1.10	0.297	0.157	0.107	0.082	0.157	0.107	0.082

PEC$_{sw}$ (µg/L)	Scenario	Step 4 Aclonifen							
Nozzle reduction	Vegetated strip (m)	None	None	None	None	None	10 m	10 m	20 m
	No spray buffer (m)	0 m	5 m	10 m	15 m	20 m	10 m	15 m	20 m
75 %		0.547	0.148	0.079	0.054	0.041	0.079	0.054	0.041
90 %		0.219	0.059	0.031	0.021	0.016	0.031	0.021	0.016
None	D4 Pond	0.076	0.065	0.047	0.037	0.031	0.047	0.037	0.031
50 %		0.038	0.033	0.023	0.019	0.016	0.023	0.019	0.016
75 %		0.019	0.016	0.012	0.009	0.008	0.012	0.009	0.008
90 %		0.008	0.007	0.005	0.004	0.004	0.005	0.004	0.004
None	D4 Stream	1.90	0.694	0.368	0.251	0.191	0.368	0.251	0.191
50 %		0.950	0.346	0.184	0.125	0.095	0.184	0.125	0.095
75 %		0.474	0.173	0.092	0.063	0.048	0.092	0.063	0.048
90 %		0.190	0.069	0.037	0.032	0.032	0.037	0.032	0.032
None	D5 Pond	0.076	0.065	0.047	0.037	0.031	0.047	0.037	0.031
50 %		0.038	0.033	0.024	0.019	0.016	0.024	0.019	0.016
75 %		0.019	0.016	0.012	0.009	0.008	0.012	0.009	0.008
90 %		0.008	0.007	0.005	0.004	0.003	0.005	0.004	0.003
None	D5 Stream	2.05	0.748	0.397	0.271	0.206	0.397	0.271	0.206
50 %		1.02	0.374	0.198	0.135	0.103	0.198	0.135	0.103
75 %		0.512	0.187	0.099	0.068	0.051	0.099	0.068	0.051
90 %		0.205	0.075	0.040	0.027	0.021	0.040	0.027	0.021
None	D6 Ditch	2.22	0.600	0.318	0.217	0.165	0.318	0.217	0.165
50 %		1.11	0.300	0.159	0.108	0.083	0.159	0.108	0.083
75 %		0.553	0.150	0.079	0.062	0.062	0.079	0.062	0.062
90 %		0.221	0.062	0.062	0.062	0.062	0.062	0.062	0.062
None	R1 Pond	0.105	0.101	0.095	0.092	0.090	0.048	0.045	0.032
50 %		0.092	0.091	0.088	0.086	0.085	0.040	0.038	0.021
75 %		0.086	0.085	0.084	0.083	0.083	0.036	0.035	0.019
90 %		0.083	0.082	0.082	0.081	0.081	0.034	0.034	0.017
None	R1 Stream	1.44	0.527	0.477	0.477	0.477	0.279	0.214	0.145
50 %		0.722	0.477	0.477	0.477	0.477	0.214	0.214	0.111
75 %		0.477	0.477	0.477	0.477	0.477	0.214	0.214	0.111
90 %		0.477	0.477	0.477	0.477	0.477	0.214	0.214	0.111
None	R3 Stream	2.01	0.732	0.513	0.513	0.513	0.388	0.265	0.201
50 %		1.00	0.513	0.513	0.513	0.513	0.234	0.234	0.123
75 %		0.513	0.513	0.513	0.513	0.513	0.234	0.234	0.123
90 %		0.513	0.513	0.513	0.513	0.513	0.234	0.234	0.123
None	R4 Stream	1.45	0.701	0.701	0.701	0.701	0.316	0.316	0.165
50 %		0.726	0.701	0.701	0.701	0.701	0.316	0.316	0.165
75 %		0.701	0.701	0.701	0.701	0.701	0.316	0.316	0.165

PEC$_{sw}$ (µg/L)	Scenario	Step 4 Aclonifen							
Nozzle reduction	Vegetated strip (m)	None	None	None	None	None	10 m	10 m	20 m
	No spray buffer (m)	0 m	5 m	10 m	15 m	20 m	10m	15 m	20m
90 %		0.701	0.701	0.701	0.701	0.701	0.316	0.206	0.165

Table 7.2.1–6: Peas: Single application FOCUS Step 4 PEC$_{sw}$ results for aclonifen, use rate 600 g/ha

PEC$_{sw}$ (µg/L)	Scenario	Step 4 Aclonifen							
Nozzle reduction	Vegetated strip (m)	None	None	None	None	None	10 m	10 m	20 m
	No spray buffer (m)	0 m	5 m	10 m	15 m	20 m	10m	15 m	20 m
None	D3 Ditch	3.12	1.02	0.541	0.369	0.281	0.541	0.369	0.281
50 %		1.56	0.510	0.270	0.185	0.140	0.270	0.185	0.140
75 %		0.778	0.255	0.135	0.092	0.070	0.135	0.092	0.070
90 %		0.311	0.102	0.054	0.037	0.028	0.054	0.037	0.028
None	D4 Pond	0.425	0.112	0.081	0.064	0.054	0.081	0.064	0.054
50 %		0.063	0.056	0.040	0.032	0.027	0.040	0.032	0.027
75 %		0.031	0.028	0.020	0.016	0.013	0.020	0.016	0.013
90 %		0.013	0.011	0.008	0.007	0.007	0.008	0.007	0.007
None	D4 Stream	2.54	1.07	0.565	0.386	0.294	0.565	0.386	0.294
50 %		1.27	0.533	0.282	0.193	0.147	0.282	0.193	0.147
75 %		0.633	0.266	0.141	0.096	0.073	0.141	0.096	0.073
90 %		0.253	0.106	0.056	0.051	0.051	0.056	0.051	0.051
None	D5 Pond	0.426	0.112	0.081	0.064	0.054	0.081	0.064	0.054
50 %		0.063	0.056	0.040	0.032	0.027	0.040	0.032	0.027
75 %		0.031	0.028	0.020	0.016	0.014	0.020	0.016	0.014
90 %		0.013	0.011	0.008	0.006	0.005	0.008	0.006	0.005
None	D5 Stream	2.60	1.09	0.578	0.395	0.300	0.578	0.395	0.300
50 %		1.30	0.545	0.289	0.197	0.150	0.289	0.197	0.150
75 %		0.648	0.272	0.144	0.099	0.075	0.144	0.099	0.075
90 %		0.259	0.109	0.058	0.039	0.030	0.058	0.039	0.030
None	D6 Ditch	3.12	1.02	0.541	0.370	0.281	0.541	0.370	0.281
50 %		1.56	0.510	0.270	0.185	0.140	0.270	0.185	0.140
75 %		0.779	0.255	0.135	0.092	0.070	0.135	0.092	0.070
90 %		0.311	0.102	0.054	0.043	0.043	0.054	0.043	0.043
None	R1 Pond	0.133	0.119	0.088	0.079	0.079	0.083	0.067	0.055
50 %		0.079	0.079	0.079	0.079	0.079	0.043	0.035	0.028
75 %		0.079	0.079	0.079	0.079	0.079	0.032	0.032	0.016

PECsw (µg/L)	Scenario	Step 4 Aclonifen							
Nozzle reduction	Vegetated strip (m)	None	None	None	None	None	10 m	10 m	20 m
	No spray buffer (m)	0 m	5 m	10 m	15 m	20 m	10 m	15 m	20 m
90 %		0.079	0.079	0.079	0.079	0.079	0.032	0.032	0.016
None	R1 Stream	2.16	0.907	0.707	0.707	0.707	0.481	0.328	0.250
50 %		1.08	0.707	0.707	0.707	0.707	0.318	0.318	0.166
75 %		0.707	0.707	0.707	0.707	0.707	0.318	0.318	0.166
90 %		0.707	0.707	0.707	0.707	0.707	0.318	0.318	0.166
None	R2 Stream	2.87	1.21	0.639	0.436	0.332	0.639	0.436	0.332
50 %		1.43	0.602	0.319	0.218	0.186	0.319	0.218	0.166
75 %		0.715	0.301	0.186	0.186	0.186	0.159	0.109	0.083
90 %		0.286	0.186	0.186	0.186	0.186	0.085	0.085	0.044
None	R3 Stream	3.05	1.28	0.680	0.502	0.502	0.680	0.464	0.353
50 %		1.52	0.641	0.502	0.502	0.502	0.340	0.232	0.176
75 %		0.761	0.502	0.502	0.502	0.502	0.229	0.229	0.120
90 %		0.502	0.502	0.502	0.502	0.502	0.229	0.229	0.120
None	R4 Stream	2.15	1.16	1.16	1.16	1.16	0.516	0.516	0.268
50 %		1.16	1.16	1.16	1.16	1.16	0.516	0.516	0.268
75 %		1.16	1.16	1.16	1.16	1.16	0.516	0.516	0.268
90 %		1.16	1.16	1.16	1.16	1.16	0.516	0.516	0.268

To reflect a situation *"consequent on application consistent with good plant protection practice and having regard to realistic conditions of use"* (1107/2009, Article 4(3b)), it should also be considered that registration of products containing aclonifen will establish an ecotoxicological assessment for the protection of aquatic habitats. Where needed, this assessment will impose measures for mitigating the exposure at the field scale to an upper bound value defined as the regulatory acceptable concentration (RAC). In practice, therefore the worst case assumption for potential exposure of surface water at the field scale for aclonifen would be the RAC of 1.36 µg/L.

Predicted exposure situation at raw water abstraction points

The second step was a review of the exposure situation expected at raw water abstraction points of relevance for technical drinking water production. Raw water for drinking water purposes will usually be abstracted from protected groundwater and surface water sources that are dedicated for this use. The attenuation factor from conservative field scale predictions to actual exposure situation at drinking water abstraction points will be significant. Within recent EU review processes explicit dilution factors have been proposed and accepted by Rapporteur Member States and EFSA within an overall range from 1:10 - <1:1000000 for various active substances.

The maximum edge of field water body exposure situation for aclonifen (1.36 µg/L) indicates that attenuation by only factor 15 upon the PECsw -> PECraw transfer would already be sufficient not exceed the parametric value of 0.1 µg/L at the raw water abstraction point. Aclonifen exposure levels which might be expected at raw water abstraction points would therefore not trigger generic concerns for water treatment, would remain well within the range previously considered acceptable for other components, and would be by far below the limits specified for surrogate disinfection by products (DBP) in the drinking water regulation.

Predicted exposure situation at the chemical stages of raw water processing

The exposure assessment identified surface water as the predominant, though overall low, exposure route of potential relevance for the assessment of water treatment. Before being subjected to chemical-oxidative treatment steps, raw water sourced from a surface water body would typically undergo either a bank filtration process, pass a sand filtration step, or other filtering systems for pre-cleaning, potentially followed by flocculation. As aclonifen is a highly adsorptive compound (K_{oc} = 5727, Log P = 4.37), it would be expected that any contact with organic material in these pre-treatment steps would further significantly reduce any aclonifen presence, via simple physical adsorption processes. It is not expected any residues of the aclonifen would be present at the chemico-oxidative stages of drinking water processing in excess of 0.1 µg/L.

Environmental monitoring information

An evaluation of public monitoring data has shown findings of aclonifen in public databases are not common (see KCA 7.5/01, Luks, Graff & May, 2019). The results from the groundwater monitoring data search indicates that aclonifen concentrations above the regulatory trigger of 0.1 µg/L are exceedingly rare (12 samples; 0.007% of 184651 analyses) and at most 10.4 µg/L. The results from the surface water monitoring data search indicate that aclonifen concentrations above the Tier 1 RAC-SW (1.36 µg/L) are exceedingly rare (22 samples, 0.007% of 311942 analyses) and are at most 65 µg/L.

Literature information related to Aclonifen behaviour upon water treatment

A public literature search was conducted on aclonifen (see Document MCA Section 9) which did not identify any relevant publications on water treatment of aclonifen. A literature search conducted for diphenylether herbicides located one publication (Okumura, 1992) in which no reactivity with chlorine or ozone was observed for four diphenylether herbicides. It can therefore be concluded that the diphenylether moiety, the characteristic core structure of aclonifen's herbicide class, is not known to raise any particular concern for critical transformation reactions under the conditions of chlorine and/or ozone-based water treatment.

Evaluation for possible risk of N-nitrosamine generation

The structure of aclonifen does not match any of the previously known precursors of N-nitrosodimethylamine (NDMA). Aclonifen does not include any substructure moieties that would be expected prone to cleavage forming N,N-dimethylhydrazine ('unsymmetrical dimethylhydrazine', UDMH), N,N-dimethylsulfamide (DMS) or N,N-dimethyl-O-aminocarbamate (DMAC). The parent molecule does not contain a dimethyl substituted amine moiety. Moreover, the aclonifen structure does not include any homologues or analogues to the UDMH, DMS, DMAC moieties. The two nitrogen atoms present are not interconnected by a potential leaving group. No environmental transformation reactions are known that might convert aclonifen to form one of the known alerting motifs.

III. CONCLUSION

In conclusion, it is not to be expected that aclonifen residues, even in the highly unlikely case of a presence in raw water abstracted for drinking water production, would bear a risk of forming N-nitrosamine type byproducts upon oxidative water processing.

Assessment and conclusion by applicant: The position paper is considered valid to aid assessment of the potential impact of drinking water treatment.

Assessment and conclusion by RMS:

CA 7.3 Fate and behaviour in air

The following studies were evaluated during the previous EU review and are still considered as reliable to assess the fate and behaviour of aclonifen in air.

Report reference	Author, Year	Comment
KCA 7.3/01 M-160441-01-1	[redacted] & [redacted], M., 1990	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.3/02 M-251228-01-1	[redacted]; [redacted], 2005	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.3/03 M-160423-01-1	[redacted]; [redacted], 1990	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.3.1/01 M-198892-01-1	[redacted], 2000	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.
KCA 7.3.2/01 M-175860-01-1	[redacted] M., [redacted], N. & [redacted], G., 1993	Submitted for first approval of aclonifen, 2006. Reviewed under UP. Considered valid and acceptable.

The vapour pressure of aclonifen is 1.6×10^{-5} Pa at 20°C with a Henry's Law constant of 3.03×10^{-3} Pa m^3 mol^{-1} at 20°C and a water solubility of 1.4 mg/L, thus the compound would not be expected to be found in any significant concentration in air.

Data Point:	KCA 7.3/01
Report Author:	[redacted]
Report Year:	1990
Report Title:	Aclonifen - Vapour pressure curve
Report No:	R001391
Document No:	M-160441-01-1
Guideline(s) followed in study:	OECD: 104, (may 1981); USEPA (=EPA): D, 63-9
Deviations from current test guideline:	Current guideline: Regulation (EC) No 1107/2009, Commission Regulation (EU) 283/2013 No deviation Current guideline for MoA: OECD 104 No deviation Current guidance for MoA: SANCO/3030/99 rev.5 Precision data not reported in full detail
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The vapour pressure of aclonifen (purity 993 g/kg) was determined using a gas saturation method. The vapour pressure of aclonifen was measured at three temperatures as follows:

At 20 °C, vapour pressure = 1.6 x 10^{-5} Pa

At 35 °C, vapour pressure = 1.4 x 10^{-4} Pa

At 50 °C, vapour pressure = 1.1 x 10^{-3} Pa

Data Point:	KCA 7.3/02
Report Author:	[redacted]
Report Year:	2005
Report Title:	Henry's Law Constant of Aclonifen Code: AE F068300
Report No:	C048312
Document No:	M-251228-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Current guideline: Regulation (EC) No 1107/2009, Commission Regulation (EU) 283/2013 No Deviation
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS, DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The Henry's Law constant was calculated from vapour pressure and water solubility. Using these values, the Henry's law constant K was found to be

K = 3.03 x 10^{-3} Pa • m³ • mol⁻¹

Data Point:	KCA 7.3/03
Report Author:	[redacted]; [redacted]
Report Year:	1990
Report Title:	Aclonifen - Water solubility at 20 degrees Celsius and at pH's 5, 7 and 9
Report No:	R001379
Document No:	M-160423-01-1
Guideline(s) followed in study:	OECD: 105, (May 1981)
Deviations from current test guideline:	Current guideline: Regulation (EC) No 1107/2009, Commission Regulation (EU) 283/2013 No deviation Current guideline for MoA: OECD 105 No deviation Current guidance for MoA: SANCO/3030/99 rev.5 Precision data not reported in full detail
Previous evaluation:	No, not previously submitted
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

The solubility of aclonifen in water (purity 997 g/kg) was determined at 20°C and at pH's 5, 7 and 9, according to OECD guidelines. The column elution method was used for generating saturated aqueous solutions which were analyzed using HPLC. The solubility was equal to 1.40 mg/L and was not pH dependent.

