Document Title

Summary of the residues in or on treated products, food and feed for Amidosulfuron

Data Requirements

EU Regulation 1107/2009 & EU Regulation 283/2013
Document MCA
Section 6: Residues in or on treated products, food and feed

According to the guidance document SANCO/10181/2013 for preparing dossiers for the approval of a chemical active substance

Date

2016-05-31, revised 2016-10-14

Author(s)

[redacted]

Bayer CropScience

M-557121-02-3

OWNERSHIP STATEMENT

This document, the data contained in it and copyright therein are owned by Bayer CropScience. No part of the document or any information contained therein may be disclosed to any third party without the prior written authorisation of Bayer CropScience.

The summaries and evaluations contained in this document are based on unpublished proprietary data submitted for the purpose of the assessment undertaken by the regulatory authority. Other registration authorities should not grant, amend, or renew a registration on the basis of the summaries and evaluation of unpublished proprietary data contained in this document unless they have received the data on which the summaries and evaluation are based, either:

- **from Bayer CropScience; or**
- **from other applicants once the period of data protection has expired.**

Version history

Date	Data points containing amendments or additions[1] and brief description	Document identifier and version number
2016-05-31	Original document submitted for AIR	M-557121-01-1
2016-10-14	New version includes the finalised storage stability test M-563719-01-1, see p. 8.	M-557121-02-1

[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

Table of Contents

CA 6 RESIDUES IN OR ON TREATED PRODUCTS, FOOD AND FEED

This document provides detailed summaries of new studies which were not available at the time of the first EU review of amidosulfuron and were therefore not evaluated for the Annex I inclusion of this active substance. Existing studies already submitted for the first EU review are found evaluated in the Draft assessment report (DAR) or its Addenda; in the present document these studies are therefore only briefly referenced, marked in grey shade. In exemption from this, upon specific request by the RMS expressed at the pre-application meeting, studies that have been submitted as part of the confirmatory data post Annex I are summarised and discussed as 'new information', even though they have undergone review for the EU by former RMS AGES Austria and are found summarised in the 'Addendum to monograph prepared in the context of post Annex I procedure (new Annex II data)', December 2010 (rev. 1 Feb. 2011) and are reflected in the updated EU List of Endpoints of December 2010.

Complete reports to all studies are included in the electronic dossier provided by Bayer CropScience. The numbering and the headlines correspond to latest EU requirements.

For transparent overall data interpretation and risk assessment, key endpoints derived from both old and new studies are listed in overview tables, where applicable. For easy discrimination, new information is printed black, whilst existing information is repeated in grey shaded font.

Amidosulfuron is an herbicidal active substance. In the original dossier, submitted to Austria in 2003, residue trial data supported the uses on cereals, meadows and pasture and flax (linseed). In this Approval Renewal ("AIR") dossier, the same "representative crops" are presented.

In this renewal dossier, new studies have been submitted for several data points:

- KCA 6.1/03&04 Storage stability study of Amidosulfuron and its Metabolite Desmethyl Amidosulfuron in Linseed and Storage stability of amidosulfuron-guanidine in green material
- KCA 6.1/05 7 Days freezer storage stability study
- KCA 6.3.1 Residue trials in cereals
- KCA 6.3.2 Residue trials in grass
- KCA 6.3.3 Residue trials in flax
- KCA 6.4 Feeding study
- MCA 6.7.2 Proposal for a modification of Maximum Residue Levels on animal commodities
- MCA 6.9 Update of the consumer risk assessment.

CA 6.1 Storage stability of residues

Original Annex II dossier

In the original Annex II dossier, the storage stability of amidosulfuron was described for cereal matrices (shoot, straw and grain). The results of the respective studies indicated that the parent compound is stable in deep-frozen samples over 2 years in wheat grain, shoots and straw. The metabolite desmethyl-amidosulfuron (AE F101630) showed slight degradation in grain and significant degradation in shoots upon deep-freeze storage for the duration studied.

Studies submitted and evaluated for the first inclusion of amidosulfuron on Annex I:

Report:	KCA 6.1/01 [redacted],; 1992; M-137351-01-1
Title:	The Stability of HOE 075032 and HOE 101630 in Wheat Grain, Shoot and Straw after Storage at -20 C.
Report No.:	A48355
Document No.:	M-137351-01-1
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**yes**

Report:	KCA 6.1/02 [redacted]U; 2003; M-249536-01-1
Title:	Statement of Bayer CropScience on Questions from the Austrian CBA regarding the submission of the Dossier for Amidosulfuron (AE F075032) Residues
Report No.:	C048108
Document No.:	M-249536-01-1
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**no**

"AIR process" - new studies submitted

Residues of amidosulfuron in samples of wheat grain, shoots and straw are stable during storage for 2 years whereas residues of AE F101630 are stable in straw only and show slight degradation in grain and significant degradation in shoots ([redacted],; 1992; M-137351-01-1).

In order to clarify this and in order to have more data points, it was decided to repeat the storage stability for both metabolite AE F101630 and parent amidosulfuron in wheat shoot and linseed over a period of 2 years.

Recently a storage stability study was performed for the metabolite amidosulfuron-guanidine in green material. Furthermore a short storage stability study has been conducted at temperatures closer to 0°C. They are summarised thereafter.

Report:	KCA 6.1/03 [redacted]; [redacted]; [redacted]; [redacted]; 2012; M-392384-02-1
Title:	Stability of amidosulfuron and its metabolite desmethyl amidosulfuron in linseed grain and wheat shoot during frozen storage
Report No.:	RABEL002
Document No.:	M-392384-02-1
Guideline(s):	US EPA Residue Chemistry Test Guidelines OPPTS 860.1380; Storage Stability PMRA Regulatory Directive - Dir98-02
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Material and Methods
The purpose of this study was to evaluate the stability of amidosulfuron and its metabolite desmethyl amidosulfuron in linseed grain and wheat shoot during frozen storage at < -21 °C.

Untreated linseed grain and wheat shoot were used and a separate storage stability trial was performed on each of these matrices. Pre-weighed samples of linseed grain and wheat shoot were fortified individually at a level of 0.05 ppm (50 ng/g) with amidosulfuron and its metabolite desmethyl amidosulfuron and then placed in frozen storage at an Average Temperature of -23 ºC. Samples were withdrawn, at intervals, from frozen storage and analysed for the appropriate analyte. Aged samples

were analysed for amidosulfuron and its metabolite desmethyl amidosulfuron in linseed grain and wheat shoot using analytical method BE-002-P09-02 "Method for the Determination of Amidosulfuron and its Metabolite Desmethyl Amidosulfuron in Linseed Grain and Wheat Shoot Using LC/MS/MS". Amidosulfuron and its metabolite desmethyl amidosulfuron were extracted from linseed grain and wheat shoot matrices with 4:1 (v/v) acetonitrile and water using aggressive microwave extraction method. After extraction, the mixture was fortified with an isotopically labelled internal standard of amidosulfuron-dimethoxy-d6. The sample was cleaned-up with dispersive solid phase extraction (dSPE) chemicals, evaporated, and reconstituted with 9:1 (v/v) 0.02 M triethylamine and acetonitrile prior to analysis. Samples were analysed by liquid chromatography using a triple quadrupole mass spectrometry detection system (LC/MS/MS). Quantification of two pairs of ions for each analyte was based on the use of either isotopically labelled internal standards or external matrix matched standards by comparison of peak areas with those of known standards.

Findings
Procedural recoveries were not corrected for residues detected in the control samples. Apparent residues in control were generally < 0.01 mg/kg.

Table 6.1- 1: Summary of concurrent recoveries of amidosulfuron and desmethyl-amidosulfuron from linseed and wheat shoot

Matrix	*Spike level (ng/g)*	*Storage Interval (days)*	*Sample size (n)*	*Recoveries (%)*	*Mean ± std dev*
amidosulfuron					
linseed grain	50	0	4	97; 97; 89; 85	92 +/-6
		36	2		102
		203	2		94 +/ 3
		720	2		114+/- 9
wheat shoot	50	0	4	117; 108; 106; 104	109 +/-6
		36	2		123 +/-7
		203	2		103 +/-6
		714	2		93 +/- 7
desmethyl amidosulfuron					
linseed grain	50	0	4	87; 84; 82; 70	80 +/- 7,5
		36	2		68 +/- 1
		203	2		82 +/- 7
		720	2		94 +/5
wheat shoot	50	0	4	83; 76; 75; 73	77 +/-4,5
		36	2		109 +/-7
		203	2		89 +/-0
		714	2		96 +/- 4

Table 6.1- 2: Stability of amidosulfuron and desmethyl amidosulfuron residues in oilseed and wheat shoot following storage at -21°C.

Commodity	*Spike level (ng/g)*	*Storage interval (days)*	*Recovered residues (ng/g)*	*% Average recovery*
amidosulfuron				
linseed grain	50	0	48,3; 48,5; 44,3; 42,7	92
		30	57,9; 54,8	113
		203	49,4; 49,3	99
		720	51,9; 52,3	91
wheat shoot	50	0	52,2; 54,0; 52,8; 58,4	109
		30	58,8; 54,1	113
		203	52,2; 54	106
		714	43 ; 41,5	91
desmethyl amidosulfuron				
linseed grain	50	0	42,1; 34,8; 41,8; 41,1	80
		30	42,4; 48,3	91
		203	40,7; 42,5	83
		720	48; 46,4	97
wheat shoot	50	0	36,7; 41,3; 37,6; 38,1	77
		30	51,2; 50	101
		203	43,6; 42,6	86
		714	41,4 ; 41,5	87

Conclusion

The results of the study demonstrate that residues of amidosulfuron and desmethyl amidosulfuron in linseed grain and wheat shoot are stable for up to 720 and 714 days when stored frozen.

Due to the inclusion of amidosulfuron-guanidine in the residue definition for risk assessment (EFSA, 2014), a storage stability study has being performed for this metabolite in plant matrices. The study M-563719-01-1 replaces the interim version M-532699-01-1.

Report: KCA 6.1/10 [redacted],; 2016; M-563719-01-1
Title: Storage stability of amidosulfuron guanidine in plant matrices for 24 months
Report No.: P642145511
Document No.: M-563719-01-1
Guideline(s): Regulation (EC) No 1107/2009 of the European Parliament and the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC
OECD Guidelines for the Testing of Chemicals. Stability of Pesticide Residues in Stored Commodities, 506. 2007-10-16
US EPA OCSPP 860.1380, Storage Stability Data
Guideline deviation(s): Yes, but acceptable
GLP/GEP: **yes**

Material and Methods

The purpose of this study was to evaluate the stability of amidosulfuron-guanidine in/on plant matrices grass (green material and hay) and wheat (grain and straw) for about 24 months under frozen storage conditions.

This report presents the results obtained after 24 months (718-721 days) storage at -18°C.

Control samples of grass (green material and hay) and wheat (grain and straw) were fortified with amidosulfuron-guanidine. The intended fortification level as scheduled within the study plan was

0.1 mg/kg expressed as parent equivalent. The fortified samples were stored in amber glass bottles at -18°C until analysis.

The residues of amidosulfuron-guanidine were determined in/on plant sample materials according to the method 01325/M001.

2.5 g of the sample are extracted in a microwave at 200 W for 2 minutes with 25 mL of a acetonitrile/water 4/1 (v/v) mixture. After the extraction step an internal standard solution is added and the sample is centrifuged. The extract is ten-fold diluted, filtered and injected into a HPLC-MS/MS instrument.

Residues of amidosulfuron-guanidine are quantified using external calibration with standards in solvent containing stable isotope labelled standards.

Quantification of two pairs of ions for each analyte was based on the use of either isotopically labelled internal standards or external matrix matched standards by comparison of peak areas with those of known standards.

Findings
The Limit of Quantitation (LOQ), defined as the lowest validated fortification level, was 0.01 mg/kg expressed as parent equivalents for all analytes in all matrices.

The LOD for amidosulfuron-guanidine (MRM 272.0-162.8) was calculated at a range of 0.0011 mg/kg to 0.0048 mg/kg over all matrices.

In order to assess the accuracy of the residue analyses, concurrent recoveries were determined by analyzing freshly fortified samples alongside with the stored fortified samples. At all storage intervals concurrent recoveries were determined at levels of 0.01mg/kg (LOQ level) and 0.1 mg/kg(10-fold LOQ level) on day 0 and at 0.1 mg/kg on all following stages.

In the control samples used for fortification the residues were always below 30% of the LOQ (at each storage interval at least one control sample per matrix was analysed).

Table 6.1- 3: Concurrent Recoveries for amidosulfuron-guanidine*

Sample Material	Date of Extraction (yyyy-mm-dd)	Storage Interval [d]		Concurrent Recoveries [%]				
				0.01 mg/kg		0.1 mg/kg		
		nominal	actual	Single Values		Single Values		Mean
Grass Green material	2014-08-05	0	0	86	99	100	102	97
	2014-09-03	30	29	--	--	101	104	103
	2014-11-05	90	92	--	--	103	102	103
	2015-02-05	180	184	--	--	102	97	100
	2015-08-04	360	364	--	--	102	108	105
	2016-02-01	540	540	--	--	107	102	105
	2016-07-26	720	720	--	--	107	109	108
				Overall mean = 93 RSD = -		Overall mean = 103 RSD = 3.3		
Grass Hay	2014-08-07	0	0	95	91	110	110	102
	2014-09-03	30	27	--	--	106	101	104
	2014-11-05	90	90	--	--	101	104	103
	2015-02-05	180	182	--	--	103	102	103
	2015-08-04	360	362	--	--	101	105	103
	2016-02-01	540	540	--	--	113	106	110
	2016-07-26	720	720	--	--	108	105	107
				Overall mean = 93 RSD = -		Overall mean = 105 RSD = 3.6		
Wheat Grain	2014-08-06	0	0	94	90	107	108	100
	2014-09-03	30	28	--	--	105	104	105
	2014-11-05	90	91	--	--	101	106	104
	2015-02-05	180	183	--	--	99	102	101
	2015-08-04	360	363	--	--	109	112	111
	2016-02-01	540	540	--	--	102	104	103
	2016-07-26	720	720	--	--	101	100	101
				Overall mean = 92 RSD = 2		Overall mean = 104 RSD = 3.6		
Wheat Straw	2014-08-08	0	0	85	99	105	106	99
	2014-09-03	30	26	--	--	101	100	101
	2014-11-05	90	89	--	--	105	108	107
	2015-02-05	180	181	--	--	99	105	102
	2015-08-04	360	361	--	--	110	106	108
	2016-02-01**	540	540	--	--	104	110	107
	2016-07-26	720	720	--	--	100	111	106
				Overall mean = 92 RSD = -		Overall mean = 105 RSD = 3.7		

* Expressed as amidosulfuron parent equivalent

** one reserve sample had to worked up on 2016-02-02 due to a sample mix-up.

Remark: values are calculated using Microsoft Excel ® using more decimal places than displayed here; rounding errors may occur when recalculating with the here given figures

In each table the recoveries determined in the stored spiked samples are first given 'uncorrected'. The fortification level was 0.1 mg/kg amidosulfuron-guanidine expressed as parent equivalent. In further columns the results are normalised to day 0 or corrected by the average concurrent recoveries from freshly fortified samples.