The potential persistence of the compound in air has been estimated (see KCA 7.3.1/01) according to the models developed by Atkinson.

Finally, a soil volatility study (see KCA 7.3.2/01) conducted in a laboratory volatilisation chamber has confirmed aclonifen is not volatile, with less than 2.5% of applied radioactivity detected in volatile traps after 24 hours.

CA 7.3.1 Route and rate of degradation in air

Data Point:	KCA 7.3.1/01
Report Author:	[redacted]
Report Year:	2000
Report Title:	Estimation of the reaction with photochemically produced hydroxyl radicals in the atmosphere Code. AE F068300
Report No:	C010366
Document No:	M-198892-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	Not applicable
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive Summary

The half-life of aclonifen in the atmosphere was calculated as being in the range of 0.84 to 1.26 days dependent upon the mean aerial OH concentration chosen.

I. MATERIALS AND METHODS

In the troposphere there are three important photochemical transformation processes that may contribute to the degradation of a chemical. These are direct phototransformation, indirect photoreaction with hydroxyl radicals and oxidation with ozone. The rate constant for the atmospheric reaction between photochemically produced hydroxyl radicals and aclonifen was estimated using the computer programme AOPWIN (version 1.88, January 1999). The estimated rate constant enabled the calculation of the atmospheric half-life of aclonifen based upon average atmospheric concentration of hydroxyl radicals. Aclonifen contains no olefin and acetylene groups, which are necessary for reaction with ozone and thus this reaction was not assessed.

The programme estimates an overall OH-radical reaction rate constant by summing the individual OH-radical reaction pathways, which can be regarded as operating independently. The overall reaction rate constant of aclonifen was based on only one type of reaction, that is, the addition of OH to the aromatic rings.

II. RESULTS AND DISCUSSION

The overall OH-radical reaction rate constant resulting from addition of OH radicals to the aromatic rings in aclonifen was calculated as:

k_{OH} = 12.7360 x 10^{-12} cm^3 $molecule^{-1}$ sec^{-1}

The resultant half life in the atmosphere was then calculated based upon two separate scenarios with differing assumptions of mean hydroxyl radical concentrations. These were stated as being standard values appropriate to assessments for Europe and USA.

Scenario	EU	USA
OH concentration [10^6 radicals cm^{-3}]	0.5	1.5

Time frame [hours day^{-1}]	24	12
OH rate constant [cm^3 molecule^{-1} sec^{-1}]	12.7360 x 10^{-12}	
Half life [hours]	30.234	10.078
Half life [days]	1.26	0.84

III. CONCLUSION

The half-life of aclonifen in the atmosphere was calculated as being in the range of 0.84 to 1.26 days, dependent upon the mean aerial OH concentration chosen for the calculation. It can be concluded that aclonifen will be readily degraded in the air due to its fast reaction with photolytically generated hydroxyl radicals.

Assessment and conclusion by applicant:

The estimate of the half-life of aclonifen in the upper atmosphere complies with current guidance.

Assessment and conclusion by RMS:

CA 7.3.2 Transport via air

The vapour pressure of aclonifen was 1.6 x 10^{-5} Pa at 20 °C which just above the trigger for volatilisation from plant surfaces but below the trigger for volatilisation from soil surfaces.

Data Point:	KCA 7.3.2.01
Report Author:	[redacted]
Report Year:	1993
Report Title:	Soil surface volatility study of aclonifen formulated as Bandur - Suspension concentrate (SC containing 600 g x l**-1 Aclonifen).
Report No:	R007912
Document No:	M-175860-01-1
Guideline(s) followed in study:	BBA: IV, 6-1, (1990)
Deviations from current test guideline:	Not applicable
Previous evaluation:	yes, evaluated and accepted Source: Study list relied upon, December 2011 (RMS: DE)
GLP/Officially recognised testing facilities:	Yes, conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	Yes

Executive summary:

The volatilisation of [^{14}C]-aclonifen formulated as BANDUR® from soil surface was investigated in a volatilisation chamber. [^{14}C]-aclonifen at a specific activity of 2.241 MBq/mg and radiochemical

purity of > 98%, was mixed with non radiolabelled formulation BANDUR. BANDUR is a SC formulation containing aclonifen (600 g/L). The radioactive formulation was applied to the surface of a sandy soil, incubated at 60% maximum water holding capacity, at a rate equivalent to 3 kg/ha.

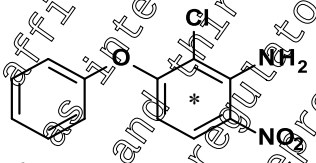
Moist air was passed over the soil surface at a speed of 1.0 m/s and passed through a series of traps, consisting of Amberlite XAD2 resin and silica wool, to collect any volatile material. Air and soil temperatures were maintained at a constant 20°C by circulation of water around the chamber. Volatile trap samples were taken after 1, 3, 6 and 24 hours. After 24 hours soil samples were taken for analysis.

The total recovery of applied radioactivity was 104.77 %. Less than 2.5% of applied radioactivity was detected in the volatile traps. 100.26% of the applied radioactivity was extracted from the soil of which the majority (>98%) was confirmed as unchanged parent by TLC analysis. Only 1.81% of applied radioactivity remained unextracted from soil. Volatilisation of aclonifen from soil surfaces was very low level.

I. MATERIALS AND METHODS

A. MATERIALS

1. **Test Material** [U-^{14}C-aniline]-aclonifen



* indicates position of ^{14}C radiolabel

Chemical name (IUPAC): 2-chloro-6-nitro-3-phenoxyaniline

Specific radioactivity: 2.241 MBq/mg

Radiochemical Purity: 98%

CA registry number: 74070-46-5

Stability of test compound: Stable, determined within study

2. **Soil** The soil used was a German standard soil, [redacted] 2.1, classified as a sand. Specific details are shown below.

Table 7.3.2- 1: Physico-chemical characteristics of the soil used for volatility study

Characteristic / Code	Units	[redacted] 2.1
Origin	Country	Germany
Location	City or Township	[redacted]
Particle Size Analysis		
Total Sand (0.063 - 2.0 mm)	%	87.4
Silt (0.002 - 0.063 mm)	%	9.1
Clay (<0.002 mm)	%	3.5
Textural Class	DIN	Sand
pH		5.7
Organic Carbon	%	0.7
Cation Exchange Capacity	mval/100g	4.9
Maximum Water Holding Capacity	%	31.9
Soil Moisture During Incubation	%	19.14
Total Nitrogen	%	0.06
Bulk Density	g/ 1000 ml	1365

B. STUDY DESIGN AND METHODS

1. In-life dates:

December 1992 to January 1993

2. Experimental design:

[U-^{14}C-aniline]-aclonifen at a specific activity of 2.241 MBq/mg and radiochemical purity of > 98%, was mixed with non-radiolabelled formulation BANDUR. BANDUR is a SC formulation containing aclonifen (600 g/L). The radioactive formulation was applied to the surface of the soil, incubated at 60% maximum water holding capacity, at a rate equivalent to 3 kg/ha. Moist air was passed over the soil surface at a speed of 1.0 m/s and passed through a series of traps, consisting of Amberlite XAD2 resin and silica wool, to collect any volatile material. Air and soil temperatures were maintained at a constant 20°C by circulation of water around the chamber. Volatile trap samples were taken after 1, 3, 6 and 24 hours. After 24 hours soil samples were taken for analysis.

The resin and silica wool from the volatile traps were extracted with acetonitrile and the volatilisation chamber rinsed with acetone. Soil samples were sequentially extracted with acetonitrile and methanol. Radioactivity in the extracts was quantified by liquid scintillation counting (LSC) and the soil extracts analysed by thin layer chromatography (TLC). Any radioactivity remaining in the trap material and soil was quantified by combustion followed by LSC.

II. RESULTS AND DISCUSSION

The total recovery of applied radioactivity was 104.77 % (Table 7.3.2- 2). Less than 2.5% of applied radioactivity was detected in the volatile traps. 100.26% of the applied radioactivity was extracted from the soil of which the majority (>98%) was confirmed as unchanged parent by TLC analysis. Only 1.81% of applied radioactivity remained unextracted from soil.

Table 7.3.2- 2: Distribution of radioactivity after volatilisation of aclonifen from soil surface

% of applied radioactivity	
Volatile traps	1 hour: 0.13 2 hours: 0.31 3 hours: 0.66 18 hours: 2.00 24 hours: 2.40
Soil extractables	100.26
Unextractable residues	1.81
Chamber wash	0.29
Recovery	104.77

III. CONCLUSION

Volatilisation of aclonifen from soil surfaces was very low level. Less than 2.5 % of applied radioactivity was found in volatile traps after 24 hours. Most of the applied radioactivity remained in the soil.

Assessment and conclusion by applicant:

The soil volatilisation study was conducted according to BBA IV, 6-1, (1990) and is considered acceptable.

Assessment and conclusion by RMS:

CA 7.3.3 Local and global effects

Being a new potential requirement this has not previously been evaluated within the process for Annex I renewal.

The potential for local effects from use of aclonifen is considered in risk assessments performed following its use under field conditions in particular by considering factors like spray drift. The combination of exposure assessments with potential effects measured in soil and surface water do thus cover the environmental compartments of interest. In contrast and since there is no aerial application envisaged, air is not a compartment regarded to be major compartment of potential for aclonifen occurrence following its intended use in the field.

The setting of global effects like contributions to global warming potential (GWP), ozone depleting potential (OPD), photochemical ozone creation potential (POCP) would require a high probability for the molecule assessed to evaporate and thus occur in the gas phase. This probability can be expressed by the volatility in terms of the vapour pressure (and the Henry constant). The very low potential of aclonifen residues to occur in the atmosphere has been addressed before under CA 7.3.1 and 7.3.2.

Any accumulation in the troposphere would require high volumes of active substance applied and a significant volatility combined with persistence in the gas phase. An acidification potential (AP) would require the generation of acidifying gases like sulfur dioxide or nitrogen oxides in a free form. An eutrophication potential (EP) would require the generation of ammonia or phosphorous compounds acting as nutrients.

Assessment of criteria to be considered a POP (Persistent Organic Pollutant), PBT (Persistent, Bioaccumulative and Toxic) or vPvB (very persistent and very bioaccumulative) substance: Summary of POP, PBT and vPvB Evaluation for Aclonifen

It is considered that aclonifen does not meet any of the necessary screening criteria to be considered a POP (Persistent Organic Pollutant). It does not meet the POP criteria for persistence in water, soil or sediment, nor the bioaccumulation criteria. The DT_{50} in air indicates that potential for long-range transport in air is low. It is considered that aclonifen does not meet all the necessary screening criteria to be classed as a PBT (Persistent, Bioaccumulative and Toxic) compound. It does not meet the PBT criteria for persistence in water, soil or sediment, nor the bioaccumulation criteria. It is considered that aclonifen does not meet any of the necessary screening criteria to be classed as a vPvB (very Persistent, very Bioaccumulative) compound.

In summary, aclonifen does not meet the criteria to be either a POP, PBT nor vPvB. It is not a candidate for substitution as it meets only one of the PBT criteria.

Detailed Assessment of POP, PBT and vPvB classification

Annex II, section 7 of Regulation 1107/2009 states that substances deemed to meet the POP, PBT or vPvB criteria cannot be approved. Also, if an active substance meets two out of three PBT criteria, it will be a 'Candidate for Substitution'. The potential for aclonifen to meet the criteria to be considered as a POP, PBT or vPvB substance is shown below.

POP (Persistent Organic Pollutant)

A POP is defined as a chemical which is extremely stable or persistent in the environment; will bioaccumulate in organisms or the food chain; is toxic to humans or animals and has potential to be transported in the environment over long distances far from the place of release. A substance that fulfils all three of the criteria below is a POP.

Criteria for Classification of a Compound as a POP

Criterion	Definition	Aclonifen Data	Criteria Met?
Persistence	$DT_{50\ water}$ > 2 months	$DegT_{50}$ (water sediment, water phase) = 6.8 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies at 20 °C). $DegT_{50}$ (water sediment, water phase) = 14.3 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies normalized to 12 °C).	No
	$DT_{50\ soil}$ > 6 months	Laboratory studies DT_{50} (soil) = 89.8 days, n=10 (Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from laboratory studies at 20 °C). Field studies DT_{50} (soil) = 89.3 days, n =6 (Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from field studies un-normalised).	

Criterion	Definition	Aclonifen Data	Criteria Met?
	$DT_{50\ sediment}$ > 6 months	$DegT_{50}$ (water sediment, sediment) = 13.8 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies at 20 °C). $DegT_{50}$ (water sediment, sediment) = 29.4 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies normalized to 12 °C. $DegT_{50}$ (water sediment, total system) = 17.8 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies at 20 °C). $DegT_{50}$ (water sediment, total system) = 37.8 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies normalized to 12 °C.	
Bioaccumulation	BCF or BAF > 5000 or in absence log K_{ow} > 5 or evidence that the substance presents other reasons for concern, such as high bioaccumulation in other non-target species, high toxicity or ecotoxicity	The BCF of aclonifen is 1349 L/kg (growth-corrected & lipid normalized bioconcentration factor).	No
Potential for Long-Range Transport (LRT)	Monitoring data showing that long range transport (LRT) may have occurred via air, water or migrating species or fate properties or modelling demonstration LRT or DT_{50} (air) > 2 days for a chemical migrating through the air.	DT_{50} (air) = 1.2 days	No

Aclonifen does not fulfil any of the criteria for classification as a POP.

PBT (Persistent, Bioaccumulative, Toxic)

A PBT is defined as a chemical which is extremely stable or persistent in the environment; will bioaccumulate in organisms or the food chain and is toxic to humans or animals. A substance that fulfils all three of the criteria below is a PBT.

Criteria for Classification of a Compound as a PBT

Criterion	Definition	Aclonifen Data	Criteria Met?
Persistence	The half-life in marine water is higher than 60 days The half-life in fresh or estuarine water is higher than 40 days	No data in marine water available. $DegT_{50}$ (water sediment, water phase) = 6.8 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies at 20 °C). $DegT_{50}$ (water sediment, water phase) = 14.3 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies normalized to 12 °C).	No

	The half-life in marine sediment is higher than 180 days The half-life in fresh or estuarine water sediment is higher than 120 days, or	No data in marine sediment or available $DegT_{50}$ (water sediment, sediment) = 13.8 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies at 20 °C). $DegT_{50}$ (water sediment, sediment) = 29.4 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies normalized to 12 °C). $DegT_{50}$ (water sediment, total system) = 17.8 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies at 20 °C). $DegT_{50}$ (water sediment, total system) = 37.8 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies normalized to 12 °C).	
	The half-life in soil is higher than 120 days Assessment of persistency in the environment shall be based on available half-life data collected under appropriate conditions, which shall be described by the applicant	Laboratory studies DT_{50} (soil) = 89.8 days, n=10 (Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from laboratory studies at 20 °C). Field studies DT_{50} (soil) = 89.3 days, n =6 (Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from field studies un-normalised).	
Bioaccumulation	BCF or BAF > 5000 or in absence log K_{ow} > 5 or evidence that the substance presents other reasons for concern, such as high bioaccumulation in other non-target species, high toxicity or ecotoxicity.	The BCF of aclonifen is 1349 L/kg (growth-corrected & lipid normalized bioconcentration factor).	No

Toxicity	The long-term NOEC for aquatic organisms is < 0.01 mg/L. The substance is classified as carcinogenic (category 1A or 1B), mutagenic (category 1A or 1B), or toxic for reproduction (category 1A, 1B or 2) pursuant to Regulation (EC) No 1272/2008 There is other evidence of chronic toxicity, as identified by the classifications STOT RE 1 or STOT RE 2 pursuant to Regulation (EC) No 1272/2008.	Chronic aquatic toxicity NOEC values range from 0.0000811 to 0.106 mg/L and therefore satisfy the criteria for aclonifen being classified as T. Aclonifen is not classified as carcinogenic (category 1A or 1B), mutagenic (category 1A or 1B), or toxic for reproduction (category 1A, 1B or 2) pursuant to Regulation (EC) No 1272/2008. No evidence of chronic toxicity, which would require classifications as STOT RE 1 or STOT RE 2 pursuant to Regulation (EC) No 1272/2008	Yes

Aclonifen only fulfils one of the criteria for classification as a PBT.

vPvB (very Persistent, very Bioaccumulative)

A vPvB is defined as a chemical which is extremely stable or persistent in the environment and will bioaccumulate in organisms or the food chain. A substance that fulfils both of the criteria below is a vPvB.

Criteria for Classification of a Compound as a vPvB

Criterion	Definition	Aclonifen Data	Criteria Met?
Persistence	The half-life in marine, fresh or estuarine water is higher than 60 days	$DegT_{50}$ (water sediment, water phase) = 6.8 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies at 20 °C). $DegT_{50}$ (water sediment, water phase) = 14.4 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from water sediment studies normalized to 12 °C).	No
	The half-life in marine, fresh or estuarine sediment is higher than	$DegT_{50}$ (water sediment, sediment) = 13.8 days, n=4 Geometric mean of SFO DT_{50} or pseudo SFO (DT_{90}/3.32) values from	

Criterion	Definition	Aclonifen Data	Criteria Met?
		water sediment studies at 20 °C). DegT$_{50}$ (water sediment, sediment) = 29.4 days, n=4 Geometric mean of SFO DT$_{50}$ or pseudo SFO (DT$_{90}$/3.32) values from water sediment studies normalized to 12 °C). DegT$_{50}$ (water sediment, total system) = 17.8 days, n=4 Geometric mean of SFO DT$_{50}$ or pseudo SFO (DT$_{90}$/3.32) values from water sediment studies at 20 °C). DegT$_{50}$ (water sediment, total system) = 37.8 days, n=4 Geometric mean of SFO DT$_{50}$ or pseudo SFO (DT$_{90}$/3.32) values from water sediment studies normalized to 12 °C).	
	The half-life in soil is higher than 180 days	Laboratory studies DT$_{50}$ (soil) = 89.8 days, n=10, (Geometric mean of SFO DT$_{50}$ or pseudo SFO (DT$_{90}$/3.32) values from laboratory studies at 20 °C). Field studies DT$_{50}$ (soil) = 89.3 days, n=6 (Geometric mean of SFO DT$_{50}$ or pseudo SFO (DT$_{90}$/3.32) values from field studies un-normalised).	
Bioaccumulation	BCF or BAF > 5000 or in absence log K$_{ow}$ > 5 or evidence that the substance presents other reasons for concern such as high bioaccumulation in other non-target species, high toxicity or ecotoxicity.	The BCF of aclonifen is 1349 L/kg (growth-corrected & lipid normalized bioconcentration factor).	No

Aclonifen does not fulfil any of the criteria for classification as vPvB.

The following statement provides the reasoning behind the selection of aclonifen endpoints for comparison to the persistence criteria for POP, PBT and vPvB compounds. The persistence criteria for the aquatic environment considered laboratory aerobic mineralisation studies and water sediment studies conducted with aclonifen, while those for soil considered laboratory aerobic soil degradation studies and terrestrial field dissipation studies. The results of a simplified kinetic model used to derive true degradation rates for aclonifen for both the water and sediment phases ($DegT_{50water}$ and $DegT_{50sediment}$) are provided.

Data Point:	KCA 7.3.3/01
Report Author:	[redacted]; [redacted]
Report Year:	2020
Report Title:	Statement on the persistence criteria for aclonifen
Report No:	VC/19/025I
Document No:	M-676480-01-1
Guideline(s) followed in study:	None
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Assessment and conclusion by applicant:

This statement provides an explanation for the selection of aclonifen endpoints for comparison to the persistence criteria for POP, PBT and vPvB compounds.

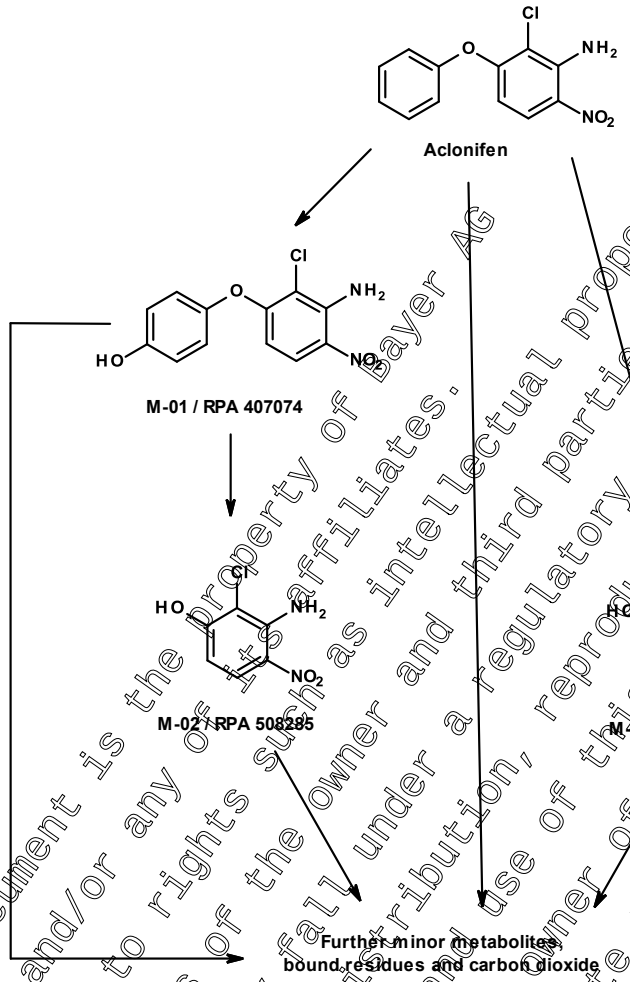
Assessment and conclusion by RMS:

CA 7.4 Definition of the residue

The overall fate of aclonifen in the environment has been extensively studied, with the metabolic pathway shown in Figure 7.3-1 being established.

In soil and water sediment systems no significant metabolites were formed. All metabolites were < 10%, and where they exceeded 5% it was for one timepoint only and they declined thereafter. Two major metabolites (≥ 10%) were formed in aerobic mineralization studies, M-01 and M-02.

Figure 7.4- 1: Metabolic pathway for aclonifen in the environment



Relevant residue in soil

The route and rate of degradation of aclonifen has been investigated in a series of laboratory studies under both aerobic and anaerobic conditions. The potential effect of sunlight upon degradation in soil has also been studied. During the course of these studies, no metabolites of aclonifen have been detected in soil at levels exceeding 5%. Consequently, it is recommended that the relevant residue in soil be defined as the parent active substance, aclonifen.

Relevant residue in water

Groundwater

Based on the FOCUS groundwater calculations, it can be concluded that even under the multiple worst-case conditions inherent to the scenarios, the use of aclonifen in Europe poses no risk to groundwater at 1 m depth with respect to use in pea and wheat for all scenarios.

Surface Water and Sediment

No major metabolites have been detected in laboratory hydrolysis and aqueous photolysis studies.

The route and rate of degradation of aclonifen has been investigated in aquatic water sediment studies. During the course of these studies, no metabolites of aclonifen have been detected in water or sediment at levels exceeding 5% at more than one timepoint.

Based upon the above information, it is recommended that aclonifen be defined as the relevant residue in water and sediment.

Relevant Residue in Air

It is recommended that aclonifen be defined as the relevant residue in air.

CA 7.4.1 Definition of the residue for risk assessment

Soil: Aclonifen

Groundwater: Aclonifen

Surface water: Aclonifen

Sediment: Aclonifen

Air: Aclonifen

CA 7.4.2 Definition of the residue for monitoring

Soil: Aclonifen

Groundwater: Aclonifen

Surface water: Aclonifen

Sediment: Aclonifen

Air: Aclonifen

CA 7.5 Monitoring data

Monitoring data from databases in the EU

Evaluation of the peer reviewed literature (CA 7.5/02 to CA 7.5/10) was undertaken to complement the evaluation of public monitoring data (CA 7.5/01). Three references were identified outlining monitored air concentrations in urban and rural locations (see Table 7.5- 1). While the extent of the dataset is quite limited, the maximum measured concentration of 4.5 ng/m^3 is below human health thresholds.

A single study was identified where monitoring of groundwater fed springs was undertaken and no detections (Limit of detection [LOD] was much lower than the regulatory acceptable concentration [RAC]) of aclonifen were reported. Five studies report investigating concentrations of aclonifen in surface water (SW). In the majority of samples aclonifen was either not detected (LOD was much lower than the SW-RAC) or at concentrations below the SW-RAC. Only 3 samples were reported by [redacted] *et al*. (2010) to be above the SW-RAC of 1.36 µg/L, relating to the peak of a chemograph measured for a flood event in a small catchment in the south west of France in 2008. A similar investigation in 2006 by the same authors at multiple locations within the same catchment in 2006 for a flood event yielded a maximum concentration of 0.5 µg/L. Similarly, a flood event investigated in a different catchment by [redacted] *et al*. (2013) in the north of France yielded a maximum concentration of 1.11 µ/L. These limited investigations suggest that exceedance of the SW-RAC might occur under extreme weather and flooding conditions. During such flooding conditions depletion of

dissolved oxygen is more likely to impact aquatic species than a minor exceedance of the SW-RAC for a few hours. A single study reported monitoring data for sea water where in all samples aclonifen was not detected (LOD was lower than RAC).

Evaluation of the limited dataset extracted from peer reviewed literature in the light of the much larger dataset comprising the public monitoring data assessment does not change the conclusion that aclonifen is not expected to occur in any environmental compartment at concentrations that would suggest it presents a risk to the environment or human health when used according to the label.

Table 7.5- 1: Summary of peer reviewed literature that informs environmental concentrations in different compartments, namely air, ground water, surface water and sea water

Study	Compartment	Location	Number of samples	Number of samples > RAC	Samples > RAC (%)	Maximum Concentration (ng/m³)
M-457521-01-1 [redacted] *et al.*, 2010.	Air	France	10	0	0	4.5
M-458632-02-1 [redacted] *et al.*, 2010	Air	France	262	0	0	4.15
M-547551-01-1 [redacted] *et al.*, 2013	Air	France	10	0	0	Not quantified (LOQ 0.24 ng/m³)
M-463221-01-1 [redacted] *et al.*, 2012.	Ground water	Italy	45	0	0	Not detected (LOD 50 ng/L)
M-474496-02-1 [redacted] *et al.*, 2013	Surface water	France	10	0	0	1.11
M-477497-01-1 [redacted] *et al.*, 2010	Surface water	France	2006 – 10 2008 – 12	2006 – 0 2008 – 3?	2006 – 0 2008 – 25	2006 – 0.5 2008 – 1.50
M-642715-01-1 [redacted] *et al.*, 2018	Surface water	Sweden	6	0	0	Not detected (LOD 8 ng/L)
M-547409-01-1 [redacted] *et al.* 2014	Surface water	Denmark	16	0	0	Not detected (LOD 0.1 ng/L)
M-670490-01-1 [redacted] *et al.*, 2016	Surface water	France	22	0	0	0.217
M-547409-01-1 [redacted] *et al.*, 2014	Sea water	Denmark	12	0	0	Not detected (LOD 0.1 ng/L)

? – Interpolated from graph

Data Point:	KCA 7.5/01
Report Author:	[redacted]
Report Year:	2019
Report Title:	Statement: Public monitoring data for aclonifen
Report No:	EnSa-19-00553
Document No:	M-673651-01-1
Guideline(s) followed in study:	none
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

In order to provide an overview of potential findings of aclonifen (parent) residues in databases reflecting 'public' monitoring programs a search was conducted, based predominantly on internet searches identifying databases and reports which are publicly accessible e.g. from the Environmental Agencies of EU Member States.

Since only parent Aclonifen was regarded relevant for this overview of monitoring results, the search was conducted with "aclonifen" as key word. If applicable, the time period for which monitoring results were searched, was from 1995 to today (September of 2019). Where monitoring data was found this was analysed for exceedance of regulatory thresholds appropriate for the compartment and the results summarised. The search revealed that monitoring information is not generally publicly available for each Member State and not necessarily available at a national level, in some cases only being available for some states/regions/provinces.

The results from the groundwater monitoring data search indicates that aclonifen concentrations above the regulatory trigger of 0.1 µg/L are exceedingly rare (12 samples; 0.007% of 184651 analyses) and at most 10.4 µg/L.

The results from the surface water monitoring data search indicate that aclonifen concentrations above the Tier 1-RAC-SW (1.36 µg/L) are exceedingly rare (22 samples, 0.007% of 311942 analyses) and are at most 65 µg/L.

The results from the monitoring data search indicate that monitoring of aclonifen is quite sparse for sediment, soil and air compartments. Aclonifen concentrations above the LOQ (varies) in sediments and air are exceedingly rare and there is no indication that aclonifen is expected to be detected in soil.

It cannot be discounted that some of the detections identified are erroneous given they stem from non-GLP monitoring networks and programmes of unknown quality.

Overall it can be concluded from assessment of readily available public monitoring datasets that aclonifen does not pose a concern for the environment or human health for the following compartments - surface water, groundwater, drinking water, soil, air and sediment.

Data Point:	KCA 7.5/02
Report Author:	[redacted]; [redacted]; [redacted] M. R.; [redacted]; [redacted]; [redacted]
Report Year:	2010
Report Title:	Temporal variations of concentrations of currently used pesticides in the atmosphere of Strasbourg, France
Report No:	M-457521-01-1
Document No:	M-457521-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities.
Acceptability/Reliability:	

Executive Summary:

Atmospheric samples were collected in Strasbourg, Alsace, France between April 18 and May 29, 2007 and analysed for a total of 71 pesticides in use at that time. 38 pesticides were detected, including aclonifen. A significant correlation between decreasing concentration and time was observed aclonifen and a number of other pesticides. The concentration of aclonifen ranged from 0.64 to 4.50 ng m^{-3} and the average concentration was 1.78 ng m^{-3}.

I. MATERIALS AND METHODS

Air samples were collected in 2007 in the botanical garden of Strasbourg University, Strasbourg, Alsace, France and analysed for a large number of pesticides, including aclonifen. The site was located approximately 0.5 km from the town centre, 2 km from industrial zones and about 5 km from sites with maize and cereal crops. None of the pesticides was used in the Botanical Garden. Particulate and gas phases were sampled simultaneously by using a high-volume sampler on a 48 h basis for the period from April 18 to May 29, 2007. The same samples are discussed in a separate publication (M-547551-01-1).

Particulate and gaseous samples were collected simultaneously for 48 h periods on average, using 30 cm diameter glass fibre filter and XAD-2 resin at a flow rate of 9.96 L min^{-1}. After sampling, filters and resins were stored in the dark at -20 °C for a maximum of 4 days until extraction. Pesticides were extracted with hexane : dichloromethane (1:1) by Soxhlet extraction for 20 hours, prior to concentration and analysis. The concentration of aclonifen was measured by GC-MS/MS method with a limit of detection (LOD) of 99.81 pg m^{-3}. Analysis of the pesticides was done separately on samples of particulate and gaseous phases and the results combined to obtain the pesticide concentrations in the total atmosphere.

II. RESULTS

The measured concentrations of aclonifen in the total atmosphere are summarised in Table 7.5- 2

Table 7.5- 2: Air concentrations of aclonifen

Pesticide	Number of samples detected in	Concentration Range [ng m^{-3}]	Average Concentration ±95% CI [a]

			[ng m^{-3}]
Aclonifen	7 out of 10	0.64–4.50	1.78 ±1.67

[a] The average and 95% confidence intervals listed were calculated from the arithmetic mean and standard deviation of samples with concentration superior to the LOD

The gas-particle distribution of the detected pesticides was reported. For aclonifen <20% of the concentration was present in the gas phase and > 80% was present in the particle phase.

Although the exact reasons for the decline were not established a correlation between decreasing concentration with time was observed for 10 of the 71 pesticides analysed, including aclonifen. No correlation between the concentrations in air and temperature, atmospheric pressure or humidity was found for aclonifen.