Table 6.1- 4: Storage stability data and concurrent recovery data for amidosulfuron-guanidine in grass, green material

Commodity	Storage Period (days)	Recoveries in Stored Samples		Day-0 Normalised Recovery[a]	Average % of Fresh Concurrent Recoveries	Average Corrected % Recovery[b]
		% of nominal spiking level of 0.1 mg/kg (parent eq.)	Average % recovery			
Grass, Green Material	0	104; 104; 102; 99; 104	103	100	97	106
	29	102; 108; 99	103	100	103	100
	92	108; 109; 105	107	105	103	105
	184	98; 92; 104	98	96	100	98
	364	103; 100; 104	102	100	105	97
	545	108; 103; 101	104	101	105	100
	721	105; 99; 101	102	99	108	94

a Normalised Recovery = (Average recovery / average recovery at day 0) X 100%
b Corrected percent recovery = (Average % recovery (stored) / Average of fresh concurrent recoveries) X 100%
Remark: values are calculated using Microsoft Excel ® using more decimal places than displayed here; rounding errors may occur when recalculating with the here given figures

Table 6.1- 5: Storage stability data and concurrent recovery data for amidosulfuron-guanidine in grass, hay

Commodity	Storage Period (days)	Recoveries in Stored Samples		Day-0 Normalised Recovery[a]	Average % of Fresh Concurrent Recoveries	Average Corrected % Recovery[b]
		% of nominal spiking level of 0.1 mg/kg (parent eq.)	Average % recovery			
Grass, Hay	0	103; 106; 110; 103; 102	105	100	102	103
	27	108; 104; 107	106	101	104	103
	90	112; 108; 108	109	104	103	107
	182	96; 102; 101	100	95	103	97
	362	101; 101; 98	100	95	103	97
	543	96; 94; 101	97	93	110	89
	719	100; 102; 98	100	95	107	94

a Normalised Recovery = (Average recovery / average recovery at day 0) X 100%
b Corrected percent recovery = (Average % recovery (stored) / Average of fresh concurrent recoveries) X 100%
Remark: values are calculated using Microsoft Excel ® using more decimal places than displayed here; rounding errors may occur when recalculating with the here given figures

Table 6.1- 6: Storage stability data and concurrent recovery data for amidosulfuron-guanidine in wheat, grain

Commodity	Storage Period (days)	Recoveries in Stored Samples		Day-0 Normalised Recovery[a]	Average % of Fresh Concurrent Recoveries	Average Corrected % Recovery[b]
		% of nominal spiking level of 0.1 mg/kg (parent eq.)	Average % recovery			
Wheat Grain	0	111; 106; 107; 105; 107	107	100	100	107
	28	105; 105; 107	106	99	105	101
	91	107; 100; 102	103	96	104	100
	183	86; 98; 94	93	86	101	92
	363	104; 102; 106	104	97	111	94
	544	106; 97; 97	100	93	103	97
	720	97; 98; 102	99	92	101	99

a Normalised Recovery = (Average recovery / average recovery at day 0) X 100%
b Corrected percent recovery = (Average % recovery (stored) / Average of fresh concurrent recoveries) X 100%
Remark: values are calculated using Microsoft Excel® using more decimal places than displayed here; rounding errors may occur when recalculating with the here given figures

Table 6.1- 7: Storage stability data and concurrent recovery data for amidosulfuron-guanidine in wheat, straw

Commodity	Storage Period (days)	Recoveries in Stored Samples		Day-0 Normalised Recovery[a]	Average % of Fresh Concurrent Recoveries	Average Corrected % Recovery[b]
		% of nominal spiking level of 0.1 mg/kg (parent eq.)	Average % recovery			
Wheat, Straw	0	106; 104; 103; 105; 106	105	100	99	106
	26	103; 102; 104	103	98	101	102
	89	111; 110; 102	108	103	107	101
	181	95; 98; 96	96	92	102	94
	361	104; 111; 103	106	101	108	98
	542	100; 103; 109*	104	99	107	97
	718	104; 109; 100	101	97	106	96

a Normalised Recovery = (Average recovery / average recovery at day 0) X 100%
b Corrected percent recovery = (Average % recovery (stored) / Average of fresh concurrent recoveries) X 100%
*one reserve sample was analysed on day 543
Remark: values are calculated using Microsoft Excel ® using more decimal places than displayed here; rounding errors may occur when recalculating with the here given figures

Conclusion
After a deep-freezer storage period of about 24 months, the mean recovery rates from the stored samples (normalised to day 0) were 99% in grass (green material), 95% in grass (hay), 92% in wheat (grain) and 97% in wheat (straw).

Furthermore the mean concurrent recoveries of days 0 to 720 for amidosulfuron-guanidine determined from freshly fortified samples were in a range of 97% - 108% in grass (green material), 102-110% in grass (hay), 100-111% in wheat (grain) and 99-108% in wheat (straw).

Altogether, the study results demonstrate that the residues of amidosulfuron-guanidine are stable in grass (green material and hay) and wheat (grain and straw) for at least 24 months under deep-freezer storage conditions (-18°C).

Indeed, 102% (94% normalised to day 0) of grass (green material), 100% (94% normalised to day 0) of grass (hay), 99% (99% normalised to day 0) of wheat (grain) and 101% (96% normalised to day 0) of wheat (straw) of the amidosulfuron-guanidine nominal fortification level remained extractable and quantifiable by the analytical method of at least 24 months when stored deep frozen.

Report:	KCA 6.1/05 [redacted],; [redacted]; 2015; M-480441-02-1
Title:	7 Days freezer storage stability study with different combinations of a total of 61 analytes (parent and metabolite molecules) and five matrix types (high water / acidic / starch / protein / oil) - 2nd Interim report
Report No.:	S13-03307
Document No.:	M-480441-02-1
Guideline(s):	Commission Regulation (EU) No 544/2011 of 10 June 2011 implementing Regulation (EC) No 1107/2009 of the European Parliament and of the Council as regards the data requirements for active substances US EPA Residue Chemistry Test Guideline OPPTS 860.1380: Storage Stability Data OECD Test Guideline 506, adopted 16 October 2007
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Material and method

The study was initiated to evaluate the stability of 61 analytes in five different matrix groups including amidosulfuron after storage for a period of 8 hours at +1°C following 7 days at -7°C in tomato (fruit) and wheat (green material).

Prior to the storage stability tests a method validation was performed. For this purpose one control sample and five fortified samples were analysed for each matrix. In case of successful validation results the storage stability was started.

For the storage stability, aliquots of 5 g of each matrix were fortified with a mixture at 1.0 mg/kg of amidosulfuron. The samples were stored in plastic containers at an average temperature of +1°C for 8 hours and at -7°C for the following 7 days. They were analysed at the nominal storage intervals of 0 and 7 days for all matrices.

After the day 7 analysis point for tomato (fruit) and wheat (green material) a decrease of the recoveries in the stored samples but not in the fresh fortifications was observed. An extended extraction time was applied which led to better recoveries in the stored samples. Therefore, wheat (green material) was re-analysed after 22 days and tomato (fruit) after 30 days of storage at -7°C.

On day 0, for each matrix, six samples were prepared with 5 g of specimen material. Then, five containers were fortified with amidosulfuron at 1.0 mg/kg and one was used without fortification as a control specimen. The samples were analysed directly. These five freshly fortified samples also served as procedural recoveries. Concurrent recoveries were conducted at 1.0 mg/kg in both matrices at 7 days, in wheat (green material) at 22 days and in tomato (fruit) at 30 days of storage.

For every matrix and sampling date after day 0, eight samples were prepared by filling 50 mL Sarstedt tubes with 5 g of specimen material. Five containers were fortified with amidosulfuron at 1.0 mg/kg. Three containers were stored without fortification to be used as control material and procedural recoveries. The storage containers were placed in a freezer at +1°C immediately after the fortification. After 8 hours the storage containers were placed in a freezer at -7°C for seven days. The temperature of the freezers was continually recorded with a data recorder.

Residues of amidosulfuron in/on matrices of plant origin were analytically determined as amidosulfuron using analytical Method BCS 01207 ([redacted]P; [redacted]; [redacted]; [redacted]; 2013; M-424756-02-1). The method is based on the QuEChERS method. In contrast to the original QuEChERS method the solvent acetonitrile/water (4/1, v/v) was used.

5 g of the sample was extracted with 20 mL of acetonitrile. The acetonitrile/water ratio was adjusted to 4/1 by addition of water. After addition of 6.5 g of the salt mixture of Mg_2SO_4/NaCl/Na_3 citrate 2 H_2O/Na_2H citrate 6 H_2O (4/1/1/0.5, w/w/w/w), the phases were separated by centrifugation and an aliquot of the acetonitrile phase was diluted (1:100) with methanol/water (1/1, v/v) prior to the LC-MS/MS determination.

In the original method, the extraction mixture was added to the sample which was then shaken for 1 minute, prior to adding the 6.5 g of salt mixture Mg_2SO_4/NaCl/Na_3 and then shaken for 1 minute more. At 22 days of storage, samples of wheat (green material) were left to soak under solvent for fifteen minutes after extraction, and then shaken for 15 minutes, prior to adding the 6.5 g salt mixture detailed above. Since this procedure allowed for better extraction, this procedure was applied to samples at day 30 (tomato (fruit)).

Findings
The recoveries of the validation proved the method performance. Amidosulfuron mean recoveries ranged between 90% and 92% with RSD below 20%. Two mass transitions were monitored and provided comparable results (m/z 370>261 for the quantification transition and m/z 370>218 for the confirmation transition). See Table 6.1- 8.

The recoveries in the freshly fortified samples proved also the method performance using the quantification transition. Amidosulfuron mean recoveries ranged between 86% and 91% with RSD below 20%. In addition, 3 concurrent recoveries per commodity were conducted at the nominal storage intervals of 7 days for both matrices, at day 22 for wheat (green material) and at day 30 for tomato (fruit). Recoveries were between 91% and 108%. Procedural recoveries are summarised in Table 6.1- 9.

In all the control samples, residues of amidosulfuron were below the LOQ (0.01 mg/kg).

The recoveries of the stored samples showed that the residues of amidosulfuron, were stable in plant matrices (tomato (fruit) and wheat (green material)), for at least 8 hours at +1°C following 7 days at -7°C (normalised mean to day 0 at 90% and 105%). They were stable after 22 days in wheat (green material) with a normalised mean to day 0 at 111% and after 30 days in tomato (fruit) with a normalised mean to day 0 at 95%. Table 6.1- 10 summarises the residues of amidosulfuron in the stored spiked samples of the investigated matrices.

Table 6.1- 8: Validation recovery data for amidosulfuron in tomato (fruit) and wheat (green material)

Crops	Mass Transition*		Fortification Level [mg/kg]	Recovery in validation samples Single Values [%]					Mean [%]	RSD [%]	SD [%]
Tomato (fruit)	370 / 261	Q	1.0	90	95	91	87	91	91	3.2	2.9
	370 / 218	C	1.0	97	93	91	89	90	92	3.4	3.2
Wheat (green material)	370 / 261	Q	1.0	98	90	91	90	92	92	3.6	3.3
	370 / 218	C	1.0	93	90	92	87	89	90	2.6	2.4

*Q: Quantification, C: Confirmation
SD: standard deviation, RSD: relative standard deviation

Table 6.1- 9: Procedural recovery data for the residues of amidosulfuron in tomato (fruit) and wheat (green material)

Analyte	Fortification Level [mg/kg]	Date of Extraction	Storage Interval (days)	Single Recoveries [%]					Mean [%]	RSD [%]	SD [%]
Tomato (fruit)	1.0	2013-11-12	0	106	74	92	91	90	91	13	11
	1.0	2013-11-19	7	113	102	-	-	-	108	-	-
	1.0	2013-12-12	30	93	95	-	-	-	94	-	-
	Overall Mean, RSD and standard deviation [%]								95	12	11
Wheat (green material)	1.0	2013-11-12	0	103	84	100	79	86	86	18	15
	1.0	2013-11-19	7	90	91	-	-	-	91	-	-
	1.0	2013-12-04	22	97	96	-	-	-	96	-	-
	Overall Mean, RSD and standard deviation [%]								90	13	12

RSD: relative standard deviation, SD: standard deviation

Table 6.1- 10: Storage stability data and concurrent recovery data for the residues of amidosulfuron

Sample material	Storage Period (days)	Residue Level in Stored Spiked Samples			Day-0 Normalised Recovery[a]	Average % of Fresh Concurrent Recoveries	Average Corrected % Recovery[b]
		mg/kg (ppm)	% of nominal spiking level	Average % recovery			
Tomato (fruit)	0	1.058	106	91	100	NA	100
		0.742	74				
		0.918	92				
		0.911	91				
		0.898	90				
	7	0.971	97	95	105	108	89
		0.901	90				
		1.007	101				
		0.942	94				
		0.936	94				
	30*	0.832	83	86	95	94	92
		0.906	91				
		0.880	88				
		0.831	83				
		0.860	86				
Wheat (green material)	0	0.810	81	79	100	NA	100
		0.764	76				
		0.708	71				
		0.851	85				
		0.808	81				
	7	0.651	65	72	90	91	78
		0.745	74				
		0.754	75				
		0.709	71				
		0.704	70				
	22*	0.800	80	79	111	97	82
		0.780	78				
		0.794	79				

[a]Normalised Recovery = (Average recovery / average recovery at day 0) x 100%

[b]Corrected percent recovery = (Average % recovery (stored spiked sample) / Average of fresh concurrent recoveries) x 100%

NA = Not applicable

*The extracts were shaken for 15 minutes on days 22 and 30 compared to 2 minutes on day 7.

Conclusion

The findings from the short-term storage stability study demonstrate that the temperature deviations during shipment did not result in a negative impact on the quality of the residue studies concerned.

The storage conditions tested (at least 8 hours at +1°C followed by 7 days at -7°C) were such that the most unfavourable conditions which were determined for all shipments are covered. Residues of amidosulfuron were shown to be stable under the experimental conditions tested.

Stability in plant extracts

Report: KCA 6.1/06 [redacted]; 2004; M-226888-01-1
Title: Modification M001 to method 00815 for the determination of residues of amidosulfuron, iodosulfuron-methyl-sodium including metabolite metsulfuron-methyl, foramsulfuron and mesosulfuron-methyl in/on flax and wheat matrices by HPLC-MS/MS
Report No.: 00815/M001
Document No.: M-226888-01-1
Guideline(s): --
Guideline deviation(s): --
GLP/GEP: **yes**

Material and Methods
Stability of residues in sample extracts was studied in wheat grain (0.01 mg/kg and 0.1 mg/kg), in wheat green material (0.05 mg/kg and 0.5 mg/kg), and in flax grain (0.01 mg/kg and 0.1 mg/kg). After initial analysis, the analytical solutions were stored in a refrigerator and reanalysed after 2 weeks. Storage conditions were the same as those used for analytical solutions (in a refrigerator at 4°C ± 3°C).

Findings
Stability tests showed that all analytes were stable in solvent for at least 2 months and in matrix solutions for at least 2 weeks when refrigerated at 4°C ± 3°C. Please refer to Table 6.1- 11.

Table 6.1- 11: Storage stability of amidosulfuron (AE F075032) in sample extracts

Crop	Sample Material	Number of days	Fortification Level [mg/kg]	Recovery [%]						Sample number	RSD [%]
				Individual					Mean		
Wheat	Grain	0	0.01	98	98	101	97	99	99	5	1.5
	Grain	0	0.10	95	90	93	98	92	94	5	3.3
	Grain	14	0.01	95	92	99	94	90	94	5	3.6
	Grain	14	0.10	98	89	96	101	95	96	5	4.6
Wheat	Green Material	0	0.05	88	89	92	87	85	88	5	2.9
	Green Material	0	0.5	89	91	88	90	88	89	5	1.5
	Green Material	14	0.05	89	90	88	89	84	88	5	2.7
	Green Material	14	0.5	88	87	86	86	85	86	5	1.3
Flax	Grain	0	0.01	86	88	84	86	88	86	5	1.9
	Grain	0	0.10	94	93	92	86	89	91	5	3.6
	Grain	14	0.01	91	86	86	85	80	86	5	4.6
	Grain	14	0.10	85	91	91	83	89	88	5	4.1

Conclusion
This investigation showed that amidosulfuron is stable in representative matrix solutions for at least two weeks under refrigerated conditions.

During the development of the enforcement method 01360 (Report MR-13/007) for the determination of amidosulfuron and other sulfonylureas in samples from plant origin by HPLC-MS/MS, the stability in final plant extracts was checked for the tested sample materials over a period of 16 to 43 days (KCA 4.2/21 [redacted]P; [redacted]; 2013; M-455564-01-1) and it has also been checked during the Independent Lab Validation over a period of 3 to 13 days (KCA 4.2/22 [redacted]; 2013; M-470160-01-1). The results are presented below and the studies are detailed in the Analytical Methods section.

Report:	KCA 6.1/07 [redacted]>; [redacted]; 2013; M-455564-01-1
Title:	Analytical method 01360 for the determination of amidosulfuron, metsulfuron-methyl, iodosulfuron-methyl-sodium, mesosulfuron-methyl, and foramsulfuron in samples from plant origin by HPLC-MS/MS
Report No.:	MR-13/007
Document No.:	M-455564-01-1
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC Guidance document on residue analytical methods, SANCO/825/00/rev. 8.1, European Commission, Directorate General Health and Consumer Protection, 16/11/2010 US EPA Residue Chemistry Test Guideline OCSPP 860.1340: Residue Analytical Method OECD Guideline, ENV/JM/MONO (2007) 17, Aug 13, 2007
Guideline deviation(s):	not applicable
GLP/GEP:	**yes**

Material and Methods

Stability of residues in sample extracts was studied in sugar beet body, sugar beet leaf, lemon fruit, oilseed rape and cereal straw (0.1 mg/kg). The following table shows the recoveries comparing initial day of analysis and analysis after storage of the final samples at 4°C ± 3°C under dark conditions over the given periods. To check the stability, after freshly prepared matrix standards were prepared and analysed together with the aged recovery samples.

Findings

Amidosulfuron was stable for all matrices at the given conditions. In sugar beet, body and leaf, in lemon fruit oil rape seed and cereal straw, no significant decrease could be observed, compared to old recovery samples.

Table CA 6.1- 12: Stability of amidosulfuron in Plant Extracts, Quantifier Mass Transition

Sample Material	Fortification Level [mg/kg]		Recovery Rates [%]					Mean
Sugar beet, body	0.1	Day 0 (initial analysis)	94	90	91	93	89	
		43 days reanalysis	97	95	92	94	91	
		deviation day 0/43 days	3.2	5.6	1.1	1.1	2.2	2.6
Sugar beet, leaf	0.1	Day 0 (initial analysis)	86	86	89	91	94	
		43 days reanalysis	88	84	91	91	88	
		deviation day 0/43 days	2.3	2.3	2.2	0.0	6.4	2.7
Lemon, fruit	0.1	Day 0 (initial analysis)	91	95	96	90	96	
		16 days reanalysis	104	102	101	106	105	
		deviation day 0/16 days	14.2	7.4	5.2	17.8	9.4	10.8
Oilseed Rape	0.1	Day 0 (initial analysis)	97	92	89	91	92	
		38 days reanalysis	88	87	85	86	86	
		deviation day 0/38 days	9.3	5.4	4.5	5.5	6.5	6.2
Cereals Straw	0.1	Day 0 (initial analysis)	93	84	83	88	86	
		30 days reanalysis	97	96	100	90	94	
		deviation day 0/30 days	4.3	14.3	20.5	2.3	9.3	10.1

The results suggest that samples could be analysed during the storage period, amidosulfuron being stable in final plant extracts at the given conditions (4°C ± 3°C under dark conditions).

Report: KCA 6.1/08 [redacted]; 2013; M-470160-01-1
Title: Independent lab validation of BCS method 01360 for the determination of residues of amidosulfuron, metsulfuron-methyl, iodosulfuron-methyl-sodium, mesosulfuron-methyl and foramsulfuron in samples from plant origin by HPLC-MS/MS
Report No.: 2013/0060/01
Document No.: M-470160-01-1
Guideline(s): Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC.

European Commission Guidance Document for Generating and Reporting Methods of Analysis in Support of Pre-Registration data Requirements for Annex II (part A, Section 4) and Annex III (part A, section 5) of directive 91/414, SANCO/3029/99.

Guidance document on residue analytical methods; SANCO/825/00 rev. 8.1, European Commission, Directorate General Health and Consumer Protection; 2010-11-16

OECD Guidance Document on Pesticide Residue analytical Methods; ENV/JM/Mono (2007); 2007-08-13

US EPA Residue Chemistry Test Guideline OCSPP 860.1340: Residue Analytical Method
Guideline deviation(s): not applicable
GLP/GEP: **yes**

Material and Methods
During the development of the Independent Lab Validation, the stability was tested after storage of the final samples in the dark at a temperature between 2 – 8 °C over three to thirteen days. The following table shows the measurements comparing initial day of analysis and analysis after storage of the final samples over the given periods. Calibration was conducted with freshly prepared matrix standards at initial analysis and for analysis after storage.

Findings

Table CA 6.1- 13: Stability of amidosulfuron in Plant Extracts, Quantifier Mass Transition

Sample Material	Fortification Level [mg/kg]	Date of analysis	Concentration [ng/ml]			Mean deviation [%]*
Sugar beet, body	0.1	2013-08-28	10.70	9.88	10.70	
		2013-09-10	10.80	11.00	11.00	
Sugar beet, leaf	0.1	2013-08-29	10.20	10.50	10.40	-16
		2013-09-09	8.94	8.74	8.49	
Lemon, fruit	0.1	2013-09-06	8.94	8.99	9.59	-71
		2013-09-09	2.84	2.49	2.64	
Oilseed Rape	0.1	2013-09-02	10.30	9.91	10.30	-73
		2013-09-09	2.78	2.67	2.66	
Cereals Straw	0.1	2013-09-04	7.05	7.51	7.38	15
		2013-09-09	8.42	8.56	8.26	

* Mean deviation [%] between initial analysis and days of reanalysis

Conclusion
Significant deviations between initial and re-analysis were observed especially for the matrices lemon fruit and oilseed rape. Therefore the analysis of the samples has to be conducted within 1 day.

Summary of amidosulfuron storage stability in plants

Table 6.1- 14: Summary of storage stability of amidosulfuron and its metabolites desmethyl-amidosulfuron and amidosulfuron-guanidine in plants

Compound	Plant matrix	Characteristics of the crop group	Stability	Storage conditions	Reference
Amidosulfuron	Wheat grain	Dry commodities	Up to 720 days	-18°C	Annex II S4, KIIA 6.0
	Wheat straw	-			
	Wheat Shoot	High water content			
	Linseed grain	High oil content	Up to 720 days		KCA 6.1/03
	Wheat green material	High water content	Up to 22 days	+1°C for 8 hours then -7°C	KCA 6.1/05
	Tomato fruit	High water content	Up to 30 days		
Desmethyl-amidosulfuron (AE F101630)	Wheat grain	Dry commodities	Up to 720 days	≤ -18°C	Annex II S4, KIIA 6.0
	Wheat straw				
	Wheat Shoot	High water content	Up to 714 days		KCA 6.1/03
	Linseed grain	High oil content	Up to 720 days		
Amidosulfuron-guanidine	Grass, green material and hay	High water content	Up to 720 days (final report)	≤ -18°C	KCA 6.1/04
	Wheat grain	Dry commodities and high starch content			
	Wheat straw				

CA 6.2 Metabolism, distribution and expression of residues

Metabolism, distribution and expression of residues were studied in plants and livestock with amidosulfuron.

CA 6.2.1 Metabolism, distribution and expression of residues in plants

Original Annex II dossier

In the original Annex II dossier, the plant metabolism studies were performed in wheat and flax. The studies were presented in the Tier 2 summary document on the active substance, Section 4, Point 6.2. No supplementary metabolism studies in plants are considered necessary.

Studies submitted and evaluated for the first inclusion of amidosulfuron on Annex I:

Cereals

Report:	KCA 6.2.1/01; [redacted]1989; M-123006-01-1
Title:	Hoe 075032-14C Residue determinations in wheat (Triticum aestivum) after application of 0.05 kg active ingredient/ha in a post-emergent treatment
Report No.:	A40402
Document No.:	M-123006-01-1
Guideline(s):	US EPA, Subdivision O, Section 171-4
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.2.1/02[redacted]Y; 1993; M-132605-01-1
Title:	Addendum to Report CM032/88 Hoe 075032-14C Residue Determinations in Wheat (Triticum aestivum) after Application of 0.05 kg Active Ingredient/ha in a Post-emergence Treatment Investigations of the Residues in Wheat Straw
Report No.:	A51696
Document No.:	M-132605-01-1
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**no**

Report:	KCA 6.2.1/03[redacted], 1993; M-132551-01-1
Title:	Metabolism in wheat model study using cut wheat plants in a nutrient solution Hoe 075032-14C
Report No.:	A51630
Document No.:	M-132551-01-1
Guideline(s):	USEPA (=EPA):
Guideline deviation(s):	--
GLP/GEP:	**no**

Report:	KCA 6.2.1/04[redacted]O; 2004; M-234958-01-1
Title:	Addendum (3) to report CM032/88 of Hoechst AG Residue determinations in wheat (Triticum aestivum) after application of 0.05 kg active ingredient/ha in a post-emergent treatment Investigation of metabolites M3 and M4, found in forage Hoe 075
Report No.:	C043659
Document No.:	M-234958-01-1
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**no**

Report:	KCA 6.2.1/05[redacted]; 1988; M-121145-01-2
Title:	Data on the residue behaviour and metabolism in plants Hoe 075032
Report No.:	C044583
Document No.:	M-121145-01-2
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**no**

Flax (linseed)

Report:	KCA 6.2.1/06[redacted],; 1997; M-142293-01-1
Title:	Amidosulfuron 75WG Codes : AE F075032 (radiolabelled) Metabolism in linseed
Report No.:	A58560
Document No.:	M-142293-01-1
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**yes**

The metabolism of amidosulfuron was investigated in <u>flax</u> at the normal rate of 30 g a.s./ha and an exaggerated rate of 90 g a.s./ha. [^{14}C]-residues in seeds were below the limit of detection. At maturity residues in treated foliage were low with 1.164 mg equivalents/kg at day 0. After 14 days residues had already declined to 0.191 mg equivalents/kg in foliage and 0.104 mg equivalents/kg in immature capsules. No detectable residues were found in seeds at harvest at the normal and the exaggerated rate. More than 85% of the residues on foliage are comprised of highly polar components.

The metabolism of amidosulfuron in <u>wheat</u> was investigated using the 2-^{14}C-pyrimidyl-labelled active ingredient. The wheat plants were treated at an early tillering stage at a rate of 50 g a.s./ha. The total radioactive residues at harvest were low, since even after treatment at the exaggerated rate of 50 g a.s./ha these residues did not exceed 0.006 mg/kg in grain and 0.1 mg/kg in straw. Identification of the extractable residues in grain was not possible due to the extremely low concentration. All the metabolites detected in wheat were also found in animal metabolism studies.

<u>*"AIR process" - new study submitted*</u>

The following study was submitted to the former RMS (Austria) in December 2010 during the post Annex I review of amidosulfuron containing formulations. The conclusions of the former RMS are compiled in the DAR Addendum Volume 3, Annex B (Addendum to monograph prepared in the context of post Annex I procedure (new annex II data) – rev. 1 February 2011).

Report:	KCA 6.2.1/01[redacted],;[redacted]; 2010; M-123006-02-1
Title:	Hoe 075032-14C [Amidosulfuron] - Residue determinations in wheat (Triticum aestivum) after application of 0.05 kg active ingredient/ha in a post-emergent treatment
Report No.:	CM032/88
Document No.:	M-123006-02-1
Guideline(s):	US EPA, Subdivision O, Section 171-4
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

In this document, an update of the metabolic pathway of amidosulfuron in wheat is presented, showing a reassignment of the structure of a metabolite in wheat straw and wheat forage.

In the initial EU dossier from 2003, the study CM032/88 ([redacted] 1989; M-123006-01-1) and an amendment to this study ([redacted],; 1993; M-132605-01-1) were submitted. A second amendment to this study ([redacted]; 2004; M-234958-01-1) was submitted during the preparation of the DAR. Since then it was shown that a metabolite of amidosulfuron in wheat forage (M1) and straw (P3) was originally attributed to an erroneous structure (AE F128870, Hoe 128870). This is due to the reassignment of the identity of a soil metabolite that had served for the identification of the fore-mentioned wheat metabolite.

For provision of a complete overview of the metabolism of amidosulfuron in wheat, all respective information from the basic study report, two addenda of the former study director [redacted] and the re-attributed metabolite structure are compiled in an amendment, presented below.

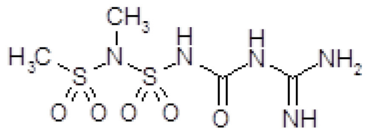
Summary
A metabolite of the sulfonyl urea herbicide amidosulfuron in wheat forage (M1) and straw (P3) was originally attributed to an erroneous structure (AE F128870, Hoe 128870) ([redacted] 1989; M-123006-01-1), based on comparison of retention times with a metabolite isolated in a soil metabolism study of ^{14}C-radiolabelled Amidosulfuron ([redacted].; 1989; M-122934-01-1, A40368; KCA 7.1). A repetition of the soil metabolism study proved that this soil metabolite had to be re-attributed unambiguously to another structure (i.e. Amidosulfuron-guanidine, BCS-CO41839) using the nowadays available state of the art, highly spectroscopy methodology (LC-MS/MS; [redacted]; 2010; M-366012-01-1, Study No. M 125 1749-9).

The amendment provides:

- An update of the study title including the Common Name "Amidosulfuron" of the test item.
- An update of the structure of the forage metabolite M1, being identical with straw metabolite P3, as erroneously shown in Figure 7 of the first addendum to report CM032/88.
- An update of the metabolic pathway of amidosulfuron in wheat as presented in Figure 5 of the second addendum to report CM032/88
- A table of the composition of amidosulfuron residues in forage and straw previously not provided in the basic study report and the two addenda.

Amended Figures
The identification of an amidosulfuron metabolite in wheat ([redacted] 1989; M-123006-01-1) was conducted by chromatographic comparison of the metabolite "A" detected in a soil metabolism study ([redacted].; 1989; M-122934-01-1, A40368; KCA 7.1.1.1/01). Since some minor soil metabolites remained unidentified in the mentioned soil metabolism study, the study had to be repeated ([redacted]K; 2010; M-366012-01-1, M125 1749-9). Apart from other soil metabolites formed, a more polar metabolite was observed. The proportion of this polar metabolite gradually increased with time to get major (> 10% of applied) in all four soils used after 10 – 20 days of incubation, as already observed with the former soil metabolite "A" ([redacted]; 1989; M-122934-01-1; KCA 7.1.1.1/01). Although the same separation conditions were applied in the new study ([redacted]; 2010; M-366012-01-1) as in the former study of [redacted]; 1989; M-122934-01-1 (KCA 7.1.1.1/01), the elution times of corresponding metabolites were slightly shifted due to different chromatographic equipment. However, the sequence of metabolites remained the same and no other major peak appeared in the corresponding region of the chromatograms (10 – 20 min).

Therefore, it is suggested that the continuously growing polar metabolite in the study of [redacted].; 2010; M-366012-01-1 is identical with the former soil metabolite "A" in the study of [redacted]; 1989; M-122934-01-1 (KCA 7.1.1.1/01). It co-eluted with the meanwhile synthesised amidosulfuron-guanidine. The identity of the structure could be confirmed unequivocally by HPLC-MS/MS using electrospray ionization in both the positive and negative mode. Consequently, the structure of the former soil metabolite "A" has now been re-assigned to be amidosulfuron-guanidine:



Amidosulfuron-guanidine
BCS-CO41839

As a further consequence of this re-evaluation the wheat metabolite P3 in straw and M1 forage also has to be re-attributed to amidosulfuron-guanidine, BCS-CO41839.

In summary, the major residue components in forage and straw following post-emergent application of ^{14}C-amidosulfuron to wheat are compiled in Table 6.2-1.

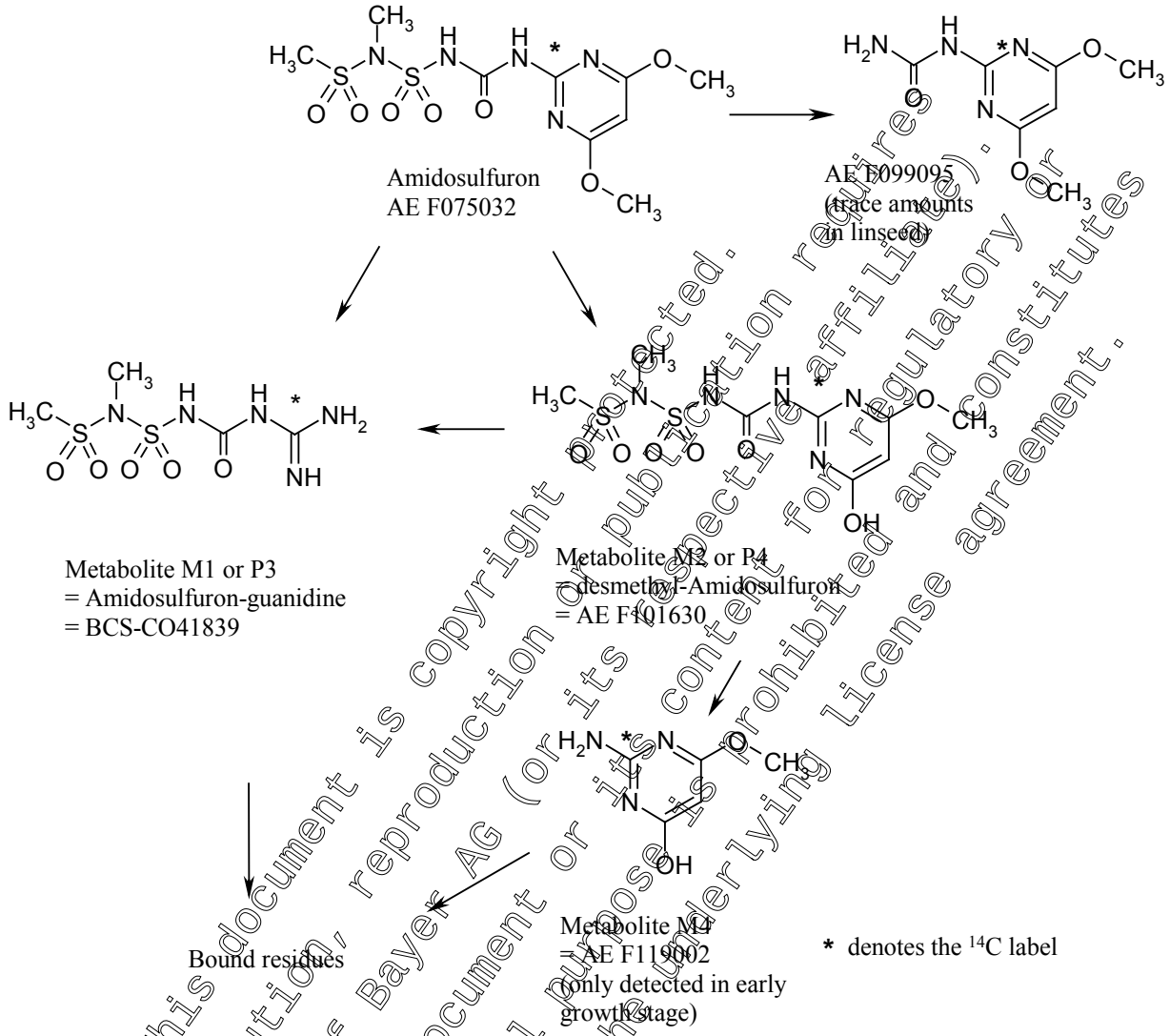
Table 6.2-1: Residues of amidosulfuron in wheat following foliar application at a rate of 50 g a.s./ha

	Designation	Forage 14 DAT *)	Straw 97 DAT	Grain 97 DAT
TRR (mg equ/kg)		0.085	0.100	0.006
Residue components in % of TRR (recalculated)				
Parent Amidosulfuron (AE F075032)	-	8.2	n.d. **)	n.a. ***)
AE F101630, Desmethyl-amidosulfuron	M2 in forage, P4 in straw	18.2	40.0	n.a.
BCS-CO41839 Amidosulfuron-Guanidine	M1 in forage P3 in straw "A" in a former soil study	19.3	14.8	n.a.
AE F119002 2-amino-4-hydroxy-6-methoxypyrimidine	M4 in forage	17.1	n.d.	n.a.
unknown	-	5.1 -	5.8 - 9.2	n.a.