Table 7.5- 3: Correlation coefficient and p-value for the correlation of time with atmospheric aclonifen concentrations

Pesticide	Correlation coefficient (r)	p-Value
Aclonifen	-0.8262	0.0125

Data Point:	KCA 7.5.03
Report Author:	[redacted]
Report Year:	2012
Report Title:	Occurrence of currently used pesticides in ambient air of centre region (France)
Report No:	M-458632-02-1
Document No:	M-458632-02-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

Air samples were collected from 2006 to 2008 at three rural and two urban sites in Centre Region, France and analysed for 56 pesticides, of which 41 were detected. The majority of pesticides showed a seasonal trend, with most of detections and highest concentrations occurring during the spring and early summer.

Aclonifen was detected in 7% of samples at an average concentration of 1.37 ng m^{-3} which was among the less frequently detected pesticides.

I. MATERIALS AND METHODS

Air samples were collected in 2006 to 2008 at three rural and two urban sites in Centre Region, France and were analysed for 56 pesticides, including aclonifen. The information on the sites is provided in in Figure 7.5- 1 and Table 7.5- 4.

Figure 7.5- 1: Sampling sites, types of agricultural crops and population density across Centre Region (France)

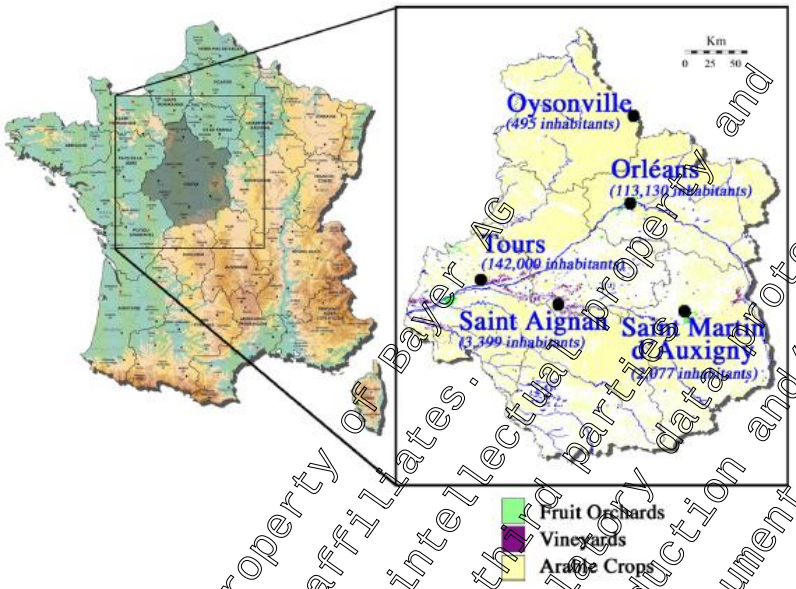


Table 7.5- 4: Description of the sampling sites and periods

Sampling site	Latitude	Longitude	Description
Tours (To)	[redacted]	[redacted]	Commercial and residential area. Samples collected at ground level.
Orléans (Or)	[redacted]	[redacted]	Parking in the city centre. Samples collected at 3.5 m above ground level
Saint Martin d'Auxigny (SM)	[redacted]	[redacted]	Rural and agricultural area surrounded by apple tree fields. Samples collected at ground level
Oysonville (Oy)	[redacted]	[redacted]	Rural and intensive agricultural area with mainly arable crops such as maize, wheat, soybean, barley and sunflowers. Samples collected at ground level.
Saint Aignan (SA)	[redacted]	[redacted]	Rural and agricultural area (vineyards). Samples collected at 6 metres above ground level.

The sampling campaign in 2006 ran for 26 weeks from 14 March to 12 September, in 2007 from 11 April to 9 July (except for Saint Martin d'Auxigny where it ran until 11 September) and in 2008 from 9 April to 2 July (except for Saint Martin d'Auxigny where it ran until 5 November).

Particulate and gaseous samples were collected using a Partisol 2000 low volume sampler fitted with quartz fibre filters and polyurethane foam (PUF) plugs. Filters and PUF plugs were exposed weekly at a flow of 1 $m^3 h^{-1}$. The total volume of air collected was approximately 168 m^3. Prior to extraction the samples were stored in the dark below 4 °C for up to 24 hours after sampling and then at -18 °C. Pesticides were extracted with dichloromethane using an ASE 300 PLE system prior to analysis. The concentration of aclonifen was measured by a GC-MS method with a LOQ of 0.24 ng m^{-3}

II. RESULTS

The use of pesticides in the Centre region, France is intense with 41 out of 56 monitored pesticide detected. Aclonifen was among the less frequently detected pesticides as indicated by an overall detection frequency of 7%. The majority of detects were a result of pesticide use in vineyards,

orchards, potatoes and arable crops such as wheat, beet, maize, barley and sunflowers, which were the main crops in the vicinity of the three rural sites sampled. The agricultural uses listed for aclonifen are arable crops (sunflower, peas) and potatoes.

An overall summary of the concentrations of aclonifen detected in air at the five urban and rural sampling sites from 2006 until 2008 are summarised in Table 7.5- 5.

Table 7.5- 5: Overall concentrations of aclonifen in all sampling sites for 2006 to 2008 [a]

Pesticide	Total samples analysed (N)	Frequency of detection (%)	Concentration Range [ng m^{-3}]	Average Concentration ±95% CI [ng m^{-3}]
Aclonifen	262	7	0.23-4.15	1.37 ±0.81

[a] No Aclonifen was detected in 2007 at any sampling site
[b] The average and 95% confidence intervals listed were calculated from the arithmetic mean and standard deviation of samples with concentration in excess of the LOD

Details of the concentrations of aclonifen detected in air each sampling site in 2006 and 2008 are summarised in Table 7.5- 6 and Table 7.5- 7, respectively. No aclonifen was detected at any of the fived sampling sites in 2007.

Table 7.5- 6: Spatial distribution of aclonifen detects during 2006 at all stations

Pesticide	Station	Frequency of detection (%)	Concentration Range [ng m^{-3}]	Average Concentration ±95% CI [a] [ng m^{-3}]
Aclonifen	Oysonville (n=25)	24	0.86-4.15	1.78
	Saint Martin d´Auxigny (n=24)	8	0.23-0.83	0.53
	Tours (n=24)	Not detected	-	-
	Saint Aignan (n=17)	Not detected	-	-
	Orléans (n=23)	Not detected	-	-

n = number of samples with one or more pesticides detected.
[a] The average was calculated from the arithmetic mean of samples with concentration above the LOD
LOQ = 0.24 ng m^{-3}

Table 7.5- 7: Spatial distribution of aclonifen detects during 2008 at all stations

Pesticide	Station	Frequency of detection (%)	Concentration Range [ng m^{-3}]	Average Concentration ±95% CI [a] [ng m^{-3}]
Aclonifen	Oysonville (n=10)	20	0.67-1.55	1.11
	Saint Martin d´Auxigny (n=29)	10	1.13-1.58	1.36
	Tours (n=12)	25	0.90-1.81	1.25
	Saint Aignan (n=12)	8	-	1.22
	Orleans (n=12)	17	1.09-2.01	1.55

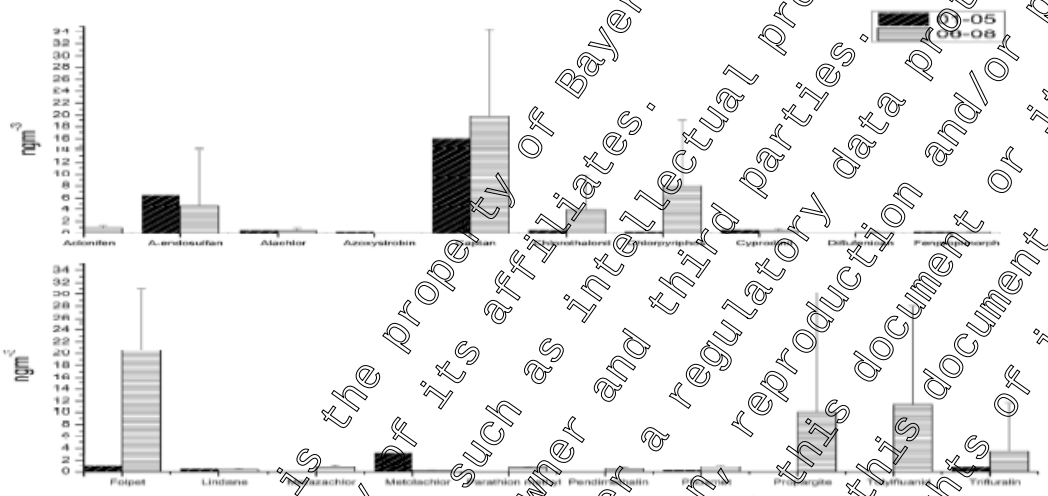
n = number of samples with one or more pesticides detected.
[a] The average was calculated from the arithmetic mean of samples with concentration above the LOD
LOQ = 0.24 ng m^{-3}

There was not enough information to correlate farmers' practices, such as application rate and application date, with pesticide occurrence in the atmosphere. However, it was clear pesticide

concentrations outside of application periods were very low. Pesticides applied in the three rural areas could be transported to the urban sites when the direction and the velocity were appropriate.

A similar study was conducted by the Lig'Air (2005) group from 2001 to 2005 on the Saint Martin d'Auxigny (SM) sampling site, with 23 pesticides in common between both studies (including aclonifen). A comparison of the average concentrations for these compounds (in 2001 to 2005 and in 2008 to 2008) is shown in Figure 7.5- 2. As can be seen aclonifen was not detected in 2001 to 2005 and when detected in 2006 to 2008 was present at very low levels.

Figure 7.5- 2: Temporal distribution of 23 pesticides during 2001-2008 in Saint Martin d´Auxigny site



Data Point:	KCA 7.5/04
Report Author:	[redacted]
Report Year:	2013
Report Title:	Particle size distributions of currently used pesticides in a rural atmosphere of France
Report No:	M-547551-01-1
Document No:	M-547551-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

Air samples were collected in spring and summer 2009 for a rural site of Centre Region (France). A gravimetric impactor was used to measure the atmospheric aerosols size distribution as four size

fractions; 0.03-0.1 mm, 0.1-1 mm, 1-10 mm and more than 10 mm. The fractions were analysed for 50 pesticides, of which 10 were detected. For the remainder, including aclonifen, none were detected.

I. MATERIALS AND METHODS

Ten air samples were collected at ground level between 30 March and 8 June 2009 at a location in the village of Oysonville (48°23'35"N and 01°56'57"E, population 501 in 2010) in Region Centre, France. The station was in the countryside in the vicinity of arable fields and orchards. The sampling period coincided with spraying of pesticides.

The sampling periods were from 30 March to 14 April 2009, 14 to 27 April 2009, 27 April to 11 May 2009, 11 May to 25 May 2009 and 25 May to 8 June 2009.

Air samples were collected on 25 mm diameter quartz fibre filters for 7 days with an average flow rate of 17 L min^{-1}. The total volume collected ranged from 139 to 193 m^3. A gravimetric impactor was used to measure the atmospheric aerosols size distribution. Particle size distribution was measured for four size fractions; 0.03-0.1 mm, 0.1-1 mm, 1-10 mm and more than 10 mm.

Pesticides were extracted from each DPLE impactor fraction with dichloromethane using an ASE 300 PLE system. The concentration of aclonifen was measured by a GC-MS method with a LOQ of 0.24 ng m^{-3}

II. RESULTS

10 out of 49 pesticides analysed for were detected in the various particle size fractions, but not aclonifen.

Data Point:	KCA 7.5.05
Report Author:	[redacted]
Report Year:	2012
Report Title:	Herbicide contamination and dispersion pattern in lowland springs
Report No:	M-463221-01-1
Document No:	M-463221-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

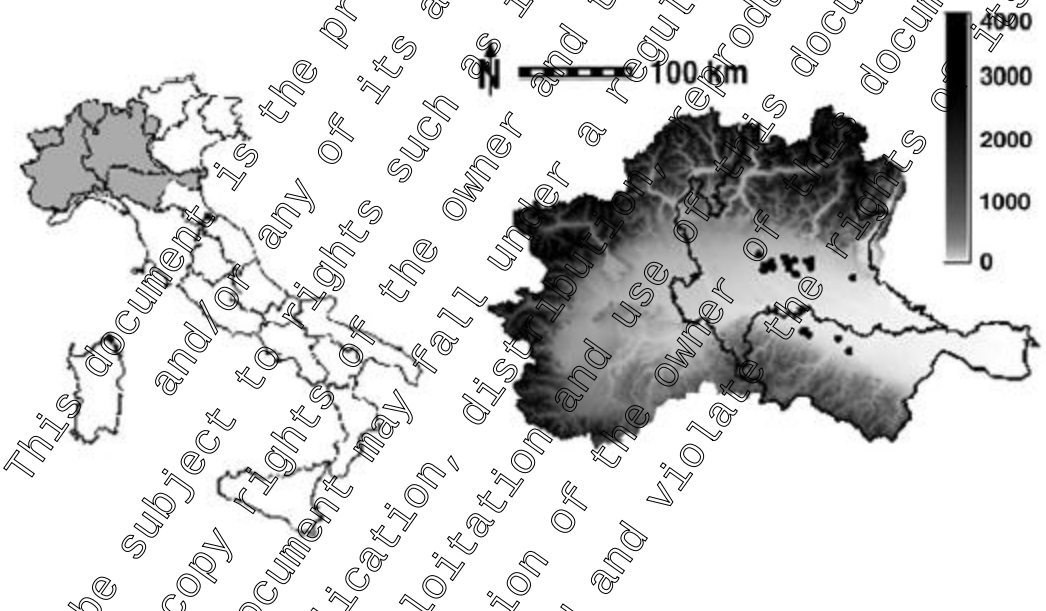
The main objectives in this paper were to: (1) to map herbicide contamination in lowland springs, (2) to evaluate the potential risk for biota and (3) to quantify the extent of the area from which the herbicide use can affect the water quality of lowland springs. In June and August 2009, nearly 23 springs within the Po River Plain (Northern Italy) were sampled and analysed for five herbicides used to control weeds in maize which included aclonifen. Hydrogeological properties, half-lives of the herbicides and their concentrations in both groundwater and springs were used to quantify the area

from which the contamination could originate. Such evaluation was performed by means of GIS techniques. Spatial analyses reveal that the theoretical area from which herbicides can contaminate spring water is within a distance varying between a few and 1800 m. The findings indicated that conservation plans should focus on the fields adjacent to or surrounding the springs and should address the optimization of irrigation practices, restoration of buffer strips, crop rotation and in general more sustainable agricultural practices in the proximity of these fragile GDE. Aclonifen was never seen at concentrations greater than the LOD 50 ng L^{-1}.

I. MATERIALS AND METHODS

The Po River Plain is heavily used for agriculture. The springs in this study, located in the left bank and in the right bank respectively of the Po River, are located in the Lombardy and Emilia Romagna that represent together approximately 63% of the Po River basin (Figure 7.5- 3). Flooding is the main irrigation practice in the Lombardy region (INEA, 2009b), whereas sprinkler irrigation is the main practice in Emilia Romagna (INEA, 2009a).

Figure 7.5- 3: Location of the sampled springs (black dots). The black line in the right picture separates Lombardy, in the North, from a portion of the Emilia Romagna to the South



In late June and late August 2009, 22 and 22 springs representative of a large portion of the central area of the Po River Plain were sampled. Selected springs were in the Lombardy region (n=18) and in Emilia Romagna region (n=6) (Figure 7.5- 3). Water samples were collected from the springs at the outlet and analysed for physical and chemical parameters and herbicides which included aclonifen.

The water samples were collected in 1 litre glass bottles, stored at 4 °C and analysed within a week. As maize is the most extensive crop in the study area, herbicides used both in pre- and post-emergence treatments in this crop were selected for investigation which included aclonifen. Water samples were filtered on Buchner filters and the herbicides extracted using a C18 SPE cartridge, concentrated and analysed by GC–MS analysis. Detection limit was 50 ng L^{-1} for aclonifen. The quantification of aclonifen was performed in single ion monitoring at 264 m/z.

II. RESULTS

Aclonifen was never seen at concentrations greater than the LOD 50 ng L^{-1}.

Data Point:	KCA 7.5/06
Report Author:	[redacted]
Report Year:	2013
Report Title:	Pesticide contamination interception strategy and removal efficiency in forest buffer and artificial wetland in a tile-drained agricultural watershed.
Report No:	M-474496-02-1
Document No:	M-474496-02-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

Field-scale monitoring of the concentration and load transfer of 16 pesticides out of a tile-drained catchment (Bray, France) was conducted for three years and the reduction of pesticides monitored through two buffer zones: an artificial wetland (AW) and a forest buffer (FB).

Pesticide load reductions between inlet and outlet ranged from 45% to 96% (AW) and from -32% to 100% (FB) depending on the pesticide molecule and the hydrological year.

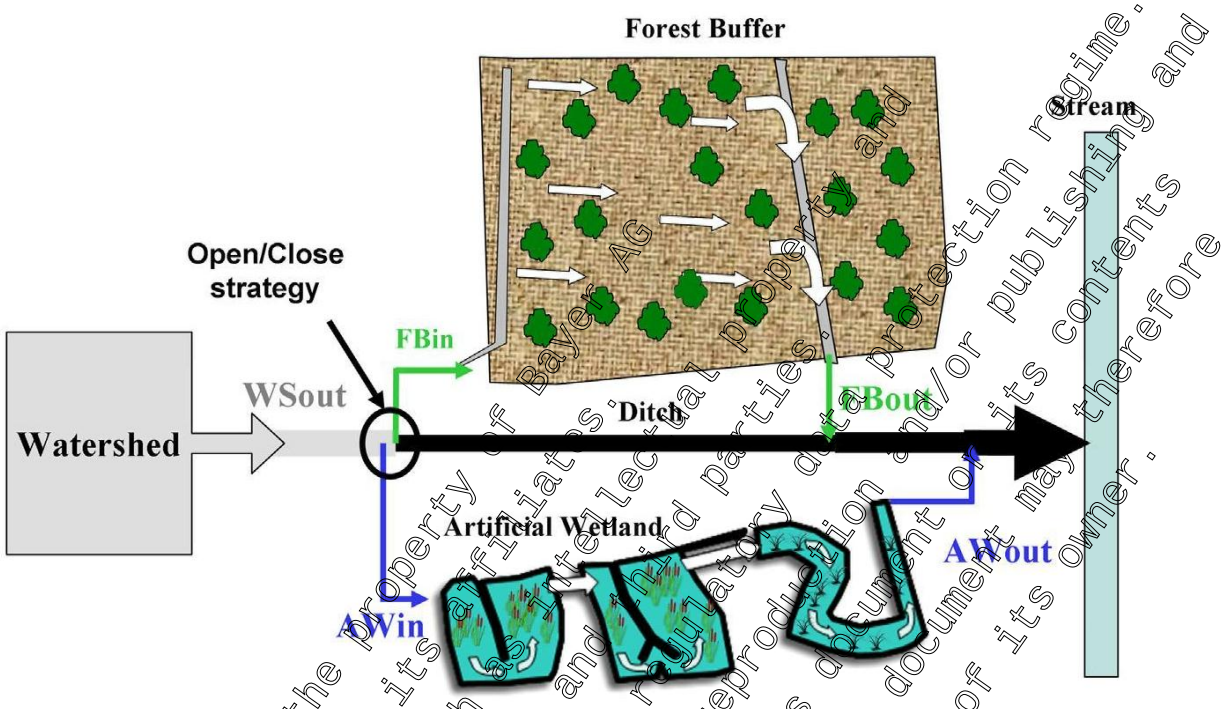
Aclonifen was detected on occasion at the watershed outlet (= buffer zone inlet) at a median concentration of 0.05 µg/L. The watershed outlet load totalled 3.40 ± 0.97 g for 2007-2010 which represented 69% reduction of the watershed load (80% AW and 34% FB, associated with high uncertainties due to the low concentrations).

I. MATERIALS AND METHODS

A field monitoring programme was undertaken from October 2007 until May 2010 in a tile-drained catchment located in Bray, France. A total of 16 pesticides were analysed for, including aclonifen which was applied by the farmer during the monitoring period and had also been applied before monitoring started from 2002 to 2007. The average annual applied mass of aclonifen was calculated as 600 g/ha/year.

Water flow rates, water volumes, pesticide concentrations and pesticide loads were collected at five locations: watershed outlet, artificial wetlands (AW) and forest buffer (FB) inlets and outlets, for three years. At the outlet of the Bray catchment parallel to the main agricultural ditch, there was a forest buffer (FB) and a series of three artificial wetlands (AW) as shown in Figure 7.5- 4. The surface area of the buffer zones was 1600 m^2 (FB) and 1280 m^2 (AW), each <0.5% of the watershed surface area. Water coming from the Bray catchment can either enter one or both of the buffer zones or flow straight through the main ditch down to Le Calais stream.

Figure 7.5- 4: Diagram of forest buffer (FB) and artificial wetland (AW) placement in the watershed (WS) showing inlet (in) and (outlet) flows.



Composite water samples, weighted for flow rate, were taken continuously from November 2007 at the watershed and buffer zone outlets using automatic samplers and were collected approximately once a week. Water samples were frozen until analysis. Water samples were filtered (0.20 µm), subject to solid-phase microextraction (SPME) and analysed by GC-MS. The limit of quantification for aclonifen was 0.10 µg/L.

II. RESULTS

Of the 16 pesticides molecules that were analysed for, nine (including aclonifen) were applied by the farmer during the monitoring period. Aclonifen was usually not detected. The concentration range for the watershed outlet is presented in Table 7.5- 8.

Table 7.5- 8: Concentration values of aclonifen at the watershed outlet during the 2007-2010 monitoring period

Compound	Concentration values (ng/L) Buffer zone inlet = watershed outlet [A]					
	Minimum	Maximum	Median	Mean	Standard deviation	Number of concentration values
Aclonifen	0.10	1.11	0.05	0.18	0.33	10

[A] Sampling equipment at buffer zone inlet corresponds to watershed outlet

Watershed outlet load and associated uncertainties are reported in Table 7.5- 9. Aclonifen presented one of the lowest watershed outlet loads and was rarely quantified, totalling 3.40 ± 0.97 g for 2007-2010. For compounds such as aclonifen usually less than 2% of the applied mass was recovered yearly at the outlet of the catchment. Consequently the loads calculated were associated with very large uncertainties e.g. aclonifen in the FB inlet (0.07 ± 0.15 g) and outlet (0.04 ± 0.10 g).

Table 7.5- 9: Artificial wetland (AW), forest buffer (FB) and watershed (WS) loads and load reductions for the whole 2007–2010 monitoring period

	Artificial wetland				Forest buffer				Watershed	
	Inlet		Outlet	η [A]	Inlet		Outlet	η [A]	Outlet	η [C]
	Load m^3 or g [B]	AW_{in}/WS_{out} %	Load m^3 or g [B]	%	Load m^3 or g [B]	FB_{in}/WS_{out} %	Load m^3 or g [B]	%	Load m^3 or g [B]	%
Water volumes	116749 ± 140	50	63915 ± 198	45	20742 ± 119	9	14611 ± 232	30	234447 ± 159	25
Aclonifen	2.54 ± 0.48	85	0.59 ± 0.33	80	0.07 ± 0.15	1	0.04 ± 0.10	34	3.40 ± 0.97	69

[A] Load reduction in the artificial wetland and forest buffer for the whole 2007–2010 monitoring period
[B] Water volumes (m^3) and pesticide loads (g) are provided at the inlet (in) and outlet (out) of the systems
[C] Reduction of the watershed load corresponds to the portion of pesticides that was actually dissipated through the two buffer zones and did not reach the stream.

Data Point:	KCA 7.5/07
Report Author:	[redacted]
Report Year:	2013
Report Title:	Flood event impact on pesticide transfer in a small agricultural catchment (Montousse at Auradé, south west France)
Report No:	M-477497-01-1
Document No:	M-477497-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

In this paper, dynamic transfer of aclonifen is studied during two flood events in a small experimental catchment close to Toulouse (south west France). The concentration of aclonifen was determined by a multi-residue technique on filtered and unfiltered waters. The results showed very high concentrations when compared to low flow periods and to the data collected by the French institutional networks in charge of the pesticide river water pollution survey. The concentration of aclonifen was greater than 1 μgL^{-1} in the unfiltered waters. The concentration and flux of aclonifen increased during the flood flows. Pesticide concentrations in unfiltered waters and partitioning between dissolved and particulate fractions (Kd=[diss]/[part]) are controlled by dissolved organic carbon and total suspended matter.

I. MATERIALS AND METHODS

The experimental area, Montoussé catchment at Auradé, is located in the Midi-Pyrénées province (south west France), 35 km west of Toulouse. The study area is a hillside of the 'Coteaux de Gascogne' with an altitude of approximately 300 m. The geological substratum is a Miocene molassic

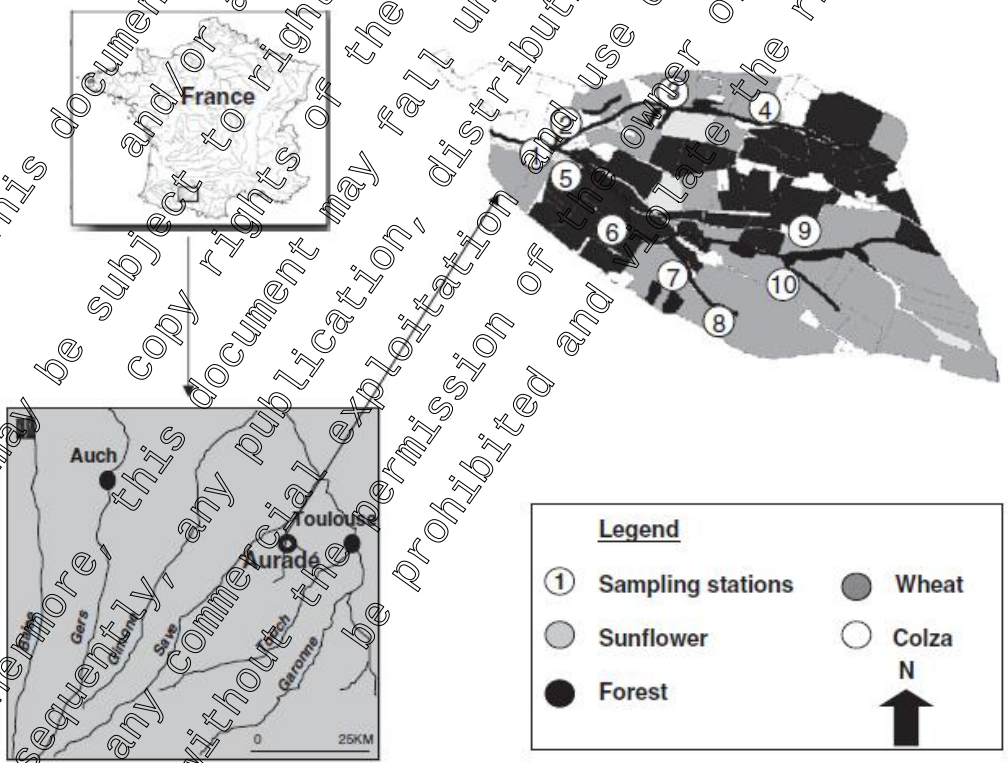
deposit (called molasse). The study area is characterised by a fairly impermeable substratum due to its widely extended clay content. As the result of this geological substratum, river discharge is mostly supplied by surface and sub-surface run-offs. Groundwater reservoirs are very limited and during the summer dry period the stream discharge is very low and sometimes the creek is dried. The land slopes to the north varying between 0% and 20%. This area is drained by a system of river flowing to the north into the main fluvial axis of south west France, the Garonne River.

The Montoussé creek at Auradé drains a catchment area of 328 hectares, of which 90% is devoted to agricultural activities on highly fertile land with calcareous (around pH=8) and clayey (36%) soils. The main crops are winter wheat (20%) and durum wheat (31%) in rotation with sunflower (47%). The main period for herbicide use is the end of April and beginning of May for triazine on sunflower and from the end of November to January for phenylurea on wheat. In this survey, 10 sampling stations were selected on the Montoussé creek and on its tributaries according to soil occupation.

The climate of Auradé area is characterised as oceanic because of the influence that the Atlantic ocean plays in regulating temperature variations and therefore determining climate conditions. The average annual precipitation is about 700mm to 800 mm, mostly in the form of rain, which is the main hydrological source of supply for surface and subsurface run-offs in this area with the highest rate of discharge in February while the water flows more slowly from June to September. The bulk of annual rainfall occurs, in the form of thunderstorms, from November through December and April to May.

Two storm events (March 2006 and May 2008) were sampled in this catchment. During the first one, two 2.5 L water samples were collected during the peak discharge in glass jars at 10 stations, spatially distributed within the whole catchment as shown in Figure 7.5- 3.

Figure 7.5- 5: Geographical situation map of the Montoussé experimental catchment at Auradé (Gers, south west France) and location of the sampling stations (1 to 10).



During the second storm, 12 samples each of between 0.8 to 2.5L depending on the discharge intensity were collected over the whole period of the event at the outlet of the catchment using an automatic

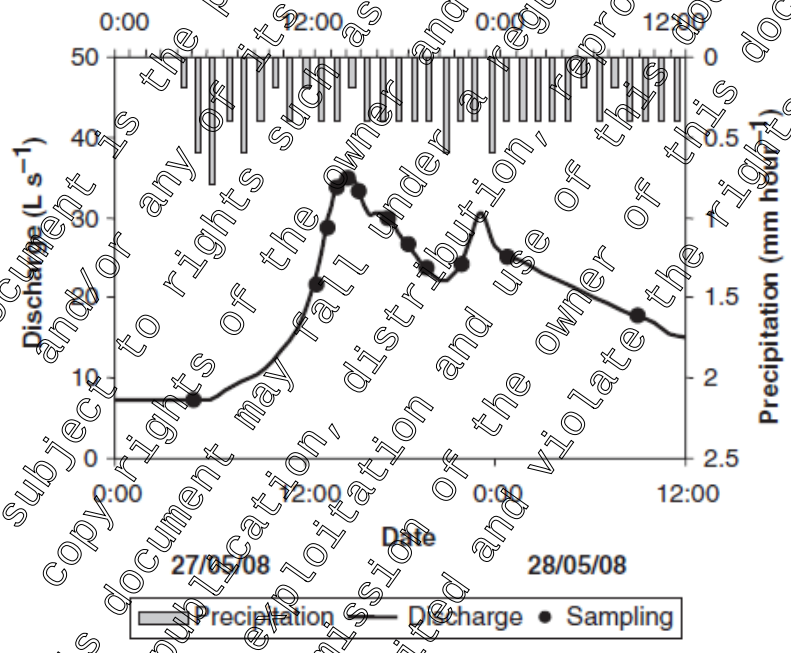
sampler. The automatic sampler was programmed to collect samples at 30 min intervals. Dichloromethane (1 : 40; v/v) was added to the unfiltered water samples, in the field to avoid bacterial activity, and stored in glass bottles with Teflon-lined lids in the dark and cold until extraction the next day.

The water samples were filtered under vacuum through 0.45 µm filters then partitioned with dichloromethane. The dichloromethane fraction was then dried over anhydrous sodium sulphate and the remaining organic phase evaporated to dryness under vacuum. The dry residue was then reconstituted in hexane. Any pesticides present in the samples was identified by gas chromatography. Recoveries were determined using water samples spiked with the individual pesticides. Average recovery rates ranged from 92 to 102%. The limit of detection based on a signal to noise ratio of 3 was estimated at 0.005 to 0.01 $\mu g L^{-1}$ according to the molecules.

II. RESULTS

Due to Montousse´ small catchment size, a direct response of the discharge to the rainfall was observed (**Table 7.5- 6**).