*DAT: days after treatment; **n.d.: not detected; ***n.a.: not analysed

Based on the upper discussion the metabolic pathway of amidosulfuron in wheat has now to be updated as presented in the Figure 6.2-1. This updated metabolic scheme is replacing the incorrect pathway presented as Figure 6.1 in the "1st Supplemental to Tier 2 Summary of Residues in or on Treated Products, Food and Feed for Amidosulfuron, Code; AE F075032".

Figure 6.2-1: Proposed metabolic pathway of amidosulfuron in plants (revised)



Conclusion

Following a repetition of a soil metabolism study, the identity of the former soil metabolite "A" has now been re-assigned to Amidosulfuron-guanidine: BCS-CO41839. Consequently, the identity of metabolite P3 in wheat straw and M1 wheat forage has to be reassigned to Amidosulfuron-guanidine as well, since the soil metabolite "A" served for identification of the wheat metabolite.

CA 6.2.2 Poultry

Original Annex II dossier

Studies submitted and evaluated for the first inclusion of amidosulfuron on Annex I:

Report:	KCA 6.2.2/01 [redacted],; 1991; M-130150-01-1
Title:	HOE 075032-14-C Metabolism in the laying hen following repeated oral administration
Report No.:	A46076
Document No.:	M-130150-01-1
Guideline(s):	USEPA (=EPA): § 171-4
Guideline deviation(s):	--
GLP/GEP:	**yes**

The livestock metabolism of amidosulfuron was investigated in laying hens. The studies were presented in Section 4, Point 6.2.2 and 6.2.3 of the Annex II. No new studies were deemed necessary for the AIR process.

2-^{14}C-pyrimidyl-labelled active ingredient was orally administered at dose rates equivalent to 10.7 ppm in the diet (hens). Amidosulfuron was shown to be rapidly and efficiently excreted. The levels of radioactive residues in eggs, and edible tissues were very low, thus indicating that there is no risk of accumulation of amidosulfuron residues in food of animal origin. The major identified residue component was parent amidosulfuron.

CA 6.2.3 Lactating ruminants

Original Annex II dossier

Studies submitted and evaluated for the first inclusion of amidosulfuron on Annex I:

Report:	KCA 6.2.3/01 [redacted],; 1991; M-130922-01-1
Title:	HOE 075032-14-C Metabolism in the lactating goat following repeated oral administration
Report No.:	A46978
Document No.:	M-130922-01-1
Guideline(s):	USEPA (=EPA): § 171-4
Guideline deviation(s):	
GLP/GEP:	**yes**

Report:	KCA 6.2.3/02 [redacted],; [redacted]; 1991; M-232270-01-1
Title:	14C-Hoe 075032 - Metabolism in the lactating goat
Report No.:	C042260
Document No.:	M-232270-01-1
Guideline(s):	--
Guideline deviation(s):	
GLP/GEP:	**yes**

The livestock metabolism of amidosulfuron was investigated in a lactating goat. The studies were presented in Section 4, Point 6.2.2 and 6.2.3 of the Annex II. No new studies were deemed necessary for the AIR process.

2-^{14}C-pyrimidyl-labelled active ingredient was orally administered at dose rates equivalent to 4.75 ppm in the diet (goat). Amidosulfuron was shown to be rapidly and efficiently excreted. The levels of radioactive residues in milk and edible tissues were very low, thus indicating that there is no

risk of accumulation of amidosulfuron residues in food of animal origin. The major identified residue component was parent amidosulfuron.

CA 6.2.4 Pigs

A pig metabolism study was not conducted, since metabolism followed comparable pathways in all other tested species (rat, dog, hen and cow).

CA 6.2.5 Fish

Since no residues above 0.01 mg/kg were found in cereal grain and no accumulation is to be expected in tissues (log Pow < 3), the fish metabolism study is not required.

CA 6.3 Magnitude of residue trials in plants

Amidosulfuron is an herbicidal active substance. In 2003, the original Annex II dossier was submitted to Austria. In that dossier, uses on cereals, flax (linseed), meadow and pasture were supported with residue trial data.
Some new studies have since been conducted with amidosulfuron containing formulations for use in cereals, grass and flax which are the "safe use" crops supported in the AIR3 process.

The Residue Trial Tables can be found in the document below. They include the supplementary trials presented in this dossier in support of the formulated product that were not already presented and evaluated during the Annex I inclusion of amidosulfuron.

Report:	KCA 6.3/01 [redacted]; 2016; M-550616-01-1
Title:	Residue trial tables - Amidosulfuron - AmidosulfuronWG 75 (750 g/kg) - Supplementary residue trials Northern zone and Southern zone
Report No.:	M-550616-01-1
Document No.:	M-550616-01-1
Guideline(s):	Regulation (EC) No 1107/2009 Section 4, Point 8.3
Guideline deviation(s):	none
GLP/GEP:	**no**

CA 6.3.1 Cereal

Studies submitted and evaluated for the first inclusion of amidosulfuron on Annex I:

In the Original Annex II dossier a total of 21 residue trials were submitted for the uses of amidosulfuron on cereals in Europe and evaluated. The application rate corresponded to a single application of 30 g/ha of amidosulfuron at growth stages between BBCH 37 and 49. Two trials were performed with application rates of 45 g/ha, 3 trials with application rates of 60 g/ha and 1 trial with an application rate of 90 g/ha. Applications were made in spring in order to cover the shortest pre-harvest interval (PHI). The trial locations were spread over main growing areas of the EU Northern and EU Southern zone thus covering different soils and climatic conditions. The same WG formulation (AE F075032 00 WG75 A1) was used in all the trials.

Samples were taken for analysis at harvest (growth stage 89-92, fully ripe). Additional samples were taken in a variety of trials at the day of application and at interim growth stages in order to determine a residue decline. For data gathering purposes, the samples taken from these trials were analysed for

parent amidosulfuron and in many cases metabolite AE F101630 using method AL-010/91-0 with a limit of quantification of 0.01 mg/kg for grain and 0.05 mg/kg for cereal shoot and straw although residue definition was defined as parent only.

For amidosulfuron and desmethyl-amidosulfuron, maximum storage periods of samples are covered by the storage stability data. For five northern trials, the periods of time for which straw samples from these trials were stored prior to analysis (819 - 865 days) exceed the maximum covered by the storage stability data (720 days) for both analytes. They are, however, considered valid as there is no known instability for this family of compounds and it is very unlikely that degradation in straw would occur between 721 days and 865 days.

Reported results correspond to residue trials conducted at the intended application rate (30 g/ha).

Table 6.3.1- 1: Summary of amidosulfuron and metabolite AE F101630 residue data from cereal trials (Annex II dossier)

Application Rate (g/ha)	Analyte	Sample material	Growth stage (BBCH code)	DALT (days)	n*	Residue level (mg/kg)		
						Min.	Max.	Median
Northern Europe								
30 g/ha at BBCH 49	Amidosulfuron	Shoots without ears	71	16-32	10	<0.05	<0.05	<0.05
		Ears	71	16-32	5	<0.05	<0.05	<0.05
		Straw	92	64-87	10	<0.05	<0.05	<0.05
		Grain	92	64-87	9	<0.01	0.012	<0.01
	AE F101630	Shoots without ears	71	16-32	10	<0.05	<0.05	<0.05
		Ears	71	16-32	5	<0.05	<0.05	<0.05
		Straw	92	64-87	10	<0.05	<0.05	<0.05
		Grain	92	64-87	9	<0.01	<0.01	<0.01
Southern Europe								
30 g/ha at BBCH 37-41	Amido-sulfuron	Shoot	51-55	10-13	5	<0.05	0.31	-
		Straw	87-91	63-75	6	<0.05	<0.05	<0.05
		Grain	87-91	63-75	6	<0.01	<0.01	<0.01
	AE F101630	Shoot	51-55	10-13	2	<0.05	<0.05	-
		Straw	91	63	3	<0.05	<0.05	-
		Grain	91	63	3	<0.01	<0.01	-

* n: number of samples

At harvest, residues of parent amidosulfuron and its metabolite AE F101630 in all cereal commodities were below the limit of quantification (grain: 0.01 mg/kg; straw: 0.05 mg/kg) except in one trial (0.012 mg/kg in grain).

Report:	KCA 6.3.1/01[redacted]; [redacted]; 1992; M-136438-01-2
Title:	Hoe 075032 - water dispersible granules - 75 % (Code: Hoe 075032 00 WG75 A104) Investigation of residues in winter wheat following a single application of Hoe 075032
Report No.:	A49846
Document No.:	M-136438-01-2
Guideline(s):	BBA:
Guideline deviation(s):	--
GLP/GEP:	**yes**

Report:	KCA 6.3.1/02[redacted]7; 1993; M-131103-01-2
Title:	AE F075032; WG 75; wheat, soft; Germany; BBA
Report No.:	A50063
Document No.:	M-131103-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/03[redacted]u; 1993; M-131104-01-2
Title:	AE F075032; WG 75; wheat, soft; Germany; BBA
Report No.:	A50064
Document No.:	M-131104-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/04[redacted]V; [redacted]; 1992; M-136440-01-2
Title:	Hoe 075032 - water dispersible granules - 75 % (Code: Hoe 075032 00 WG75 A104) Investigation of residues in winter rye following a single application of Hoe 075032
Report No.:	A49848
Document No.:	M-136440-01-2
Guideline(s):	
Guideline deviation(s):	
GLP/GEP:	**yes**

Report:	KCA 6.3.1/05[redacted]k; 1991; M-130745-01-2
Title:	AE F075032; WG 75; rye; Germany; BBA
Report No.:	A46743
Document No.:	M-130745-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/06[redacted]; 1993; M-131107-01-2
Title:	AE F075032; WG 75; oats; Germany; BBA
Report No.:	A50067
Document No.:	M-131107-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/07[redacted],;[redacted]; 1992; M-136437-01-2
Title:	Hoe 075032 - water dispersible granules - 75 % (Code: Hoe 075032 00 WG75 A104) Investigation of residues in winter barley following a single application of Hoe 075032
Report No.:	A49539
Document No.:	M-136437-01-2
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**yes**

Report:	KCA 6.3.1/08[redacted],; 1993; M-131105-01-2
Title:	AE F075032; WG 75; barley; Germany; BBA
Report No.:	A50065
Document No.:	M-131105-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/09[redacted],; 1993; M-131106-01-2
Title:	AE F075032; WG 75; barley; Germany; BBA
Report No.:	A50066
Document No.:	M-131106-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/10[redacted]; 1993; M-132558-01-2
Title:	AE F075032; WG 75; wheat; Italy; BBA
Report No.:	A51637
Document No.:	M-132558-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/11[redacted]; 1993; M-132559-01-2
Title:	AE F075032; WG 75; wheat; Italy; BBA
Report No.:	A51638
Document No.:	M-132559-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/12[redacted]; 1993; M-132560-01-2
Title:	AE F075032; WG 75; wheat; Italy; BBA
Report No.:	A51639
Document No.:	M-132560-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/13[redacted],; 1993; M-132561-01-2
Title:	AE F075032; WG 75; wheat; Italy; BBA
Report No.:	A51640
Document No.:	M-132561-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/14[redacted],,[redacted]; 1996; M-139751-01-1
Title:	Amidosulfuron water dispersible granule 75 % Code: Hoe 075032 00 WG75 A110 Determination of the active substance at harvest following one application of amidosulfuron in wheat to establish a maximum residue level. European Union (southern z
Report No.:	A55871
Document No.:	M-139751-01-1
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**yes**

Report:	KCA 6.3.1/15[redacted],; 1996; M-140795-01-1
Title:	Amidosulfuron water dispersible granule (WG) 750 g/kg Code: Hoe 075032 00 WG75 A109 Determination of Residues of Hoe 075032 to establish a Maximum Residue Level following one application in Durum Wheat
Report No.:	A57035
Document No.:	M-140795-01-1
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**yes**

Report:	KCA 6.3.1/16[redacted],,1993; M-131783-01-2
Title:	AE F075032; WG 75; wheat; Greece; BBA
Report No.:	A50776
Document No.:	M-131783-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Report:	KCA 6.3.1/17[redacted],; 1993; M-131784-01-2
Title:	AE F075032; WG 75; wheat; Greece; BBA
Report No.:	A50777
Document No.:	M-131784-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

"AIR process" - new studies submitted

The critical GAPs with respect to consumer intake and risk assessment for the preparation amidosulfuron WG 75 (AMS WG 75) are presented in **Table 6.3.1- 2**.

Table 6.3.1- 2: critical GAPs (and respective fall-back GAPs, if applicable)

Crop and/ or situation **	Zone	Product code	F, Fn, Fpn G, Gn, Gpn or I***	Pests or Group of pests controlled	Formulation		Application				Application rate per treatment			PHI (days)	Remark
					Type	Conc. of as	method kind	growth stage & season	number min max	interval between applications (min)	kg as/hL min max	water L/ha min max	kg as/ha min max		
Winter cereals	NEU	**AMS WG 75**	F	Dicot. weeds incl. *Galium, Sinapis arvensis, Raphanus raphanistrum, Capsella, Myosotis, Scandix, Tordylium, Ranunculus,* volunteer oil seed rape and sunflower	WG	750 g/kg	Foliar	21-49* End of winter, beginning of vegetation	1	-	0.0075-0.03	100-400	0.030	NR	(*) All EU except FRA/ITA (up to BBCH 32)
		AMS WG 75	F		WG	750 g/kg	Foliar	13-49* End of winter, beginning of vegetation	1		0.00375-0.015	100-400	0.015	NR	
Spring cereals	NEU	**AMS WG 75**	F	Dicot. weeds incl. *Galium, Sinapis arvensis, Raphanus raphanistrum, Capsella, Myosotis, Scandix, Tordylium, Ranunculus,* volunteer oil seed rape and sunflower	WG	750 g/kg	Foliar	12-49* Spring application	1		0.00375-0.03	100-400	0.015-0.030	NR	(*) All EU except FRA/ITA (up to BBCH 32)

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0 should be given in column 1
** Use also code numbers according to Annex I of Regulation (EU) No 396/2005
*** F: professional field use, Fn: non-professional field use, Fpn: professional and non-professional field use, G: professional greenhouse use, Gn: non-professional greenhouse use, Gpn: professional and non-professional greenhouse use, I: indoor application
NEU= Northern Europe; SEU= Southern Europe
NR= not relevant

New studies on the magnitude of residue are submitted by the applicant in the framework of this application.
In this document, the critical GAP used for setting the EU MRL is compared to critical GAP for the formulated product.

Table 6.3.1- 3: Amidosulfuron: summary of critical GAPs used for setting the EU MRL on cereals

Crop	F, G or I*	Region	Growth stage	Maximum Number of Applications	Maximum Rate (g a.s./ha)	PHI (days)
Cereals	F	N-EU and S-EU	BBCH 13-49	1	**30**	-

* F Field; G Greenhouse; I Indoor.

In Southern Europe, the critical GAP for the uses supported in this dossier is less critical than the critical GAP used for setting the EU MRL.

Supplementary residue trials – Southern Europe
A total of 2 supplementary residue trials were conducted on wheat or barley with Amidosulfuron WG 75 formulation in Southern Europe. The trials were conducted during the 2014 growing season. The purpose of the studies was to determine the residue levels of amidosulfuron and its metabolite AE F101630 (desmethyl-amidosulfuron) after a spray application of 30 g a.s./ha at the growth stage 49. This represents a worst case compared to the intended use as the application should be performed latest at growth stage 32 in Southern Europe. These trials cover the use supported in this dossier.

Table 6.3.1- 4: Number of residue trials conducted with Amidosulfuron WG 75 per geographical region and vegetation period

Crop	Region	Formulation*	Number of Trials		Report-No.	Dossier Ref.
			Vegetation period	Total		
			2014			
Wheat, barley	S-EU	WG	2	2	14-2007 14-2008	KCA 6.3.1/19 & 20

N-EU: northern Europe S-EU: southern Europe

Report: KCA 6.3.1/1[redacted]; 2016; M-546210-02-1
Title: Amendment no. 1 to report no: 14-2007 - Determination of the residues of amidosulfuron in/on barley after spray application of amidosulfuron WG 75 in southern France
Report No.: 14-2007
Document No.: M-546210-02-1
Guideline(s): REGULATION (EC) No 1107/2009 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 21 October 2009 concerning the placing of plant protection products on the market
OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009)
US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Guideline deviation(s): none
GLP/GEP: **yes**

Material and methods
One field trial was conducted in/on barley during the 2014 growing season in Southern France.

The formulation WG 75, a wettable granule formulation containing 750 g/kg of amidosulfuron, was applied once with an application rate of 30 g/ha amidosulfuron and 300 L water per ha at growth stage 49.

Samples of grain, green material and straw were taken for analysis at the day of treatment and at various intervals up to 65 days in order to investigate the residue in barley. The samples were analysed for the parent compound and its metabolite AE F101630 using method 01325 (former BE-002-P09-02) with a limit of quantitation of 0.01 mg/kg.

Findings:
- Method performance: Mean recoveries were within the acceptable range of 70-110 %, RSD <20% as shown in **Table 6.3.1- 5** and **Table 6.3.1- 6**. They validate the study results.

Table 6.3.1- 5: Recovery data for amidosulfuron

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
barley / grain	0.01	94; 94; 96	95	1.2	0.01
	0.10	98; 99; 101	99	1.5	
		Overall recovery (n = 6)	**97**	**2.9**	
barley / green material	0.01	96; 98; 106	100	5.3	0.01
	0.10	100; 102; 104	102	2.0	
	1.0	93	-	-	
		Overall recovery (n = 7)	**100**	**4.6**	
barley / straw	0.01	89; 96; 96	94	4.3	0.01
	0.10	94; 94; 95	94	0.6	
	1.0	87	-	-	
		Overall recovery (n = 7)	**93**	**3.8**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with amidosulfuron, determined as amidosulfuron and calculated as amidosulfuron
These recoveries were performed during the conduct of the study 14-2007.

Table 6.3.1- 6: Recovery data for AE F101630

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
barley / grain	0.01	92; 96; 96	95	2.4	0.01
	0.10	95; 96; 97	96	1.0	
		Overall recovery (n = 6)	**95**	**1.8**	
barley / green material	0.01	98; 99; 104	100	3.2	0.01
	0.10	97; 101; 103	100	3.0	
		Overall recovery (n = 6)	**100**	**2.8**	
barley / straw	0.01	98; 103; 103	101	2.8	0.01
	0.10	105; 105; 105	105	0.0	
	1.0	93	-	-	
		Overall recovery (n = 7)	**102**	**4.5**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with AE F101630, determined as AE F101630 and calculated as AE F101630
These recoveries were performed during the conduct of the study 14-2007

- Storage stability: The storage periods of deep-frozen samples for amidosulfuron and its metabolite AE F101630 ranged between 368 and 433 days and are covered by the storage stability study.

Use patterns for the trials conducted on grass are listed in **Table 6.3.1- 7**. Residues of the parent compound amidosulfuron and its metabolite AE F101630 are summarised below in **Table 6.3.1- 8**.

- Residue results:
No residues above the LOQ of 0.01 mg/kg could be detected in any of the control samples.

Table 6.3.1- 7: Actual use pattern of residue trials conducted in/on barley with the formulation amidosulfuron WG 75

Study, Trial No., Trial SubID, GLP, Year	Crop Variety	Country	Application						
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	Water rate (L/ha)	Spray interval (days)	GS
Southern Europe									
14-2007 14-2007-01 GLP: yes 2014	Barley Limpid-Winter Barley	Southern France	WG 75	1	0.030	0.01	300	-	49

FL: Formulation No: number of applications a.s.: active substance
GS: growth stage (BBCH code) at application

Table 6.3.1- 8: Residues of amidosulfuron and its metabolite AE F101630 in/on barley, treated with the formulation amidosulfuron WG 75

Trial No. Country, GLP, Year	Sample material	Growth stage (BBCH) at sampling	DALT (days)	Residues [mg/kg]	
				amidosulfuron	AE F101630
Southern Europe					
14-2007 14-2007-01 GLP: yes 2014	green material	49 75	0 35	0.30 <0.01	<0.01 <0.01
	grain	89	65	<0.01	<0.01
	straw	89	65	0.012	<0.01

DALT = Days after last treatment

Analyte:	Final determination as:	Residues calculated as:
amidosulfuron AE F101630	amidosulfuron AE F101630	amidosulfuron AE F101630

Conclusion
One supervised field trial was conducted in/on barley in Southern Europe during the 2014 growing season, under similar application conditions than the intended use pattern with the Amidosulfuron WG 75 product.

The formulation was applied once with an application rate of 30 g/ha amidosulfuron.

This trial was conducted according to GLP.

Residues of amidosulfuron and AE F101630 in grain, green material and straw are summarised below. At harvest, residues of parent were below LOQ of 0.01 mg/kg in grain. In straw residues of parent were at 0.012 mg/kg and residues of metabolite were below LOQ of 0.01 mg/kg.

Report: KCA 6.3.1/19 [redacted]; 2016; M-546213-01-1
Title: Determination of the residues of amidosulfuron in/on wheat after spray application of amidosulfuron WG 75 in Italy
Report No.: 14-2008
Document No.: M-546213-01-1
Guideline(s): REGULATION (EC) No 1107/2009 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 21 October 2009 concerning the placing of plant protection products on the market
OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009)
US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Guideline deviation(s): none
GLP/GEP: **yes**

One field trial was conducted in/on wheat during the 2014 growing season in Italy (Southern Europe).

The formulation WG 75, a wettable granule formulation containing 750 g/kg of amidosulfuron, was applied once with an application rate of 30 g/ha amidosulfuron and 300 L water per ha at growth stage 49.

Samples of grain, green material and straw were taken for analysis at the day of treatment and at various intervals up to 66 days in order to investigate the residue wheat. The samples were analysed for the parent compound and its metabolite AE F101630 using method 01325 (former BE-002-P09-02) with a limit of quantitation of 0.01 mg/kg.

Findings:
- Method performance: Mean recoveries were within the acceptable range of 70-110 %, RSD <20% as shown in **Table 6.3.1- 9** and **Table 6.3.1- 10**. They validate the study results.

Table 6.3.1- 9: Recovery data for amidosulfuron

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
wheat / grain	0.01	73; 74; 78	75	3.5	0.01
	0.10	72; 75; 79	75	4.7	
	1.0	77	-	-	
		Overall recovery (n = 7)	**75**	**3.5**	
wheat / green material	0.01	88; 92; 100	93	6.5	0.01
	0.10	76; 79; 85	80	5.7	
	1.0	72	-	-	
		Overall recovery (n = 7)	**85**	**11.5**	
wheat / straw	0.01	70*; 72; 73	72	2.1	0.01
	0.10	77; 82; 83	81	4.0	
	1.0	82	-	-	
		Overall recovery (n = 7)	**77**	**7.0**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with amidosulfuron, determined as amidosulfuron and calculated as amidosulfuron
These recoveries were performed during the conduct of the study 14-2008
*recovery from single injection

Table 6.3.1- 10: Recovery data for AE F101630

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
wheat / grain	0.01	75; 81; 98	85	14.1	0.01
	0.10	90; 91; 96	92	3.5	
	1.0	89	-	-	
		Overall recovery (n = 7)	**89**	**9.1**	
wheat / green material	0.01	106; 108; 108	107	1.1	0.01
	0.10	89; 96; 97	94	4.6	
	1.0	94	-	-	
		Overall recovery (n = 7)	**100**	**7.6**	
wheat / straw	0.01	87; 93; 100	93	7.0	0.01
	0.10	94; 94; 100	96	3.6	
	1.0	93	-	-	
		Overall recovery (n = 7)	**94**	**4.8**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with AE F101630, determined as AE F101630 and calculated as AE F101630
These recoveries were performed during the conduct of the study 14-2008.

- Storage stability: The maximum storage periods of deep-frozen samples for amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine ranged between 384 and 450 days and is covered by the storage stability studies (cf. MCA Point 6.1.).

Use patterns for the trials conducted on grass are listed in **Table 6.3.1- 11**. Residues of the parent compound amidosulfuron and its metabolite AE F101630 are summarised below in **Table 6.3.1- 12.**

- Residue results:
The apparent residues in the control samples were below the LOQ.

Table 6.3.1- 11: Actual use pattern of residue trials conducted in/on wheat with the formulation amidosulfuron WG 75

Study, Trial No., Trial SubID, GLP, Year	Crop Variety	Country	Application						
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	Water rate (L/ha)	Spray interval (days)	GS
Southern Europe									
14-2008 14-2008-01 GLP: yes 2014	Wheat Grecale - Durum wheat	Italy	WG 75	1	0.030	0.01	300	-	49

FL: Formulation No: number of applications a.s.: active substance
GS: growth stage (BBCH code) at application

Table 6.3.1- 12: Residues of amidosulfuron and its metabolite AE F101630 in/on wheat, treated with the formulation amidosulfuron WG 75

Trial No. Country, GLP, Year	Sample material	Growth stage (BBCH) at sampling	DALT (days)	Residues [mg/kg]	
				amidosulfuron	AE F101630
Southern Europe					
14-2008 14-2008-01 GLP: yes 2014	green material	49	0	0.40	0.054
		59	7	< 0.01	0.017
		65	14	< 0.01	0.020
	grain	89	66	< 0.01	< 0.01
	straw	89	66	< 0.01	0.01

DALT = Days after last treatment

Analyte:	Final determination as:	Residues calculated as:
amidosulfuron AE F101630	amidosulfuron AE F101630	amidosulfuron AE F101630

Conclusion
One supervised field trial was conducted in/on wheat in Southern Europe during the 2014 growing season, under similar application conditions than the intended use pattern with the Amidosulfuron WG 75 product.

The formulation was applied once with an application rate of 30 g/ha amidosulfuron.

The trial was conducted according to GLP.

The residues of amidosulfuron and AE F101630 in grain, green material and straw are summarised below. At harvest, residues of parent and metabolite were below LOQ of 0.01 mg/kg in grain and straw.

Summary of 2 supplementary residue trials conducted on cereals in Southern Europe
Residues of amidosulfuron and AE F101630 in grain, green material and straw are summarised below. At harvest, residues of parent were always below LOQ of 0.01 mg/kg in grain. In straw, residues of parent ranged between <0.01 mg/kg and 0.012 mg/kg and residues of metabolite were always below LOQ.

Results in cereal grains are in agreement with the MRL of 0.01 mg/kg set in EFSA Journal 2014; 12(3):3614 and Commission Regulation (EU) 2015/1200 of 22 July 2015.

Table 6.3.1- 13: Residues on cereals after application of Amidosulfuron WG 75

Application rate (g/Ha) Growth stage	Crop	No of trials	Analyte	Sample material	DALT	Residue [mg/kg] min - max
30 g/Ha GS 49	Wheat, barley	2	amidosulfuron	green material	0	0.30-0.40
				grain	65-66	< 0.01
				straw	65-66	<0.01-0.012
		2	AE F101630	green material	0	<0.01-0.054
				grain	65-66	0.01
				straw	65-66	< 0.01

DALT = Days after last treatment, -0: before the last application

CA 6.3.2 Pastures / Meadows

Studies submitted and evaluated for the first inclusion of amidosulfuron on Annex I:

Report: KCA 6.3.2/01 [redacted]; 2003; M-214116-01-1
Title: Residue behaviour in meadows European Union (Northern Zone) 2002 Amidosulfuron AE F075032 water dispersible granule (WG) 75% w/w Code: AE F075032 00 WG75 A113
Report No.: C007836
Document No.: M-214116-01-1
Guideline(s): EU (=EEC): 7029/VI/95 rev. 5 - 22/07/97; NA: Part I
Guideline deviation(s): --
GLP/GEP: **yes**

Report: KCA 6.3.2/02 [redacted]; [redacted]; 1992; M-135909-01-2
Title: Investigation of the residues of HOE 075032 in grass (meadows and pastures)
Report No.: C000286
Document No.: M-135909-01-2
Guideline(s):
Guideline deviation(s): --
GLP/GEP: **yes**

Report: KCA 6.3.2/03 [redacted]; 1992; M-137452-01-2
Title: Report on plant protection residue trial AE F075032 00 WG75 A103
Report No.: A592
Document No.: M-137452-01-2
Guideline(s): BBA:
Guideline deviation(s): --
GLP/GEP: **no**

Report:	KCA 6.3.2/04[redacted],; 1992; M-137455-01-2
Title:	Report on plant protection residue trial Hoe 075032 00 WG75 A103
Report No.:	A59262
Document No.:	M-137455-01-2
Guideline(s):	BBA:
Guideline deviation(s):	--
GLP/GEP:	**no**

Report:	KCA 6.3.2/05[redacted],; 1992; M-137456-01-2
Title:	Report on plant protection residue trial Hoe 075032 00 WG75 A103
Report No.:	A59263
Document No.:	M-137456-01-2
Guideline(s):	BBA:
Guideline deviation(s):	--
GLP/GEP:	**no**

Report:	KCA 6.3.2/06[redacted],;[redacted];[redacted]; 1992; M-136905-01-2
Title:	Hoe 075032 - water dispersible granules - 75 % (Code: Hoe 075032 00 WG75 A104) Investigation of the residues in meadows and pastures following a single application of Hoe 075032
Report No.:	A59999
Document No.:	M-136905-01-2
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**yes**

Report:	KCA 6.3.2/07[redacted]; 1994; M-137448-03-2
Title:	AE F075032; 50 WG; grasses; Germany; BBA
Report No.:	A48428
Document No.:	M-137448-03-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.3.2/08[redacted]; 1994; M-133093-01-2
Title:	AE F075032; WG 50; grasses; Germany; BBA
Report No.:	A52248
Document No.:	M-133093-01-2
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.3.2/09[redacted],;[redacted]; 1997; M-141636-01-1
Title:	AE F075032 Water dispersible granule 50 % w/w Code: AE F075032 00 WG50 A101 Residue study in grassland to determine amidosulfuron derived residues following one application under field conditions, (Germany, 1992)
Report No.:	A57961
Document No.:	M-141636-01-1
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**yes**

A total of 25 trials were performed; all studies were considered acceptable. Amidosulfuron was applied to grass land once per season at a rate of 45 g/ha in order to investigate the residue pattern. Applications were made either in April or in September in order to cover the GAP which allows spring or autumn application. The locations covered a broad spectrum of differing localities (most were

situated in Germany, two in the UK and one in the northern part of France) thus covering different soils and climatic conditions; therefore they are representative for the EU Northern zone.
Two different wettable granule formulations (AE F075032 00 WG75 A1 or AE F075032 00 WG50 A1) were used containing amidosulfuron as the only active substance.
The results of analysis are summarised in the following table for amidosulfuron only, because the parent compound was defined as the relevant residue.