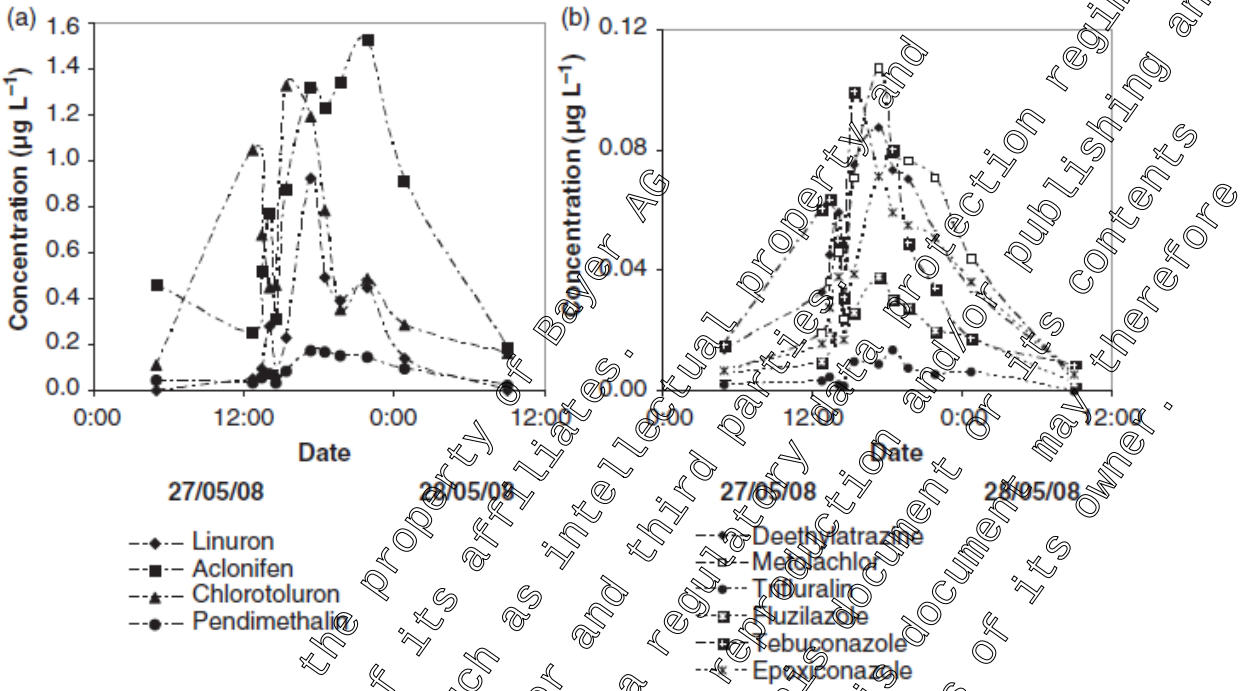
Figure 7.5- 6: Hourly precipitation and stream discharge measured at the outlet of the Montoussé catchment (station 1) during the flood event of May 2008. Black circles on the hydrograph represent the sampling periods



In the unfiltered waters most of the pesticide concentrations increase during the flood event with increasing discharge although there were two magnitudes for pesticide concentration as shown in Figure 7.5- 7 for unfiltered waters.

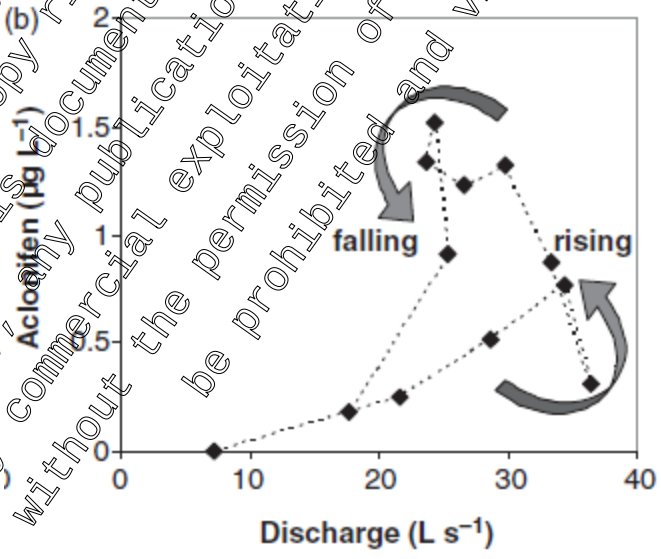
Aclonifen reached a maximum concentration of 1.5 µg L^{-1}. The concentration of aclonifen in the unfiltered water was higher after the peak discharge during the recession period than during the rising discharge, showing a hysteresis between the two hydrological periods. This means most of the aclonifen is mainly exported by the subsurface run-off Figure 7.5- 7 (a).

Figure 7.5- 7: Variations of pesticide concentrations in unfiltered waters at the outlet of the Montoussé catchment (station 1) during the flood event of May 2008 (a) High concentration molecules, (b) low concentration molecules.



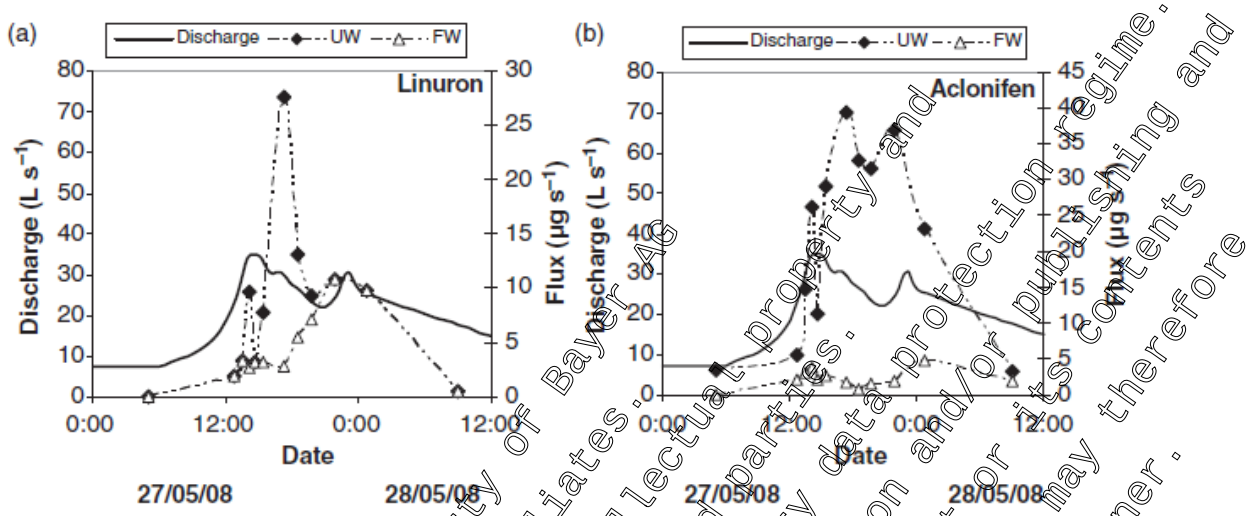
This hysteresis phenomenon is illustrated in Table 7.5- 8 for aclonifen, which shows there is a lag of concentration between the rising period and the falling limb of the hydrograph for aclonifen concentrations during the recession period, when the sub-surface run-off contribution reaches its maximum, than during the rising discharge.

Figure 7.5- 8: The relationships between aclonifen concentration and stream discharge at the outlet of the Montoussé catchment during the flood event of May 2008, showing the hysteresis phenomenon between rising and falling limbs of the storm hydrograph



As with concentration there was an increase in flux during the flood event in the unfiltered waters as well as in the filtered waters for aclonifen.

Figure 7.5- 9: Discharge and variations of linuron (a) and aclonifen (b) fluxes measured in filtered (FW) and unfiltered (UW) waters during the flood flow of May 2008.



The results obtained during the flood of March 2006 show that for the different stations, the pesticide concentrations are very high in different fractions (filtered water, unfiltered water and suspended matter) compared to low flow periods and also to the data collected by the French institutional networks in charge of the river water pesticide pollution survey. In the unfiltered water aclonifen had concentrations between 0.1 and 0.5 $\mu g L^{-1}$.

Data Point:	KCA 7.5.08
Report Author:	[redacted]
Report Year:	2018
Report Title:	Concentrations, fluxes and field calibration of passive water samplers for pesticides and hazard-based risk assessment
Report No:	M-642713-01-1
Document No:	M-642713-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

The performance of three passive sampler types; Chemcatcher® C18, polar organic chemical integrative sampler-hydrophilic–lipophilic balance (POCIS-HLB) and silicone rubber (SR) based on polydimethylsiloxane (PDMS) was evaluated at two sampling locations in southern Sweden over a period of 6 weeks and compared to time-proportional composite active sampling. Analysis for 124 pesticides including aclonifen was performed.

In total 90 pesticides were detected, 52 using active sampling and 32, 58, and 69 using the passive samplers Chemcatcher® C18, POCIS-HLB and SR, respectively. No concentrations of aclonifen were detected by active sampling, or by passive samplers Chemcatcher® C18 and POCIS-HLB at either site over the six week period. Aclonifen was detected at both sites at a mean concentration of 0.56 and 0.15 ng/L using passive SR samplers.

I. MATERIALS AND METHODS

Water samples were collected between 8 July to 19 August 2013 from two monitoring stations included in the Swedish national pesticide monitoring programme using three different passive samplers; Chemcatcher® C18, polar organic chemical integrative sampler-hydrophilic–lipophilic balance (POCIS-HLB) and silicone rubber (SR) based on polydimethylsiloxane. Active sampling, that is, time-proportional composite active samples (subsamples taken every ~90 minutes) were collected every week during the exposure period of the passive samplers. At site 2 the passive samplers were deployed 1 km further downstream of the active sampling point because of low water concentrations at the active sampling site.

The aim of the work was to compare the performance of the three passive samplers compared to active sampling and to compare *in situ* sampling rates (R_S) and passive sampler-water partition coefficients (K_{PW}) with those obtained under laboratory conditions.

Some details of the sampling sites are given below.

Table 7.5- 10: Details of the sampling sites

Parameter	Site 1	Site 2
Catchment area	14 km^2	8 km^2
Agricultural activities	85%	92%
Average annual water flow	15540 m^3	5744 m^3
Median flow during the sampling period	2900 m^3d	92 m^3d^{-1}

Six passive samplers of each type were deployed at each site for 7 days in total covering a period of six weeks (8 July to 19 August 2013). 2 x 4 additional samplers of each type were deployed in duplicate for 7, 14, 28 and 42 days at Site 1.

In total 124 pesticides were analysed for using a variety of methods. Aclonifen was analysed by gas chromatography–mass spectrometry (GC–MS). For this method the SR strips were extracted with petroleum ether/acetone (50/50, v/v) by Soxhlet extraction for 19h, the POCIS-HLB was extracted with ethyl acetate by solid-phase extraction (SPE) and the Chemcatcher® C18 was sonicated with ethyl acetate. Extracts were concentrated under nitrogen and redissolved in cyclohexane/acetone (90/10, v/v) prior to analysis by GC-MS in selective ion monitoring mode using electron ionization (EI).

The LODs for the various methods were 8 ng/L for the active sampling method (see Table 7.5- 11) and 15, 290 and 0.11 ng/L for the Chemcatcher® C18, POCIS-HLB and SR passive samplers (see Table 7.5- 12).

Table 7.5- 11: Details of the active sampling method

Substance	Method	LOD (ng/L)	LOQ (ng/L)	Recovery (%)
Aclonifen	GC-EI-MS	8	20	81

Table 7.5-12: Calculated limit of detection (LOD) for passive sampler methods

Substance	Chemcatcher® C18 (ng/L)	POCIS-HLB (ng/L)	SR (ng/L)
Aclonifen	15	290	0.11

II. RESULTS

52 out of 124 pesticides analysed for were detected using active sampling at site 1 and site 2 over a period of six weeks, while 32, 58, and 69 individual pesticides were detected using the passive samplers Chemcatcher® C18, POCIS-HLB and SR, respectively. No concentrations of aclonifen were detected by active sampling, or by passive samplers Chemcatcher® C18 and POCIS-HLB at either site over the six week period. Aclonifen was detected at both sites at a mean concentration of 0.56 and 0.15 ng/L using passive SR samplers.

Table 7.5- 13: Concentration of aclonifen in samples collected over 6 week sampling period (8 July to 19 August 2013)

	Concentration of aclonifen (ng/L)							
	Site 1				Site 2			
Method	Active sampling	Chemcatcher® C18	POCIS-HLB	SR	Active sampling	Chemcatcher® C18	POCIS-HLB	SR
DF	-	-	-	67%	-	-	-	50%
Mean	-	-	-	0.56	-	-	-	0.15
Median	-	-	-	0.33	-	-	-	0.12
Min	-	-	-	0.21	-	-	-	0.25
Max	-	-	-	2	-	-	-	0.42

DF = Detection frequency

Laboratory sampling rates (R_S) in L/day and sampler-water partitioning coefficients (K_{PW}) are summarised for aclonifen in Table 7.5- 14. *In situ* R_S and K_{PW} could not be calculated for all detected pesticides at sites 1 and 2, including aclonifen (see Table 7.5- 15).

Table 7.5- 14: Laboratory sampling rates (R_S, L/day) and sampler-water partitioning coefficients (K_{PW}, L/ kg) applied for the calculation of TWA concentrations

Substance	SR		POCIS-HLB		Chemcatcher® C18	
	R_S (L/day)	K_{PW} (L/kg)	R_S (L/day)	K_{PW} (L/kg)	R_S (L/day)	K_{PW} (L/kg)
Aclonifen	0.54	3.5	0.003	2.7	0.05 [A]	3.2 [A]

[A] Calculated value

Table 7.5- 15: *In situ* sampling rates (R_S, L/day) and sampler-water partitioning coefficients (K_{PW}, L/kg)

Substance	SR		POCIS-HLB		Chemcatcher® C18	
	R_S (L/day)	K_{PW} (L/kg)	R_S (L/day)	K_{PW} (L/kg)	R_S (L/day)	K_{PW} (L/kg)
Aclonifen	NC	NE/NC	NPW	NPW	NPW	NPW

NC = not calculable
NE/NC = not equilibrated in the passive sampler and not detected in the water.
NPW = not detected in passive

Data Point:	KCA 7.5/09
Report Author:	[redacted]
Report Year:	2013
Report Title:	New priority substances of the European Water Framework Directive: Biocides, pesticides and brominated flame retardants in the aquatic environment of Denmark
Report No:	M-547409-01-1
Document No:	M-547409-01-1
Guideline(s) followed in study:	--
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

The biocides cybutryn (Irgarol) and terbutryn, the herbicides aclonifen and bifenox, the insecticides cypermethrin and heptachlor/heptachlor epoxide and the brominated flame retardant hexabromocyclododecane (HBCD) are new priority substances of the Water Framework Directive of the European Union. In order to gain knowledge about their presence in the aquatic environment in an off-season situation with regard to pesticide and biocide applications, these substances were analysed in freshwater, seawater and fish samples from Denmark. Aclonifen was below the limit of detection (LOD) in all samples. No concentration was above maximum allowable concentration (MAC)-EQS values.

I. MATERIALS AND METHODS

As far as possible, the selection of samples and locations were based on the compound use and thus, expected emission sources their geographical distribution is illustrated in Figure 7.5- 3. However, compromises were made to allow multi-component analyses. It should also be noted that all samples were collected in the autumn 2012, i.e. outside the main season of biocide and pesticide applications. This approach was chosen as the main interest was on concentrations of priority substances in relation to AA-EQS values, i.e. background rather than peak concentrations. This approach probably has implications for the concentrations of biocides and pesticides, which will likely be lower than during their main usage period.