Overview of European residue data for amidosulfuron in grass

Crop, Region, Countries (no. of trials), year	Application		Residue				Reference
	Formulation content of a.s.	Rate [kg a.s./ha]	DAT [days]	Part of crop	Amidosulfuron [mg/kg]	AE F101630	
Grass, North Germany (2), France (1), UK (2), 2002, spring application	WG 750 g/kg	0.045	0 7 13-14 21 24-29 26	Sprout Sprout Sprout Sprout Hay Shoot	0.91-2.8 0.05-0.10 <0.05 <0.05 <0.05 <0.05	Not analysed	KIIA 6.3.1.3/01 02R610
Grass, North Germany (4) 1990, Spring application	WG 750 g/kg	0.045	0 3 7 14-15 21 28-59	Sprout Sprout Sprout Sprout Sprout Sprout	1.0-3.6 0.12-1.0 0.05-0.62 <0.05 <0.05 <0.05	Not analysed	KIIA 6.3.1.3/06 ER90DEU521
Grass, North Germany (4) 1991, Spring application	WG 500 g/kg	0.045	0 3 7 14 21 28 33-65	Sprout Sprout Sprout Sprout Sprout Sprout Sprout	1.3-3.0 0.09-2.4 <0.05-0.36 <0.05 <0.05 <0.05 <0.05	<0.05 <0.05 <0.05-0.09 <0.05 <0.05 <0.05 <0.05	KIIA 6.3.1.3/07 ER91DEU521 (in 2 of these 4 trials, metabolite was not analysed)
Grass, North Germany (5) 1991, Autumn application	WG 500 g/kg	0.045	0 3 7 14 21 28 150-277	Sprout Sprout Sprout Sprout Sprout Sprout Sprout	3.32-6.78 0.12-3.98 0.09-1.22 <0.05-0.14 <0.05-0.12 <0.05-0.06 <0.05	<0.05-0.15 <0.05-0.36 <0.05-0.35 <0.05-0.20 <0.05-0.25 <0.05-0.19 <0.05-0.11	KIIA 6.3.1.3/08 ER91DEU522
Grass, North Germany (4) 1992, Spring application	WG 500 g/kg	0.045	0 2-3 6-8 13-14 21 28 64 64	Sprout Sprout Sprout Sprout Sprout Sprout Sprout Hay	1.78-4.37 <0.05-1.28 <0.05-0.12 <0.05-0.07 <0.05 <0.05 <0.05 <0.05	<0.05-0.10 <0.05-0.10 <0.05-0.09 <0.05-0.07 <0.05-0.11 <0.05 <0.05 <0.05	KIIA 6.3.1.3/09 ER92DEU521

Crop, Region, Countries (no. of trials), year	Application		Residue				Reference
	Formulation content of a.s.	Rate [kg a.s./ha]	DAT [days]	Part of crop	Amidosulfuron [mg/kg]	AE F101630	
Grass, North Germany (3) 1989, Autumn application	WG 750 g/kg	0.045	0	Sprout	2.8-5.2	0.06-0.09	KIIA 6.3.1.3/02 ER89DEU509
			3	Sprout	<0.05-2.2	<0.05-0.6	
			7-8	Sprout	<0.05-0.82	<0.05-0.79	
			11-15	Sprout	<0.05-0.10	<0.05-0.71	
			21-22	Sprout	<0.05	<0.05-048	
			28-29	Sprout	<0.05	<0.05-0.46	
			181-218	Sprout	<0.05	<0.05-0.06	
			216-256	Sprout	<0.05	<0.05	
			257-269	Sprout	<0.05	<0.05	

At a 7 day-PHI, residues of parent amidosulfuron ranged between <0.05-1.22 mg/kg in fresh grass. Residues declined to 0.12 mg/kg in hay at a 21 day-PHI. At a 7 day-PHI, residues of AE F101630 in grass sprouts were ranging from <0.05 up to 0.79 mg/kg. Residues declined to 0.48 mg/kg in hay at a 21 day-PHI.

"AIR process" - new studies submitted

The critical GAPs with respect to consumer intake and risk assessment for the preparation AMS WG 75 are presented in Table 6.3.2- 1.

Table 6.3.2- 1: critical GAPs

Crop and/ or situation **	Zone	Product code	F, Fn, Fpn G, Gn, Gpn or I***	Pests or Group of pests controlled	Formulation		Application				Application rate per treatment				PHI (days)	Remark
					Type	Conc. of as	method kind	growth stage & season	number min max	interval between applications (min)	kg as/hL min max	water L/ha min max	kg as/ha min max			
Pastures, meadows	NEU & SEU	**AMS WG 75**	F	Dicot. weeds incl. *Rumex spp.* And *Taraxacum spp.*	WG	750 g/kg	Foliar		1	-	0.01125-0.0225	200-400	0.045		7 for fresh grass, 21 for hay	

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0 should be given in column 1

** Use also code numbers according to Annex I of Regulation (EU) No 396/2005

*** F: professional field use, Fn: non-professional field use, Fpn: professional and non-professional field use, G: professional greenhouse use, Gn: non-professional greenhouse use, Gpn: professional and non-professional greenhouse use, I: indoor application

NEU= Northern Europe; SEU= Southern Europe

NR= not relevant

New studies on the magnitude of residues are submitted by the applicant in the framework of this application.

In this document, the critical GAP used for setting the EU MRL is compared to critical GAP for the formulated product.

Table 6.3.2- 2: Amidosulfuron: summary of critical GAPs used for setting the EU MRL on pastures/meadows

Crop	F, G or I*	Region	Growth stage	Maximum Number of Applications	Maximum Rate (g a.s./ha)	PHI (days)
Pastures, meadows	F	N-EU and S-EU	-	1	45	7 for fresh grass, 21 for hay

* F Field; G Greenhouse; I Indoor.

The Residue Trial Tables can be found in the document below. They include the supplementary trials presented in this dossier in support of the formulated product that were not already presented and evaluated during the Annex I inclusion of amidosulfuron.

Supplementary residue trials

A total of 15 supplementary residue trials were conducted on grass with Amidosulfuron WG 75 formulation in Northern and Southern Europe. The trials were conducted during the 2010, 2012 and 2014 growing seasons. The purpose of the studies was to determine the residue levels of amidosulfuron and its metabolites AE F101630 (desmethyl-amidosulfuron) and amidosulfuron-guanidine after a spray application of 45 g a.s./Ha. These trials cover the use supported in this dossier.

Table 6.3.2- 3: Number of residue trials conducted on grass with Amidosulfuron WG 75 per geographical region and vegetation period

Crop	Region	Formulation	Number of Trials					Report-No.	Dossier Ref.
			Vegetation period				Total		
			1996	2000	2012	2014*			
Grass	N-EU	WG	1	1		4	6	BKA/629/96/RES BKA/630/96/RES 10-2096 12-2154 14-2009 14-2053	KCA 6.3.2/10-15
	S-EU	WG	2	2	4	4	12		

N-EU: northern Europe S-EU: southern Europe

* 4 trials were recommended by EFSA in Northern and Southern Europe according to residue definition modified in EFSA Journal 2014;12(3):3614

Report:	KCA 6.3.2/10 [redacted],; 1997; M-358859-01-1
Title:	Determination of amidosulfuron (HOE 075032) in rye grass following post emergence application in Southern France - 1996
Report No.:	BKA/629/96/RES
Document No.:	M-358859-01-1
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.3.2/11 [redacted],; 1997; M-358860-01-1
Title:	Determination of amidosulfuron (HOE 075032) in white clover following post emergence application in France - 1996
Report No.:	BKA/630/96/RES
Document No.:	M-358860-01-1
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Material and methods
Three field trials were conducted in/on grass and clover during the 1996 growing season in northern (1 trial) and southern France (2 trials).

The formulation WG75, a wettable granule formulation containing 750 g/kg of amidosulfuron, was applied once with an application rate of 45-48 g/ha amidosulfuron at different growth stages.

Samples of grass sprouts were taken for analysis at the day of treatment and at various intervals up until 21 days in order to investigate the residue decline. The samples were analysed for the parent compound only.

For data gathering purposes the samples taken from these trials were analysed for parent amidosulfuron using method A50084 ([redacted]; 1992; M-131119-01-1) with a limit of quantification of 0.05 mg/kg for shoot. Please refer to MCA Section 4.

Findings:
- Mean concurrent recoveries were within the acceptable range of 70-110 %, RSD <20% as shown in Table below. They validate the study results.

Table 6.3.2- 4: Concurrent recoveries in/on white clover and ryegrass for amidosulfuron

Sample Material	FL [mg/kg]	Single Values [%]		Mean Value [%]	RSD [%]	LOQ [mg/kg]
White Clover (Shoot)	0.05	70	85	78	-	0.05
	0.50	85	81	83	-	
	Overall Recovery (n = 4)			80	8.8	
Ryegrass (Shoot)	0.05	80	79	85	-	0.05
	0.50	80	87	84	-	
	Overall Recovery (n = 4)			84	6.4	

FL = Fortification Level, RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification.

- Storage period of samples:
The longest period of time for which samples from these trials were stored prior to analysis is given in **Table 6.3.2- 5**. The maximum storage period of samples is covered by the storage stability data.

Table 6.3.2- 5: Maximum storage period of samples from trials from BKA/629/96/RES and BKA/630/96/RES

Compound	Crop	Sample material	Maximum storage period (days)	Duration Covered (days)
Amidosulfuron	White Clover	Shoot	202	720

- Residue results:
No residues above the LOQ of 0.05 mg/kg (shoot) could be detected in any of the control samples.

Residues of amidosulfuron in shoots (white clover and ryegrass) at the day of application were ranging from 1.17 to 3.79 mg/kg. These residues declined to values below the analytical limit of quantification after 20-21 days (0.05 mg/kg).

Residue levels in treated samples are summarised in **Table 6.3.2- 6**.

Table 6.3.2- 6: Residues of amidosulfuron from residue trials conducted on grass

Study **Trial No.** **Plot No.** **GLP** **Year**	**Crop Variety**	**Country**	**Application**					**Residues (mg/kg)**		
			FL	**No**	**kg/ha (a.s.)**	**kg/hL (a.s.)**	**GS**	**Portion analysed**	**DALT (days)**	**Amido sulfuron**
BKA/629/96/ RES BKA/629/96/ RES 1 GLP: yes 1996	Ryegrass Sirano	France 82230 Monclar de Quercy Europe, South	75 WG	1	0.0479	0.0186	33	shoot	0 7 14 21	3.79 1.66 0.367 <0.05
BKA/630/96/ RES BKA/630/96/ RES 2 GLP: yes 1996	Clover, white Aram	France 82230 Monclar de Quercy Europe, South	75 WG	1	0.0455	0.0146	06	shoot	0 0* 7 7* 14 14* 21 21*	1.17 1.46 0.491 0.467 0.653 0.506 <0.05 <0.05
BKA/630/96/ RES BKA/630/96/ RES 1 GLP: yes 1996	Clover, white Huia	France 80250 Remien-court Europe, North	75 WG	1	0.0466	0.0166	Four stems, 15 cm, some flowers	shoot	0 7 14 20	1.72 0.109 <0.05 <0.05

* Duplicate sampling, mean at DALT 0=1.32 mg/kg, mean at DALT 7=0.48 mg/kg, mean at DALT 14=0.58 mg/kg, mean at DALT 21=<0.05 mg/kg

Conclusion
Three supervised field trials on white clover and ryegrass were conducted in northern and southern France during the 1996 growing season, under similar application conditions than the intended use pattern with the product Amidosulfuron WG75 product.

The formulation was applied once with an application rate of 45-48 g/ha amidosulfuron.

All trials were conducted according to GLP.

As shown in **Table 6.3.2- 6**, residues of amidosulfuron in shoots (white clover and ryegrass) at the day of application were ranging from 1.17 to 3.79 mg/kg. These residues declined to values below the analytical limit of quantification after 20-21 days (0.05 mg/kg).

Three supplementary trials were conducted in 2010 in Southern or Northern France and in Spain. They are summarised below.

Report:	KCA 6.3.2/12[redacted],; 2012; M-416644-02-1
Title:	Amendment no. 1 to determination of the residues of amidosulfuron in/on grass after spraying of amidosulfuron WG 75 in the field in France (North and South) and Spain
Report No.:	10-2096
Document No.:	M-416644-02-1
Guideline(s):	EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8 Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s):	none
GLP/GEP:	**yes**

Material and methods
Three field trials were conducted in/on grass and clover during the 2010 growing season in France (South and North) (2 trials) and in Spain (1 trial).

The formulation WG75, a wettable granule formulation containing 750 g/kg of amidosulfuron, was applied once with an application rate of 45-48 g/ha amidosulfuron at growth stages 30-32.

Samples of green material were taken for analysis at the day of treatment and at various intervals up to 21 days in order to investigate the residue decline. The samples were analysed for the parent compound and the metabolite AE F101630 using method BE-P002-P09-02 with a limit of quantitation of 0.01 mg/kg.

Findings:
- Method performance: Mean recoveries were within the acceptable range of 70-110 %, RSD <20% as shown in **Table 6.3.2- 7** and **Table 6.3.2- 8**. They validate the study results.

Table 6.3.2- 7: Recovery data for AE F101630

Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
grass / green material	0.01	101; 105; 105; 111; 118	108	6.1	0.01
	0.10	96; 103; 105; 114; 116	107	7.7	
	1.0	82	-	-	
		Overall recovery (n = 11)	**105**	**9.7**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification Fortified with AE F101630, determined as AE F101630 and calculated as AE F101630
These recoveries were performed during the conduct of the study 10-2096

Table 6.3.2- 8: Recovery data for amidosulfuron

Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
grass / green material	0.01	94; 96; 98; 99; 100	97	2.5	0.01
	0.10	90; 93; 93; 98; 102	95	5.0	
	1.0	81	-	-	
	5.0	104			
	7.5	74	-	-	
		Overall recovery (n = 13)	**94**	**8.9**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification Fortified with amidosulfuron, determined as amidosulfuron and calculated as amidosulfuron.
These recoveries were performed during the conduct of the study 10-2096

- Storage stability: The storage periods of deep-frozen samples for amidosulfuron and its metabolite AE F101630 ranged between 388 and 417 days and are covered by the storage stability study.

- Residue results:
No residues above the LOQ of 0.01 mg/kg (green material) could be detected in any of the control samples.

Residues of amidosulfuron and AE F101630 in green material at the day of application were ranging from 1.4 to 2.5 mg/kg and from 0.05 to 0.14 mg/kg respectively. Residues of amidosulfuron declined to values up to 0.08 mg/kg after 21 days and residues of AE F101630 ranged from 0.02 mg/kg to 0.14 mg/kg.

Use patterns and Residue levels in treated samples are summarised in **Table 6.3.2- 9**.

Table 6.3.2- 9: Residues of amidosulfuron and AE F101630 from residue trials conducted on grass in 2010

Study Trial No. GLP Year	Crop Variety	Country	Application					Residues (mg/kg)			
			FL	No	kg/ha (a.s.)	kg/hl (a.s.)	GS	Portion analysed	DALT (days)	Amido sulfuron	AE F101630
10-2096 10-2096-01 GLP: yes 2010	Meadow	France 95420 Magy en Vexin Europe, North	75 WG	1	0.045	-	31	Green material	0 7 14 21	2.5 1.0 0.71 0.08	0.14 0.37 0.31 0.14
10-2096 10-2096-02 GLP: yes 2010	Reduncа	France 79120 Vancais Europe, South	75 WG	1	0.045	-	30	Green material	0 7 14 21	2.4 0.20 0.07 0.01	0.11 0.04 0.03 0.02
10-2096 10-2096-03 GLP: yes 2010	-	Spain 08520 Llerona – Can Ruqueries Europe, South	75 WG	1	0.048	-	32	Green material	0 6 13 21	1.4 0.24 0.04 < 0.01	0.05 0.07 0.04 0.02

Conclusion
Three supervised field trials on grass were conducted in Southern or Northern France or in Spain during the 2010 growing season, under similar application conditions than the intended use pattern with the Amidosulfuron WG75 product.

The formulation was applied once with an application rate of 45-48 g/ha amidosulfuron.

All trials were conducted according to GLP.

As shown in **Table 6.3.2- 10**, residues of amidosulfuron and AE F101630 in green material at the day of application were ranging from 1.4 to 2.5 mg/kg and from 0.05 to 0.14 mg/kg respectively. Residues of amidosulfuron declined to values up to 0.08 mg/kg after 21 days and residues of AE F101630 ranged from 0.02 mg/kg to 0.14 mg/kg.

Table 6.3.2- 10: Residues on grass after application of Amidosulfuron WG 75

Application	Analyte	Crop	No of trials	Sample material	DALT	Residue [mg/kg] min - max
Northern Europe						
30 g amidosulfuron/Ha PHI=7 days	amidosulfuron	grass	1	green material	0 7 14 21	2.5 1.0 0.71 0.08
	AE F101630		1	green material	0 7 14 21	0.14 0.37 0.31 0.14
Southern Europe						
30 g amidosulfuron/Ha PHI=7 days	amidosulfuron	grass	2	green material	0 7 14 21	1.4-2.4 0.20-0.24 0.04-0.07 <0.01-0.01
	AE F101630		2	green material	0 6-7 13-14 21	0.05-0.11 0.04-0.07 0.03-0.04 0.02

DALT = Days after last treatment

Four supplementary trials were conducted in 2012 in Southern Europe. They are summarised below.

Report:	KCA 6.3.2/13[redacted],;[redacted]; 2013; M-473282-01-1
Title:	Determination of the residues of amidosulfuron in/on grass after spray application of amidosulfuron WG 75 in Spain, southern France and Italy
Report No.:	12-2154
Document No.:	M-473282-01-1
Guideline(s):	Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market and repealing Council Directives 79/117/EEC and 91/414/EEC EC guidance working document 7029/VI/95 rev. 5 (July 22, 1997) OECD 509 Adopted 2009-09-07, OECD GUIDELINE FOR THE TESTING OF CHEMICALS, Crop Field Trial US EPA OCSPP Guideline No. 860.1500
Guideline deviation(s):	none
GLP/GEP:	**yes**

Four residue trials were done with the formulation Amidosulfuron WG 75, a WG (Water dispersible granules) formulation containing 75 % amidosulfuron during the 2012 growing seasons. The residue trials were carried out in the field in Spain (2), Southern France and Italy for Southern Europe.

The formulation was applied once with an application rate of 0.06 kg/ha and 300-400 L water per ha, corresponding to a spray concentration of 0.011-0.015 % and 0.045 kg/ha of amidosulfuron.

The application was carried out at growth stages BBCH between 13 and 31.

Green material and hay of grass were taken between days 0 prior to the application and 21, including 0, 7 and 14 days after the last application (DALT) in order to investigate the residue decline. Residues of amidosulfuron and its metabolite AE F101630 in these matrices were determined according to method BE-002-P09-02 (BCS internal no. 01325) with a limit of quantitation of 0.01 mg/kg.

Findings

- Method performance: The average recoveries were performed at fortification levels between 0.01 and 10 mg/kg per analyte for amidosulfuron and its metabolite AE F101630 and were within the acceptable range of 70 - 110% (except for the fortification of hay with AE F101630 at LOQ level (112%)).

As the RSD values were below 20% as shown in **Table 6.3.2- 11** and **Table 6.3.2- 12**, there is no major impact on the study results.

Table 6.3.2- 11: Recovery data for amidosulfuron

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
grass / green material	0.01	80; 80; 81	80	0.7	0.01
	0.10	72; 81; 83	79	7.4	
	10	77	-	-	
		Overall recovery (n = 7)	**79**	**4.6**	
grass / hay	0.01	78; 82; 95	85	10.5	0.01
	0.10	80; 83; 83	82	2.1	
	9.9	71	-	-	
		Overall recovery (n = 7)	**82**	**8.8**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with amidosulfuron, determined as amidosulfuron and calculated as amidosulfuron
These recoveries were performed during the conduct of the study 12-2154.

Table 6.3.2- 12: Recovery data for AE F101630

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
grass / green material	0.01	72; 73; 75	73	2.1	0.01
	0.10	76; 79; 83	79	4.4	
	10	116	-	-	
		Overall recovery (n = 7)	**82**	**18.8**	
grass / hay	0.01	110; 113	112	-	0.01
	0.10	96; 108; 114	106	8.6	
	9.9	94	-	-	
		Overall recovery (n = 6)	**106**	**8.2**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with AE F101630, determined as AE F101630 and calculated as AE F101630
These recoveries were performed during the conduct of the study 12-2154.

- Storage stability: The maximum storage periods of deep-frozen samples for amidosulfuron and its metabolite AE F101630 ranged between 369 and 445 days and are covered by the storage stability studies (cf. MCA Point 6.1.).

Table 6.3.2- 13: amidosulfuron and its metabolite AE F101630 in/on grass - Storage period of samples

Study No.	Trial SubID	Targets crop	Sample Material	Substance	Storage Period [Max. days]	Dates of Sampling to Analysis
Southern Europe						
12-2154	12-2154-01	grass	green material	amidosulfuron and AE F101630	370	2012-09-12 to 2013-09-17
	12-2154-02	grass	green material	amidosulfuron and AE F101630	445	2012-06-29 to 2013-09-17
	12-2154-03	grass	green material	amidosulfuron and AE F101630	409	2012-08-04 to 2013-09-17
	12-2154-04	grass	green material	amidosulfuron and AE F101630	369	2012-09-13 to 2013-09-17

Use patterns for the trials conducted on grass are listed in **Table 6.3.2- 14**. Residues of the parent compound amidosulfuron and its metabolite AE F101630 are summarised below in **Table 6.3.2- 15**.

- Residue results:

The apparent residues in the control samples were below the LOQ. Recoveries were not corrected for apparent residues in the control samples used for these recoveries.

Table 6.3.2- 14: Actual use patterns of residue trials conducted in/on grass with the formulation amidosulfuron WG 75

Study, Trial No., Trial SubID, GLP, Year	Crop Variety	Country	Application						
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	Water rate (L/ha)	Spray interval (days)	GS
Southern Europe									
12-2154 12-2154-01 GLP: yes 2012	Grass Pasto de Sudan	Spain	WG 75	1	0.045	0.015	300	-	13
12-2154 12-2154-02 GLP: yes 2012	Grass Axis	France	WG 75	1	0.045	0.015	300		31
12-2154 12-2154-03 GLP: yes 2012	Grass common meadow grass mixture	Italy	WG 75	1	0.045	0.011	400	-	30
12-2154 12-2154-04 GLP: yes 2012	Grass Pasto de Sudan	Spain	WG 75	1	0.045	0.015	300	-	13

FL: Formulation No: number of applications a.s.: active substance
GS: growth stage (BBCH code) at application

Table 6.3.2- 15: Residues of amidosulfuron and its metabolite AE F101630 in/on grass, treated with the formulation amidosulfuron WG 75

Trial No. Country, GLP, Year	Sample material	Growth stage (BBCH) at sampling	DALT (days)	Residues [mg/kg]	
				amidosulfuron	AE F101630
Southern Europe					
12-2154 12-2154-01 GLP: yes 2012	green material	13	0	4.0	0.043
		19	7	0.61	0.15
		40	13	0.41	0.21
		59	20	0.013	0.17
	hay	59	20	0.014	0.56
12-2154 12-2154-02 GLP: yes 2012	green material	31	0	2.8	0.45
		52	7	< 0.01	0.038
		57	14	< 0.01	0.041
		59	21	< 0.01	0.024
	hay	59	21	< 0.01	0.17
12-2154 12-2154-03 GLP: yes 2012	green material	30	0	2.6	0.061
		37	7	0.87	0.28
		51	14	0.56	0.27
		59	21	0.34	0.11
	hay	59	21	0.24	0.23
12-2154 12-2154-04 GLP: yes 2012	green material	13	0	0.87	0.025
		19	8	0.35	0.031
		40	13	0.17	0.031
		59	20	<0.01	<0.01
	hay	59	20	<0.01	0.048

DALT = Days after last treatment

Analyte:	Final determination as:	Residues calculated as:
amidosulfuron AE F101630	amidosulfuron AE F101630	amidosulfuron AE F101630

Conclusion

To support the use of Amidosulfuron WG 75 formulation in/on grass, 4 residue trials were conducted in Southern Europe during the 2012 growing season, at the corresponding use pattern. The formulation WG 75 was applied once with an application rate of 45 g/ha amidosulfuron and a DALT of 7 days for fresh grass and 21 days for hay.

The results of amidosulfuron and its metabolite AE F101630 are summarised below.
Residues of amidosulfuron ranged between <0.01-0.87 mg/kg in fresh grass at a PHI of 7 days and between < 0.01 - 0.24 mg/kg in hay at a PHI of 21 days. Residues of desmethyl-amidosulfuron ranged between 0.031 - 0.28 mg/kg in fresh grass at a PHI of 7 days and between 0.048 - 0.56 mg/kg in hay at a PHI of 21 days.

Table 6.3.2- 16: Residues in/on grass after application of Amidosulfuron WG 75

Analyte	Crop	No of trials	Sample material	Growth stage [BBCH]	DALT	Residue [mg/kg] min - max
amidosulfuron	grass	4	green material	13 - 31	0	0.87 - 4.0
				19 - 52	7 - 8	< 0.01 - 0.87
				40 - 57	13 - 14	< 0.01 - 0.56
				59	20 - 21	< 0.01 - 0.34
			hay	59	20 - 21	< 0.01 - 0.24
AE F101630	grass	4	green material	13 - 31	0	0.025 - 0.45
				19 - 52	7 - 8	0.034 - 0.28
				40 - 57	13 - 14	0.031 - 0.27
				59	20 - 21	0.01 - 0.17
			hay	59	20 - 21	0.048 - 0.56

DALT = Days after last treatment

Analyte:	Final determination as:	Residues calculated as:
amidosulfuron	amidosulfuron	amidosulfuron
AE F101630	AE F101630	AE F101630

To support the use of Amidosulfuron WG 75 formulation in/on grass, 8 new supplementary residues trials were conducted in Northern and Southern Europe in 2014. The analysis has been done according to residue definition set by EFSA, 2014.

Report: KCA 6.3.2/14[redacted]1; [redacted]; 2016; M-546207-01-1
Title: Determination of the residues of amidosulfuron in/on grass after spray application of amidosulfuron WG 75 in Germany, the United Kingdom, the Netherlands and Belgium
Report No.: 14-2009
Document No.: M-546207-01-1
Guideline(s): REGULATION (EC) No 1107/2009 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 21 October 2009 concerning the placing of plant protection products on the market
OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009)
US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Guideline deviation(s): none
GLP/GEP: **no**

Four residue trials were done with the formulation Amidosulfuron WG 75, a WG (Water dispersible granules) formulation containing 75 % amidosulfuron during the 2014 growing season. The residue trials were carried out in the field in Germany, The United Kingdom, The Netherlands and Belgium for Northern Europe.

The formulation was applied once with an application rate of 0.06 kg/ha and 200-400 L water per ha, corresponding to a spray concentration of 0.011-0.023 % and 0.045 kg/ha of amidosulfuron.

The application was carried out at growth stages BBCH 19 to 33.

Green material of grass was taken between days 0 prior to the application and 21 days, including 0, 7 and 14 days after the last application (DALT) in order to investigate the residue decline. 21 days after the application, hay was prepared by drying of green material. Residues of amidosulfuron and its

metabolites AE F101630 and amidosulfuron-guanidine in these matrices were determined according to method 01325 (compiling SMM/LIMS methods 01325 (former BE-002-P09-02) and 01325/M001) with a limit of quantitation of 0.01 mg/kg.

Findings

- Method performance: The average recoveries were performed at fortification levels between 0.01 and 4.0 mg/kg per analyte for amidosulfuron and its metabolite AE F101630 and amidosulfuron-guanidine were within the acceptable range of 70 – 110%. As the RSD values were below 20% as shown in **Table 6.3.2- 17** to **Table 6.3.2- 19**, there is no major impact on the study results.

Table 6.3.2- 17: Recovery data for amidosulfuron

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
grass / green material	0.01	65; 68; 77	70	8.9	0.01
	0.10	65; 66; 77; 83; 85	75	12.4	
	1.0	95	-	-	
	4.0	91	-	-	
		Overall recovery (n = 10)	**77**	**14.4**	
grass / hay	0.01	101; 102; 103	102	1.0	0.01
	0.10	99; 101; 104	101	2.5	
	4.0	96	-	-	
		Overall recovery (n = 7)	**101**	**2.6**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with amidosulfuron, determined as amidosulfuron and calculated as amidosulfuron
These recoveries were performed during the conduct of the study 14-2009.

Table 6.3.2- 18: Recovery data for AE F101630

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
grass / green material	0.01	72; 72; 79	74	5.4	0.01
	0.10	70; 75; 82; 91; 102	84	15.2	
	1.0	108	-	-	
	4.0	106	-	-	
		Overall recovery (n = 10)	**86**	**17.4**	
grass / hay	0.01	83; 86; 89	86	3.5	0.01
	0.10	89; 92; 95	92	3.3	
	4.0	98	-	-	
		Overall recovery (n = 7)	**90**	**5.7**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with AE F101630, determined as AE F101630 and calculated as AE F101630
These recoveries were performed during the conduct of the study 14-2009.

Table 6.3.2- 19: Recovery data for amidosulfuron-guanidine

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
grass / green material	0.01	105; 108; 110	108	2.3	0.01
	0.10	97; 102; 102; 103; 107	102	3.5	
	1.0	74	-	-	
	4.0	83	-	-	
		Overall recovery (n = 10)	**99**	**11.8**	
grass / hay	0.01	91; 111; 116	106	12.5	0.01
	0.10	98; 102; 107	102	4.4	
	4.0	78	-	-	
		Overall recovery (n = 7)	**100**	**12.8**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with amidosulfuron-guanidine, determined as amidosulfuron-guanidine and calculated as amidosulfuron
These recoveries were performed during the conduct of the study 14-2009.

- Storage stability: The maximum storage periods of deep-frozen samples for amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine ranged between 369 and 481 days and are covered by the storage stability studies (cf. MCA Point 6.1.).

Table 6.3.2- 20: amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine in/on grass - Storage period of samples

Study No.	Trial SubID	Targets crop	Sample Material	Substance	Storage Period [Max. days]	Dates of Sampling to Analysis
Northern Europe						
14-2009	14-2009-01	grass	green material	amidosulfuron and metabolites	481	2014-04-10 to 2015-08-04
			hay	amidosulfuron and metabolites	463	2014-05-06 to 2015-08-12
	14-2009-02	grass	green material	amidosulfuron and metabolites	383	2014-07-17 to 2015-08-04
			hay	amidosulfuron and metabolites	369	2014-08-08 to 2015-08-12
	14-2009-03	grass	green material	amidosulfuron and metabolites	462	2014-05-05 to 2015-08-10
			hay	amidosulfuron and metabolites	439	2014-05-30 to 2015-08-12
	14-2009-04	grass	green material	amidosulfuron and metabolites	444	2014-05-23 to 2015-08-10
			hay	amidosulfuron and metabolites	418	2014-06-20 to 2015-08-12

Use patterns for the trials conducted on grass are listed in **Table 6.3.2- 21**. Residues of the parent compound amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine are summarised below in **Table 6.3.2- 22.**

- Residue results:
The apparent residues in the control samples were below the LOQ. Recoveries were not corrected for apparent residues in the control samples used for these recoveries.

Table 6.3.2- 21: Actual use patterns of residue trials conducted in/on grass with the formulation amidosulfuron WG 75

Study, Trial No., Trial SubID, GLP, Year	Crop Variety	Country	Application						
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	Water rate (L/ha)	Spray interval (days)	GS
Northern Europe									
14-2009 14-2009-01 GLP: yes 2014	Grass Standard G V	Germany	WG 75	1	0.045	0.0150	300	-	33
14-2009 14-2009-02 GLP: yes 2014	Grass No 9 Italian rye grass	United kingdom	WG 75	1	0.045	0.0225	200	-	29
14-2009 14-2009-03 GLP: yes 2014	Grass unknown cow feeding	The Netherlands	WG 75	1	0.045	0.0113	400	-	19
14-2009 14-2009-04 GLP: yes 2014	Grass Stargreen Ryegrass	Belgium	WG 75	1	0.045	0.0150	300	-	31

FL: Formulation No: number of applications a.s.: active substance
GS: growth stage (BBCH code) at application

Table 6.3.2- 22: Residues of amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine in/on grass, treated with the formulation amidosulfuron WG 75 – Northern Europe

Trial No. Country, GLP, Year	Sample material	Growth stage (BBCH) at sampling	DALT (days)	Residues [mg/kg]		
				amidosulfuron	AE F101630	amidosulfuron-guanidine
Northern Europe						
14-2009 14-2009-01 GLP: yes 2014	green material	33	0	1.9	0.069	0.057
		38	7	0.44	0.11	0.071
		51	14	0.19	0.11	0.073
		59	22	<0.01	0.031	0.028
	hay	59	22	0.036	0.14	0.11
14-2009 14-2009-02 GLP: yes 2014	green material	29	0	2.2	0.046	0.041
		31	6	0.034	0.043	0.048
		31	13	0.01	0.038	0.045
		51	22	0.01	0.021	0.030
	hay	51	22	< 0.01	0.11	0.11
14-2009 14-2009-03 GLP: yes 2014	green material	19	0	2.9	0.017	<0.01
		19	7	< 0.01	0.041	<0.01
		51	14	< 0.01	0.021	<0.01
		59	21	< 0.01	0.017	<0.01
	hay	59	21	< 0.01	0.059	0.020
14-2009 14-2009-04 GLP: yes 2014	green material	31	0	2.5	0.13	0.13
		33	7	0.15	0.10	0.062
		51	14	< 0.01	0.045	0.027
		59	21	<0.01	0.025	0.015
	hay	59	21	<0.01	0.15	0.080

DALT = Days after last treatment

Analyte:	Final determination as:	Residues calculated as:
amidosulfuron	amidosulfuron	amidosulfuron
AE F101630	AE F101630	AE F101630
amidosulfuron-guanidine	amidosulfuron-guanidine	amidosulfuron

Conclusion

To support the use of Amidosulfuron WG 75 formulation in/on grass, 4 residue trials were conducted in Northern Europe during the 2014 growing season, at the corresponding use pattern. The formulation WG 75 was applied once with an application rate of 45 g/ha amidosulfuron and an envisaged DALT of 7 days for fresh grass and 21 days for hay.

Residues of amidosulfuron ranged between < 0.01 - 0.44 mg/kg in fresh grass at a PHI of 7 days and ranged between < 0.01 – 0.036 mg/kg in hay at a PHI of 21 days.

Residues of desmethyl-amidosulfuron (AE F101630) ranged between 0.041 - 0.11 mg/kg in fresh grass at a PHI of 7 days and ranged between 0.059 - 0.15 mg/kg in hay at a PHI of 21 days.

Residues of amidosulfuron-guanidine ranged between < 0.01 - 0.071 mg/kg in fresh grass at a PHI of 7 days and ranged between 0.020 – 0.11 mg/kg in hay at a PHI of 21 days.

The results of amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine are summarised below.