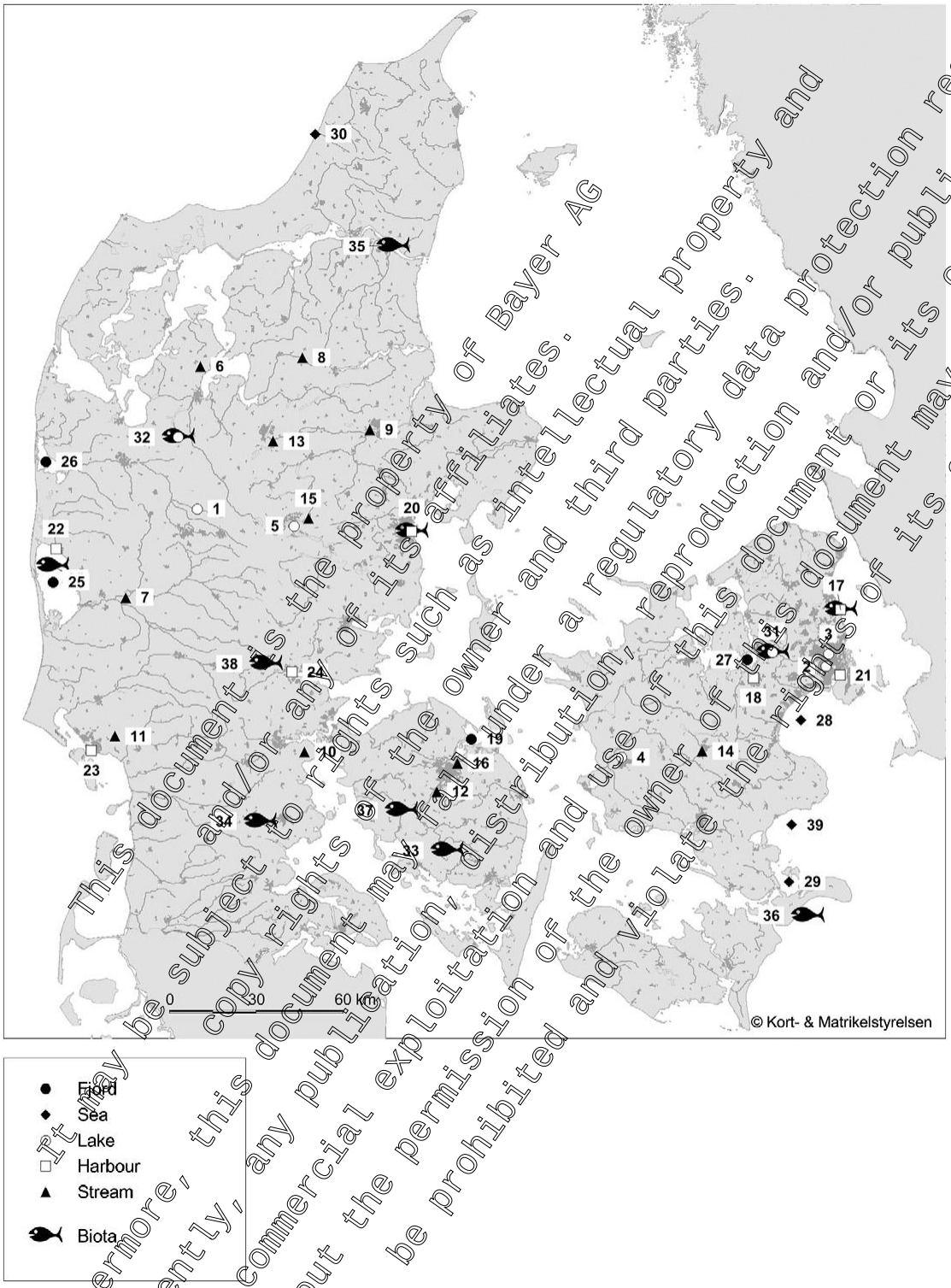
In general, stations in agricultural areas were the first choice for analyses of the pesticides aclonifen and bifenox were analysed together with cybutryn and terbutryn, meaning that these four compounds were determined in samples with potential influences from agricultural and urban areas as well as marinas and harbours. For aclonifen, bifenox, cybutryn and terbutryn, 1 L grab samples were collected. In addition, four 24 h composite freshwater samples were sampled.

The water samples were collected manually in glass flasks, which had been solvent rinsed and heated at 450 °C. Four samples were collected as 24-hour-time controlled composite samples These samples were automatically mixed subsamples of 50–100 mL taken every 15 minutes and combined into one composite sample. For discrete sampling, closed glass flasks were lowered into the water and opened approximately 30 cm under the water surface, to avoid contributions from potential surface films. The flasks were rinsed several times and then filled to the top under water. The samples were stored in the dark at approximately 4°C for a maximum of ten days prior to processing in the laboratory. Individual

fish samples were stored in nylon bags (Rilsan®) at −20 °C prior to pooling and in pre-cleaned amber glasses after pooling. Samples were not re-frozen after pooling but processed immediately.

Aclonifen was extracted from the water by solid-phase extraction (SPE) and analysed by high performance liquid chromatography (HPLC) using a neutral methanol/water gradient and tandem mass spectrometry (MS/MS) with atmospheric pressure chemical ionization (APCI−). Two multi-reaction monitoring transitions were recorded for aclonifen. Procedural blanks were analysed for every 10th sample. The limits of detection were determined as three times the signal-to-noise ratio.

Figure 7.5- 10: Map of Denmark with the geographical distribution of the fish and water samples of this study



II. RESULTS

Aclonifen was not found at concentrations above the limit of detection of 0.1 ngL-1 (Table 7.5- 2), possibly a result of the off-season sampling. Approximately 21,000 kg of aclonifen was sold in 2011, mainly for use on potatoes and vegetables. The same number had been reported for 2009, while only about 10,500 kg had been sold in 2010. Given the snapshot character of the sampling strategy and the limited number of samples, the non-detectable concentrations of aclonifen in this study do not exclude

the presence of aclonifen in the aquatic environment of Denmark. It cannot be concluded either that findings comply with AA-EQS and MC-EQS values of the Water Framework Directive. However, the off-season sampling indicates limited emission of aclonifen and/or rapid removal from water, assuming that the sampling locations represent areas of applications of aclonifen.

Table 7.5- 16: Summary of environmental quality standards (EQSs) for annual averages (AA-EQSs) and maximum allowable concentrations (MAC-EQSs), numbers of samples, detection frequencies, frequencies of AA-EQS exceedance, limits of detection (LODs), minimum and maximum concentrations. ww: wet weight. n.a.: not available (because of LOD N AA-EQS)

Water	No of samples	Detection frequency	AA-EQS or biota EQS	MAC-EQS	Exceedance of AA-EQS	LOD	Minimum conc	Maximum conc
Freshwater	16[a]	0%	120 ng/mL	120 ng/mL	0%	0.1 ng/mL	<LOD	<LOD
Seawater	14	0%	12 ng/mL	12 ng/mL	0%	0.1 ng/mL	<LOD	<LOD

[a] Including four 24-hour-samples

Data Point:	KCA 7.5.1.0
Report Author:	[redacted]
Report Year:	2016
Report Title:	Trace element and pesticide dynamics during a flood event in the save agricultural watershed: soil-river transfer pathways and controlling factors
Report No:	M-670490-01-1
Document No:	M-670490-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	-
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

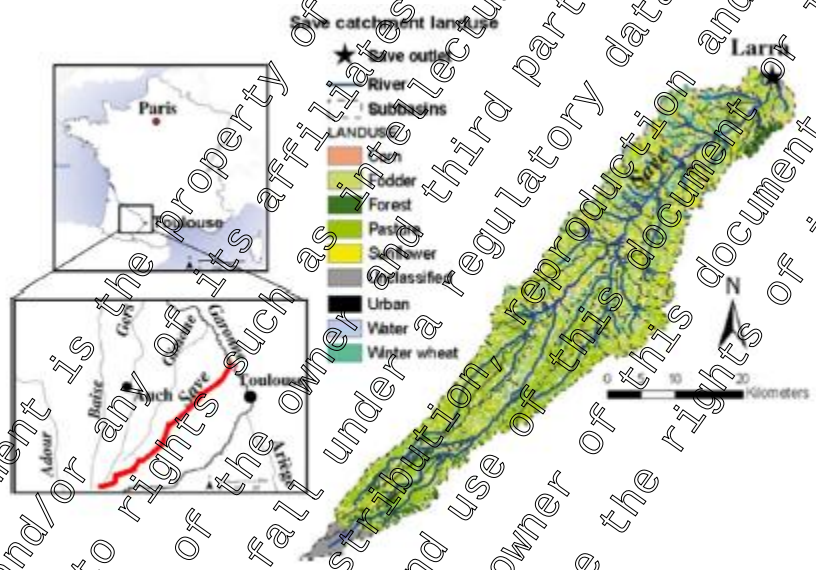
This article looks at the riverine transfer of both pesticides and trace elements which includes aclonifen from rainfall events and subsequently soil leaching and storm runoff. These periods are also very important to better understand their dynamics, particularly their different soil-river transfer pathways (surface runoff SR, subsurface runoff SSR, and groundwater flow GF). High-resolution monitoring of water discharge and water sampling were performed during a flood event that occurred in May 2010 in an agricultural catchment of SW France. The different stream flow components (SR, SSR, and GF) were assessed using two independent hydrograph separation methods: a hydrological approach based on Maillets formula (1905) for the recession period and a chemical approach based on physico-chemical tracers, TSM (Total Suspended Matter) for SR and PO_4^{3-} for GF. Both methods exhibited important contributions of SR (33 percent) and SSR (40 percent) to the total riverine pollutant transfers. This summary will concentrate on aclonifen. Moreover, significant relationships

were highlighted between the controlling factors (DOC, POC, and TSM) and SR, SSR, and GF contributions: DOC and the complexed pollutants were highly correlated to SSR while POC, TSM, and the adsorbed pollutants were linked to SR. The maximum concentration of aclonifen detected following a storm run off event was 0.139 µg L^{-1}.

I. MATERIALS AND METHODS

The Save River, located in the Coteaux de Gascogne region (south-west of France), originates from the piedmont of the Pyrénées Mountains. It is around 140 km in length and drains an agricultural catchment of 1110 km^2. Sampling was conducted at Larra station (43° 43′ 4″ N-01° 14′ 40″ E), ahead of the confluence of the Save River with the Garonne River as shown in Figure 7.5- 3.

Figure 7.5- 11: Location and land-use maps of the Save watershed with indication of the LARRA sampling station (star) at the outlet.



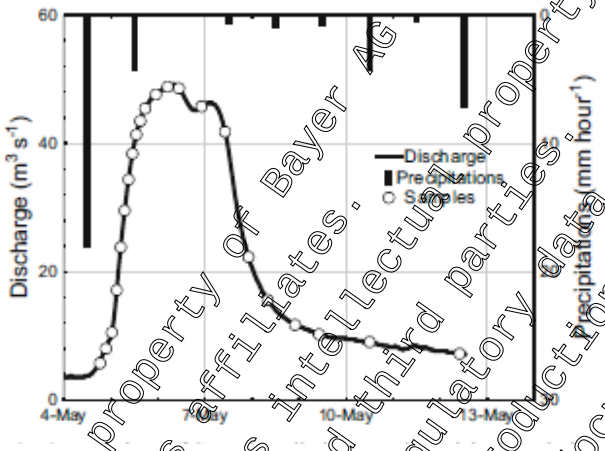
Ninety percent of the catchment is used for agriculture of which the lower part is devoted to intensive agriculture with mainly a 2-year crop rotation of sunflower and winter wheat. The climate is oceanic with an average annual precipitation of about 750 mm, mostly occurring in the form of thunderstorms. The Save River hydrological regime is mainly pluvial with a mean annual discharge of maximum discharge 6.1 m^3 s^{-1}. During the low water period, from June to September, river flow is sustained upstream by the Neste canal (1.3 $m^3 s^{-1}$). The catchment lies on detrital calcareous and clayey sediments. Calcic soils represent over 90% of the whole catchment with a clay content ranging from 40 to 50%. Non-calcic silty soils represent less than 10% of the soil in this area (50–60% silt). Storm events in this watershed lead to significant surface runoff and erosion. In fact, average mechanical and chemical erosion in this area are estimated to 27 and 70 t km^{-2} $year^{-1}$.

This study concerns a flood event which occurred from the 4th until the 13th of May 2010 which corresponded to the period of pesticide applications in the watershed. During the studied flood event, the instantaneous discharge increased from 3 m^3 s^{-1} to a peak storm flow of almost 50 m^3 s^{-1}. Twenty-two sampling points are represented on the storm hydrograph (In the unfiltered waters most of the pesticide concentrations increase during the flood event with increasing discharge although there were two magnitudes for pesticide concentration as shown in Figure 7.5- 7 for unfiltered waters.

Aclonifen reached a maximum concentration of 1.5 µg L-1. The concentration of aclonifen in the unfiltered water was higher after the peak discharge during the recession period than during the rising discharge, showing a hysteresis between the two hydrological periods. This means most of the aclonifen is mainly exported by the subsurface run-off Figure 7.5- 7 (a).

Figure 7.5- 7). Two hours separated the samples during the rising limb of the hydrograph whereas longer time separated samples during the recession limb. Waters samples were collected manually by standing on a bridge at the outlet of the drainage basin at the Larra station using a 10 L bucket.

Figure 7.5- 12: Variations of Save river discharge and precipitations during the storm event of May 2010. Sampling points are represented with the squares.



Two 2.5 L water samples were sampled at source and stored in glass bottles with Teflon-lined lids. One of the two bottles was first filtered through a 0.45 µm filter, dichloromethane was then added (1:40; v/v) to inhibit bacterial activity. Previously weighed filters were used to calculate the TSM content at each sampling point. To the second unfiltered sample, dichloromethane was added directly at sampling (1:40; v/v). The glass bottles were then stored in dark until analysed. Three filter blanks were taken at each sampling point were analysed and their pesticide concentrations were always under the detection limit (1 to 3 ng L^{-1}).

The concentrations of aclonifen was measured in both filtered and unfiltered waters. The water samples were first partitioned with dichloromethane. The dichloromethane fraction was then dried over anhydrous sodium sulphate and the remaining organic phase evaporated to dryness under vacuum. The dry residue was reconstituted in 2 mL of hexane. Any aclonifen present in the samples was identified by gas chromatography. The detection limit, based on a signal-to-noise ratio of 3, was estimated to 1 to 3 ng L^{-1} depending on the pesticide under investigation. Recoveries were determined using water samples spiked with the individual pesticides. Average recovery rates ranged from 91 to 102%.

In order to identify the distribution of pollutants between the dissolved and the particulate phases and estimate their availability, the partition coefficient (K_d in g L^{-1}) of each pollutant was calculated as follows:

$$K_d\left(g.L^{-1}\right) = \frac{C_{dissolved}\left(\mu g.L^{-1}\right)}{C_{particulate}\left(\mu g.g^{-1}\right)}$$

$C_{dissolved}$ is the concentration of the pollutant in the filtered water and $C_{particulate}$ is the concentration of the pollutant in the TSM. The concentration of the pesticide in particulate fraction in micrograms per gram is obtained by difference between the concentration (micrograms per litre) in unfiltered and filtered water, weighted by the TSM concentration in grams per litre.

II. RESULTS

Pesticides were detected in all filtered and unfiltered samples, the levels of aclonifen found in the samples is shown in (TABLE **7.5- 2**).

Table 7.5- 17: Characteristics and minimum, maximum and average of aclonifen content (µg L^{-1}, n = 22 samples) in unfiltered (UF) and filtered (F) water during the flash flood event of May 2010 in the Save River

Pesticide	Solubility (mg/ L^{-1})	Log *Kow*	Water	Min	Max	Average
Aclonifen	1.40	4.37	UF	0.000	0.217	0.057
			F	0.000	0.139	0.025

All the pesticides were classified into three groups depending on their concentrations in both filtered and unfiltered waters. Aclonifen was placed into Group II which contained the pesticides found at higher concentrations (0.1 to 0.3 µg L^{-1}). By studying the concentrations of the pesticide in both the unfiltered and filtered waters the concentration of pesticides exported by the suspended matter could be estimated, by difference. The degree of export depends on each molecule's characteristics, particularly, the water solubility (Sw mg L^{-1}) and the octanol-water partitioning coefficient. Most of the pesticides studied showed increasing K_d at the beginning of the flood. This K_d decreased gradually after. Thus, the presence of pesticides in soluble unstable phases was proven at the beginning of the flood event. The increasing K_d, despite the increase of TSM, showed not only a decrease in the number of available fixation sites on TSM due to interactions between particles and to probable obstruction, but also an increase in the DOC contents that are capable of complexing these pesticides.

Data Point:	KCA 7.5/11
Report Author:	[redacted]; [redacted]; [redacted] M. R.; [redacted]; [redacted]
Report Year:	2010
Report Title:	Gas/particle partitioning of currently used pesticides in the atmosphere of Strasbourg (France)
Report No:	M-457557-01-1
Document No:	M-457557-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	-
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

Atmospheric particle (P) and gas (G) phase samples were collected in Strasbourg, Alsace, France between April 17 and May 29, 2007 and analysed for a total of 71 pesticides in use at that time. 38 pesticides were detected, including aclonifen. The results were used to study the G/P partitioning of the pesticides. For aclonifen the regression was not significant., and thus no further meaningful correlations could be derived.

The concentration of aclonifen in the particle phase ranged from 0.64 to 3.67 ng m^{-3} and the average concentration was 1.99 ng m^{-3}. The concentration of aclonifen in the gaseous phase ranged from 0.82 to 2.22 ng m^{-3} and the average concentration was 1.29 ng m^{-3}.

I. MATERIALS AND METHODS

Air samples were collected in 2007 in the botanical garden of Strasbourg University, Strasbourg, Alsace, France and analysed for a large number of pesticides, including aclonifen. The site was located approximately 0.5 km from the town centre, 2 km from industrial zones and about 5 km from sites where pesticides were applied. None of the pesticides was used in the Botanical Garden. Particulate and gas phases were sampled simultaneously by using a high-volume sampler on a 48 h basis for the period from April 18 to May 29, 2007. The same samples are discussed in a separate publication (M-457521-01-1).

Particulate and gaseous samples were collected simultaneously for 48 h periods on average, using 30 cm diameter glass fibre filter and XAD-2 resin at a flow rate of 10 m^3/h. After sampling, filters and resins were stored in the dark at -20 °C for a maximum of 4 days until extraction. Pesticides were extracted with hexane : dichloromethane (1:1) by Soxhlet extraction for 20 hours, prior to concentration and analysis. Extraction of the pesticides from filter and resin was done separately to provide concentrations in particulate and gaseous phases. The concentration of aclonifen was measured by GC-MS/MS method

II. RESULTS

The measured concentrations of aclonifen in the total atmosphere are summarised in Table **7.5- 2**.

Table 7.5- 18: Gaseous and particle phase concentrations of aclonifen

Pesticide	Phase	Number of samples detected in	Concentration Range [ng m^{-3}]	Average Concentration ±95% CI [a] [ng m^{-3}]
Aclonifen	Particle phase	7 out of 10	0.64–3.67	1.99 ±1.22
	Gas phase	3 out of 10	0.82-2.22	1.29 ± 0.81

[a] The average and 95% confidence intervals listed were calculated from the arithmetic mean and standard deviation of samples with concentration in excess of the LOD

The gas-particle distribution of the detected pesticides was studied:

$$K_P = \frac{[P]}{[G][TSP]}$$

where P and G are the pesticide concentrations in the particle and the gas phase, respectively, and TSP is the total amount of suspended particles.

From the gas particle partitioning coefficient Kp it was possible to estimate the sorption of semi-volatile compounds to aerosols and thus show adsorption has an influence on the phase-partitioning process.

$$\log K_p = m_r \cdot \log P_L^0 + b_r$$

where P_L^0 is the subcooled liquid vapour pressure. The regression of log K_P against log P_L^0 provides the values for the slope m_r and the intercept b_r.

It was possible to calculate regressions for 27 compounds (see Table 7.5- 19) but for aclonifen the regression was not significant., and thus no further meaningful correlations could be derived.

Table 7.5- 19: Regression of Log K_P against log P_L^0 for aclonifen

Pesticide	Slope m_r	Intercept b_r	R^2	Number of datapoints	Level of significance
Aclonifen	-2.44	-9.44	0.97	3	NS

Data Point:	KCA 7.5/12
Report Author:	[redacted]
Report Year:	2013
Report Title:	Application of XAD-2 resin-based passive samplers and SPME-GC-MS/MS analysis for the monitoring of spatial and temporal variations of atmospheric pesticides in Luxembourg
Report No:	M-462592-01-1
Document No:	M-462592-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	No, not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

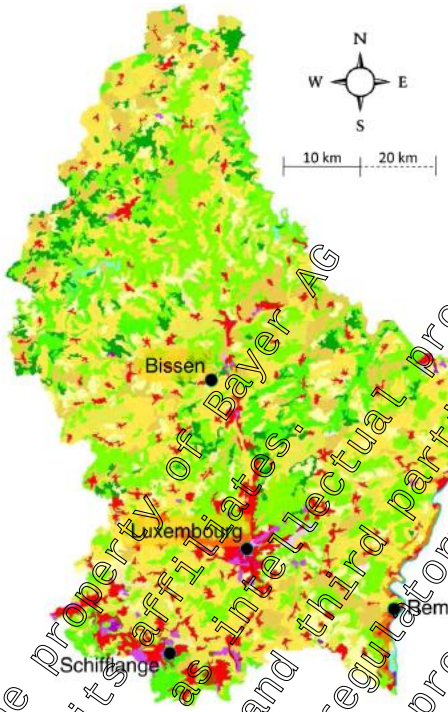
Executive Summary:

Air samples were monitored for a year at two rural and three urban sites within a 20 km radius in southern Luxembourg. The passive samplers were analysed for 50 pesticides, of which 22 were detected. However for 8 of them, including aclonifen, the concentration never exceeded the limit of quantification (LOQ).

I. MATERIALS AND METHODS

Air samples were monitored in 2008 and 2009 at five locations in southern Luxembourg for one year. for 50 pesticides, including aclonifen. The location of the sites is provided in in Figure 7.5- 1. The five locations were in an area with a radius of about 20 km. Bissen and Remich were rural sites with intense agricultural activity and about 3000 inhabitants each. Schifflange was an urban site (3500 inhabitants) as were the two sites in Luxembourg-City (85,000 inhabitants), one in the north of the town (Luxembourg 1) on the university campus in an area with a dense population and one in the city centre (Luxembourg 2) with dense circulation (16 000 cars/day) and a bus terminal.

Figure 7.5- 13: Sampling sites



Sampling was started on 20 June 2008 in Bissen, Luxembourg 1 and Remich, and on 26 September 2008 in Luxembourg 2 and Schifflange. Periods of exposure varied between 1 and 3 months. The sampling campaign was stopped on 26 June 2009 in Bissen, Luxembourg 1 and Remich and on 20 October 2009 in Luxembourg 2 and Schifflange. Full details of the sampling campaign are given below.

Table 7.5- 20: Sampling periods

Bissen	Remich	Schifflange	Luxembourg 1	Luxembourg 2
20.06. – 21.07.08	20.06. – 21.07.08	26.09. – 31.10.08	20.06. – 21.07.08	26.09. – 31.10.08
21.07. – 19.08.08	21.07. – 19.08.08	31.10. – 28.11.08	21.07. – 19.08.08	31.10. – 28.11.08
19.08. – 26.09.08	19.08. – 26.09.08	28.11. – 28.01.09	19.08. – 26.09.08	28.11. – 28.01.09
26.09. – 31.10.08	26.09. – 31.10.08	28.01. – 02.04.09	26.09. – 31.10.08	28.01. – 07.04.09
31.10. – 30.11.08	31.10. – 29.12.08	02.04. – 27.06.09	31.10. – 30.11.08	07.04. – 26.06.09
30.11. – 29.12.08	29.12. – 09.02.09	27.06. – 15.09.09	30.11. – 29.12.08	26.06. – 15.09.09
29.12. – 10.02.09	09.02. – 09.03.09	15.09. – 20.10.09	29.12. – 10.02.09	15.09. – 20.10.09
10.02. – 09.03.09	09.03. – 16.04.09	10.02. – 09.03.09		
09.03. – 16.04.09	16.04. – 22.05.09	09.03. – 16.04.09		
16.04. – 22.05.09	22.05. – 26.06.09	16.04. – 22.05.09		
22.05. – 26.06.09	22.05. – 26.06.09			

Air samples were collected using a passive sample filled in XAD-2 resin. Pesticides were extracted from the sampler with acetonitrile using an accelerated solvent extraction system. The extract was concentrated on a solid phase microextraction (SPME) fibre prior to analysis by GC-MS/MS.

II. RESULTS

The method including extraction, concentration, SPME extraction and GC-MS/MS analysis was evaluated in terms of sensitivity, quantification and detection limits. The results of the validation are presented in Table 7.5- 21.

Table 7.5- 21: Method validation data

Compound	R^2	Recovery		Coefficient of variation (intra-day)		Coefficient of variation (inter-day)		LOD	LOQ
		200 ng	800 ng	200 ng	800 ng	200 ng	800 ng	ng/sampler	
Aclonifen	0.989	45.80%	108.30%	8.90%	7.10%	12.30%	11.30%	25	153

An overall summary of the concentrations of aclonifen detected in air at the five urban and rural sampling sites for the year 2008 to 2009 are summarised in Table 7.5- 22.

Table 7.5- 22: Concentration of aclonifen at the sampling sites

Compound	Bissen	F.D.	Remich	F.D.	Luxem-bourg 1	F.D.	Luxem-bourg 2	F.D.	Schifflange	F.D.
Aclonifen	<LOQ	1/11	<LOQ	1/10	-		-	-	-	-

F.D. = Frequency of detection

22 out of 50 pesticides were detected at least once on the samplers, though for 8 of them including aclonifen the concentration never exceeded the limit of quantification (LOQ).

The following paper was found during the literature search and provides supplemental data regarding the dissipation of aclonifen and confirms the results from the GLP studies provided.

Data Point:	KCA 7.5/13
Report Author:	[redacted]; [redacted]
Report Year:	2007
Report Title:	The effect of citrus pulp amendment on sunflower production and the dissipation of the herbicide aclonifen.
Report No:	M-547563-01-1
Document No:	M-547563-01-1
Guideline(s) followed in study:	not applicable
Deviations from current test guideline:	--
Previous evaluation:	
GLP/Officially recognised testing facilities:	Not not conducted under GLP/Officially recognised testing facilities
Acceptability/Reliability:	

Executive Summary:

The research evaluated the effects that amendment with 3 kg m^{-2} and 9 kg m^{-2} of citrus pulp had both on the production of sunflowers and the dissipation of the aclonifen herbicide. At the same time any eventual effect of the use of the herbicide on sunflower production was verified. The use of the citrus pulp determined an increase in the height of the plants, the diameter of the flower-heads and their achenes production and a reduction in the sterile zone. The effect of amending was not proportional to the quantity of citrus pulp added: in fact, the maximum agronomic efficiency was reached with the lowest quantity of amendment (97 kg of achenes per ton of citrus pulp used, as against the 53 kg obtained with the higher quantity). The herbicide had no effect on sunflower production. The dissipation of aclonifen was not influenced by the addition of citrus pulp in field conditions but in laboratory conditions a faster degradation was found. The mean half-life time was 14 days in the field and 30 and 13 days respectively, in untreated soil and soil treated with citrus pulp, in laboratory conditions.

I. MATERIALS AND METHODS

The dissipation of aclonifen was studied in a sandy loam soil with the following characteristics:

Parameter	Results
Geographic Location	-
Texture Class	Sandy loam
pH	8.0
Organic matter (%)	0.78%
Cation exchange capacity (meq/100 g)	12.3
Sand (>20 µm) % Silt (2 - 20 µm) % Clay (<2 µm) %	- - 11.7
Maximum water holding capacity (MWHC)	35.3%

The citrus pulp came from a citrus fruit processing factory in Caltagirone, Catania, Italy and was air dried for six months before use.

Field studies

The following six soil conditions were used in the field studies:

1		2		3	
A	A1	B	B1	C	C1
Unamended soil without chemical weed control	Unamended soil with chemical weed control	Soil amended with 3 kg m^{-2} of citrus pulp without chemical weed control	Soil amended with 3 kg m^{-2} of citrus pulp with chemical weed control	Soil amended with 9 kg m^{-2} of citrus pulp without chemical weed control	Soil amended with 9 kg m^{-2} of citrus pulp with chemical weed control

The experimental test was carried out in 0.9 m x 0.9 m x 0.5 m lysimeter maintained under field conditions. The citrus pulp was broken into 0.5 to 10 mm diameter pieces and added to the lysimeters which were then covered with 0 - 30 cm of soil in October 2005.

Sunflowers were sown on the 7 April 2005 at a density of 6 plants per m^2. The plants were regularly watered during cultivation. Technical aclonifen (Challenge 49% a.i) was applied pre-emergence at a recommended rate of 1.0 kg ha^{-1}. Five soil cores (25.0 cm length, 5.8 cm diameter) were taken from each plot using a continuous sampling tube which fits into an electrical hollow- stem auger column, 0, 7, 16, 28, 60 and 120 days after the treatment. Samples for each plot were mixed, sieved through a 2 mm mesh sieve and immediately frozen at -25 °C until analysis.

Laboratory studies

Soil samples were taken to a depth of 0-25 cm from plots A1 and C1 after amendment and prior to application of the herbicide to be used in the laboratory experiments. After collection, the soils were dried to 10% water content (w/w), sieved to obtain a < 2 mm fraction and stored at 4 °C until use. Aclonifen was added to soils at 4 μg^{-1} dry soil as an aqueous solution. The soils were then incubated in the dark at 25 °C at a soil moisture content of 50% of WHC. 0.05 N NaOH was used to trap any carbon dioxide produced by the respiration process. Three individual incubation systems were removed at 0, 7, 14, 28, 56, and 91 days and analysed for aclonifen residual concentration.

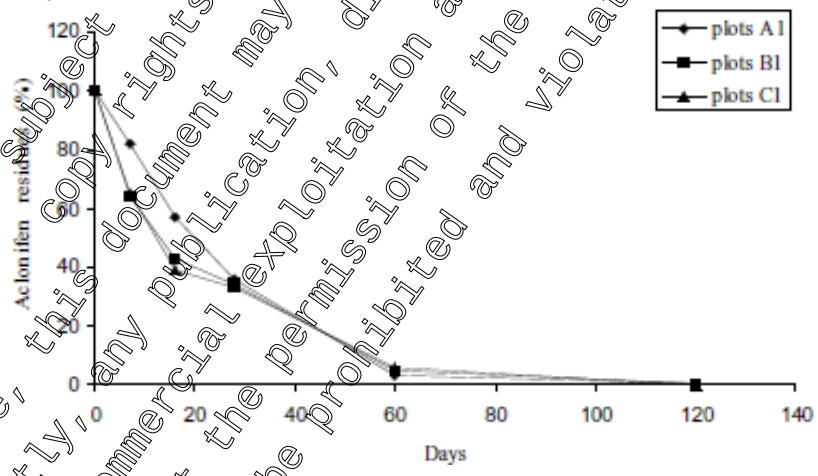
Each soil sample was extracted 3x with water:methanol 50:50 (v/v) by shaking for 30 minutes, then centrifuged for 10 minutes at 3000 rpm. The supernatants were combined in a 500 ml separatory funnel and the combined extracts partitioned with dichloromethane (3 x 50 ml); the organic phase was dried by filtration over anhydrous sodium sulphate and evaporated to dryness in a rotary evaporator. The residue was dissolved in acetonitrile:water acidified to pH 3 with H_3PO_4 70:30 (v/v). The concentration of aclonifen in the soil extracts was determined by reverse phase HPLC. The recovery was 94% ± 2.

II. RESULTS

The use of the citrus pulp determined an increase in the height of the plants, the diameter of the flower-heads and their achenes production and a reduction in the sterile zone. The effect of amending was not proportional to the quantity of citrus pulp added: in fact, the maximum agronomic efficiency was reached with the lowest quantity of amendment (97 kg of achenes per ton of citrus pulp used, as against the 53 kg obtained with the higher quantity).

The dissipation of aclonifen in the field samples is summarised in Figure 7.5- 3. No differences in the rate of dissipation of the herbicide were found between the three experimental conditions. The mean DT_{50} was 14 days and the mean quantity found after 120 days from application was 0.3% of the initial amount.

Figure 7.5- 14: Degradation kinetics of aclonifen in amended and unamended soils: field test. Plots A1 = unamended soil; plots B1 = soil amended with 3 kg m^{-2} of citrus pulp; plots C1 = soil amended with 9 kg m^{-2} of citrus pulp



The dissipation of aclonifen in the laboratory samples is summarised in Figure 7.5- 15. The DT_{50} of aclonifen were 30 and 13 days in soils A and C respectively. When compared to the field tests, the laboratory tests demonstrated a greater persistence of aclonifen in soil A while in soil C the half-life time appeared similar. The addition of organic matter probably, meant that the soil microflora adapted better to the laboratory conditions.

Figure 7.5- 15: Degradation kinetics of aclonifen in amended and unamended soils: field test. Plots A1 = unamended soil; plots B1 = soil amended with 3 kg m^{-2} of citrus pulp; plots C1 = soil amended with 9 kg m^{-2} of citrus pulp