Table 6.3.2- 23: Residues in/on grass after application of Amidosulfuron WG 75, Northern Europe

Analyte	Crop	No of trials	Sample material	Growth stage [BBCH]	DALT	Residue [mg/kg] min - max
amidosulfuron	grass	4	green material	19 - 33	0	1.9 - 2.9
				19 - 38	6/7	< 0.01 - 0.44
				31 - 51	13/14	< 0.01 - 0.19
				51-59	21/22	< 0.01
			hay	51-59	21/22	< 0.01 - 0.036
AE F101630	grass	4	green material	19 - 33	0	0.015 - 0.13
				19 - 38	6/7	0.041 - 0.11
				31 - 51	13/14	0.021 - 0.11
				51-59	21/22	0.019 - 0.031
			hay	51-59	21/22	0.059 - 0.15
amidosulfuron-guanidine	grass	4	green material	19 - 33	0	< 0.01 - 0.13
				19 - 38	6/7	< 0.01 - 0.071
				31 - 51	13/14	< 0.01 - 0.077
				51-59	21/22	< 0.01 - 0.030
			hay	51-59	21/22	0.020 – 0.11

DALT = Days after last treatment

Analyte:	Final determination as:	Residues calculated as:
amidosulfuron	amidosulfuron	amidosulfuron
AE F101630	AE F101630	AE F101630
amidosulfuron-guanidine	amidosulfuron-guanidine	amidosulfuron

Report: KCA 6.3.2/15 [redacted]; [redacted]; 2016; M-546876-01-1
Title: Determination of the residues of amidosulfuron in/on grass after spray application of amidosulfuron WG 75 in Greece, Spain and Italy
Report No.: 14-2053
Document No.: M-546876-01-1
Guideline(s): REGULATION (EC) No 1107/2009 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 21 October 2009 concerning the placing of plant protection products on the market
OECD Guideline for the Testing of Chemicals on Crop Field Trial (TG 509 published in September 2009)
US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Guideline deviation(s): none
GLP/GEP: **yes**

Four residue trials were done with the formulation Amidosulfuron WG 75, a WG (Water dispersible granules) formulation containing 75 % amidosulfuron during the 2014 growing seasons. The residue trials were carried out in the field in Greece, Spain (2) and Italy for Southern Europe.

The formulation was applied once with an application rate of 0.06 kg/ha in a range of 250-400 L water per ha, corresponding to a spray concentration of 0.011-0.018 % and 0.045-0.49 kg/ha of amidosulfuron.

The application was carried out at growth stages BBCH 13 to 30.
Green material and hay of grass were taken between days 0 prior to the application and 21 days, including 0, 7 and 14 days after the last application (DALT) in order to investigate the residue decline. Residues of amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine in these matrices were determined according to method 01325 (compiling SMM/LIMS methods 01325 (former BE-002-P09-02) and 01325/M001) with a limit of quantitation of 0.01 mg/kg.

Findings
- Method performance:
Recoveries were not corrected for apparent residues in the control samples used for these recoveries.
The average recoveries were within the range of 70 – 110% except for amidosulfuron in green material at a fortification level of 0.01 and 0.10 mg/kg (119% and 113 respectively). This is accepted according to the OECD guideline. The RSD values were below 20%.
The results are summarised below.

Table 6.3.2- 24: Recovery data for amidosulfuron

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
grass / green material	0.01	118,119	119	-	0.01
	0.10	106, 111,115, 119	113	4.9	
	1.0	113		-	
	4.0	101		-	
		Overall recovery (n = 8)	**113**	**5.8**	
grass / hay	0.01	104	-	-	0.01
	0.10	94,97	96	-	
	1.0	85	-	-	
		Overall recovery (n = 4)	**95**	**8.3**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with amidosulfuron, determined as amidosulfuron and calculated as amidosulfuron
These recoveries were performed during the conduct of the study 14-2053.
Accepted according to OECD guideline; mean is 70 – 120%; RSD < 20%

Table 6.3.2- 25: Recovery data for AE F101630

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
grass / green material	0.01	101,113	107	-	0.01
	0.10	101,104,104,112	105	4.5	
	1.0	100	-	-	
	4.0	101	-	-	
		Overall recovery (n = 8)	**105**	**4.9**	
grass / hay	0.01	108	-	-	0.01
	0.10	106,103	105	-	
	1.0	96	-	-	
		Overall recovery (n = 4)	**103**	**5.1**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with AE F101630, determined as AE F101630 and calculated as AE F101630
These recoveries were performed during the conduct of the study 14-2053.

Table 6.3.2- 26: Recovery data for amidosulfuron-guanidine

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
grass / green material	0.01	100; 103	102	-	0.01
	0.10	99; 99; 100; 102	100	1.4	
	1.0	98	-	-	
	4.0	88	-	-	
		Overall recovery (n = 8)	**99**	**4.7**	
grass / hay	0.01	91	-	-	0.01
	0.10	96; 101	99	-	
	1.0	98	-	-	
		Overall recovery (n = 4)	**97**	**4.4**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with amidosulfuron-guanidine, determined as amidosulfuron-guanidine and calculated as amidosulfuron
These recoveries were performed during the conduct of the study 14-2053.

- Storage stability: The maximum storage periods of deep-frozen samples for amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine ranged between 335 and 427 days and are covered by the storage stability studies (cf. MCA Point 6.1.).

Table 6.3.2- 27: amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine in/on grass - Storage period of samples

Study No.	Trial SubID	Targets crop	Sample Material	Substance	Storage Period [Max. days]	Dates of Sampling to Analysis
Southern Europe						
14-2053	14-2053-01	Sudan Grass	green material	amidosulfuron and metabolites	413	2014-07-02 to 2015-08-19
			hay	amidosulfuron and metabolites	399	2014-07-23 to 2015-08-26
	14-2053-02	Sorghum	green material	amidosulfuron and metabolites	427	2014-06-18 to 2015-08-19
			hay	amidosulfuron and metabolites	414	2014-07-08 to 2015-08-26
	14-2053-03	Sorghum	green material	amidosulfuron and metabolites	413	2014-07-03 to 2015-08-20
			hay	amidosulfuron and metabolites	399	2014-07-23 to 2015-08-20
	14-2053-04	Grass	green material	amidosulfuron and metabolites	350	2014-09-04 to 2015-08-20
			hay	amidosulfuron and metabolites	335	2014-09-25 to 2015-08-26

Use patterns for the trials conducted on grass are listed in **Table 6.3.2- 28**. Residues of the parent compound amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine are summarised below in **Table 6.3.2- 29.**

- Residue results:
The apparent residues in the control samples used for the performance of recoveries were below 30% of the LOQ.

Table 6.3.2- 28: Actual use patterns of residue trials conducted in/on grass with the formulation amidosulfuron WG 75

Study, Trial No., Trial SubID, GLP, Year	Crop Variety	Country	Application						
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	Water rate (L/ha)	Spray interval (days)	GS
Southern Europe									
14-2053 14-2053-01 GLP: yes 2014	Sudan Grass Topsilo	Greece	WG 75	1	0.045	0.011	400	-	18
14-2053 14-2053-02 GLP: yes 2014	Sorghum Sudanense	Spain	WG 75	1	0.049	0.018	273*	-	13
14-2053 14-2053-03 GLP: yes 2014	Sorghum Sudanense	Spain	WG 75	1	0.045	0.018	250	-	13
14-2053 14-2053-04 GLP: yes 2014	Grass nap	Italy	WG 75	1	0.045	0.011	400	-	30

FL: Formulation No: number of applications a.s.: active substance
GS: growth stage (BBCH code) at application
* 9.33% overdosed.

Table 6.3.2- 29: Residues of amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine in/on grass, treated with the formulation amidosulfuron WG 75 – Southern Europe

Trial No. Country, GLP, Year	Sample material	Growth stage (BBCH) at sampling	DALT (days)	Residues [mg/kg]		
				amidosulfuron	AE F101630	amidosulfuron-guanidine
Southern Europe						
14-2053 14-2053-01 GLP: yes 2014	green material	18	0	2.7	0.068	0.12
		23	7	0.23	0.066	0.096
		51	14	0.011	0.017	0.020
		59	21	<0.01	<0.01	<0.01
	hay	59	21	<0.01	0.025	<0.019
14-2053 14-2053-02 GLP: yes 2014	green material	13	0	1.8	<0.01	0.015
		19	8	0.13	0.075	0.11
		40	14	0.038	0.022	0.033
		59	20	0.011	0.013	0.023
	hay	59	20	<0.01	0.012	0.024
14-2053 14-2053-03 GLP: yes 2014	green material	13	0	1.8	0.095	0.15
		19	7	0.64	0.026	0.040
		40	14	0.093	<0.01	0.011
		59	20	0.018	<0.01	<0.01
	hay	59	20	0.020	<0.01	<0.01
14-2053 14-2053-04 GLP: yes 2014	green material	30	0	2.7	0.50	0.073
		39	7	0.15	0.59	0.071
		49	14	0.047*	0.30*	0.041*
		59	21	0.057*	0.48*	0.065*
	hay	59	21	0.14	0.82	0.21

DALT = Days after last treatment

Analyte:	Final determination as:	Residues calculated as:
amidosulfuron	amidosulfuron	amidosulfuron
AE F101630	AE F101630	AE F101630
amidosulfuron-guanidine	amidosulfuron-guanidine	amidosulfuron

* The samples were extracted and measured twice. Here, mean values are reported.

Conclusion

To support the use of Amidosulfuron WG 75 formulation in/on grass, 4 residue trials were conducted in Northern Europe during the 2014 growing season, at the corresponding use pattern. The formulation WG 75 was applied once with an application rate of 45 g/ha amidosulfuron and a DALT of 0-21 days.

The results of amidosulfuron and its metabolites AE F101630 and amidosulfuron-guanidine are summarised below.

Residues of amidosulfuron ranged between 0.13 - 0.64 mg/kg in fresh grass at a PHI of 7 days and ranged between < 0.01 – 0.14 mg/kg in hay at a PHI of 21 days.

Residues of desmethyl-amidosulfuron ranged between 0.026 - 0.59 mg/kg in fresh grass at a PHI of 7 days and ranged between < 0.01 – 0.82 mg/kg in hay at a PHI of 21 days.

Residues of amidosulfuron-guanidine ranged between 0.040 – 0.11 mg/kg in fresh grass at a PHI of 7 days and ranged between < 0.01 - 0.21 mg/kg in hay at a PHI of 21 days.

Table 6.3.2- 30: Residues in/on grass after application of Amidosulfuron WG 75 - Southern Europe

Analyte	Crop	No of trials	Sample material	Growth stage [BBCH]	DALT	Residue [mg/kg] min - max
amidosulfuron	grass	4	green material	13 - 30	0	1.8 - 2.7
				19 - 39	7/8	0.13 - 0.64
				40 - 51	14	0.011 - 0.093
				59	20/21	< 0.01 - 0.057
			hay	59	20/21	< 0.01 - 0.14
AE F101630	grass	4	green material	18 - 30	0	< 0.01 - 0.50
				19 - 39	7/8	0.026 - 0.59
				40 - 51	14	< 0.01 - 0.30
				59	20/21	< 0.01 - 0.48
			hay	59	20/21	< 0.01 – 0.82
amidosulfuron-guanidine	grass	4	green material	18 - 30	0	0.015 - 0.15
				19 - 39	7/8	0.040 – 0.11
				40 - 51	14	0.011-0.041
				59	20/21	<0.01 - 0.065
			hay	59	20/21	< 0.01 - 0.21

DALT = Days after last treatment

Analyte:	Final determination as:	Residues calculated as:
amidosulfuron	amidosulfuron	amidosulfuron
AE F101630	AE F101630	AE F101630
amidosulfuron-guanidine	amidosulfuron-guanidine	amidosulfuron

Summary of all supplementary residue trials conducted on grass in Southern and Northern Europe

Table 6.3.2- 31: Summary of all supplementary trials in/on grass after application of Amidosulfuron WG 75

Zone	Crop	No of trials	Sample material	DALT	Residue amidosulfuron [mg/kg]	Residue AE F101630 [mg/kg]	Residue amidosulfuron-guanidine [mg/kg]
Northern Europe							
Northern	grass	6	green material	0	1.72-2.9	1 nd/0.017-0.14	2 nd/< 0.01 - 0.13
				6-7	<0.01-1.0	1 nd/0.041-0.37	2 nd/< 0.01 - 0.071
				13-14	<0.01-0.71	1 nd/0.021-0.31	2 nd/< 0.01 - 0.077
				20-22	<0.01-0.08	1 nd/0.017-0.14	2 nd/< 0.01 - 0.030
			hay	20-22	2 nd/<0.01-0.036	2 nd/0.059-0.15	2 nd/0.020 – 0.11
Southern Europe							
Southern	grass	12	green material	0	0.87-4.0	2 nd/<0.01-0.50	8 nd/0.015 - 0.15
				6-8	<0.01-1.66	2 nd/0.026-0.59	8 nd/0.040 - 0.11
				13-14	<0.01-0.58	2 nd/<0.01-0.30	8 nd/0.011 - 0.041
				20-21	<0.01-0.34	2 nd/<0.01-0.48	8 nd/<0.01 - 0.065
			hay	20-21	4 nd/<0.01-0.24	4 nd/<0.01-0.82	8 nd/< 0.01 - 0.21
Northern and Southern Europe							
Europe	grass	18	green material	0	0.87-4.0	3 nd/<0.01-0.50	10 nd/< 0.01 - 0.15
				6-8	<0.01-1.66	3 nd/0.026-0.59	10 nd/< 0.01 - 0.11
				13-14	<0.01-0.71	3 nd/<0.01-0.31	10 nd/< 0.01 - 0.077
				20-22	<0.01-0.34	3 nd/<0.01-0.48	10 nd/< 0.01 - 0.065
			hay	20-22	6 nd/<0.01-0.24	6 nd/<0.01-0.82	10 nd/<0.01 – 0.21

nd: not determined

Report: KCA 6.3.2/16 [redacted]; 2016; M-555920-01-1
Title: Statement amidosulfuron on pastures
Report No.: M-555920-01-1
Document No.: M-555920-01-1
Guideline(s): --
Guideline deviation(s): --
GLP/GEP: **no**

The position paper (M-555920-01-1) presents an overview of all residue trials conducted on grass. Based on these exhaustive data, tentative MRLs proposed by EFSA on fresh grass in 2014 are discussed: MRL of 1.5 mg/kg in Northern Europe can be confirmed. For Southern Europe, the MRL proposed at 5 mg/kg could be reduced to 3 mg/kg.

CA 6.3.3 Flax/linseed

Studies submitted and evaluated for the first inclusion of amidosulfuron on Annex I:

Report:	KCA 6.3.3/01 [redacted]; [redacted]; 1996; M-134936-01-1
Title:	Amidosulfuron water dispersible granule (WG) 750 g/kg Code Hoe 075032 00 WG75 A109 Determinafion of Residues of Hoe 075032 to establish a Maximum Residue Level following 1 application in linseed / flax European Union (Northern Zone) 1994
Report No.:	A54357
Document No.:	M-134936-01-1
Guideline(s):	--
Guideline deviation(s):	--
GLP/GEP:	**yes**

A total of 3 trials were performed in Northern Europe. All studies were considered acceptable. Amidosulfuron was applied to flax plants (Linum usitatissimum) once per season at a rate of 30 g/ha in order to investigate the residue pattern. The trial locations were spread over main growing areas of the Northern zone thus covering different soils and climatic conditions.

A wettable granule formulation (AE F075032 00 WG75 A1) was used in all trials containing 750 g/kg amidosulfuron.

Samples were taken for analysis at the day of treatment and at harvest. No additional samples were taken at interim growth stages. The samples of seeds, shoots and straw were analysed for the parent compound.

Overview of European residue data for amidosulfuron in flax/linseeds

Crop, Region, Countries (no. of trials), year	Application		Residue			Reference
	Formulation content of a.s.	Rate [kg a.s./ha]	DAT [days]	Part of crop	Amidosulfuron [mg/kg]	
Flax/linseeds, North Germany (1), France (1), UK (1) 1994	WG 750 g/kg	0.030	0 75-85 75-85	Shoot Straw Seeds	0.12-0.39 <0.05 <0.05	[redacted]; [redacted]; 1996; M-134936-01-1 GLP: Yes

Residues of amidosulfuron in shoots at the day of application ranged from 0.12 up to 0.39 mg/kg. At harvest, the residues had decreased to levels of < 0.05 mg/kg in straw and seeds.

"AIR process" - new studies submitted

The critical GAPs with respect to consumer intake and risk assessment for the preparation AMS WG 75 are presented in Table 6.3.3-1.

Table 6.3.3- 1: Critical GAPs

Crop and/ or situation **	Zone	Product code	F, Fn, Fpn G, Gn, Gpn or I***	Pests or Group of pests controlled	Formulation		Application				Application rate per treatment			PHI (days)	Remark
					Type	Conc. of as	method kind	growth stage & season	number min max	interval between applications (min)	kg as/hL min max	water L/ha min max	kg as/ha min max		
Flax/Linseed (use on oilseed and fiber production)	NEU & SEU	**AMS WG 75**	F	Dicot. weeds incl. *Galium, Sinapis arvensis, Raphanus raphanistrum, Capsella, Myosotis, Scandix, Tordylium, Ranunculus*, volunteer oil seed rape and sunflower	WG	750 g/kg	Foliar	49 before flower buds are visible	1	-	0.00375-0.03	100-400	0.015-0.030	90	

* Use number(s) in accordance with the list of all intended GAPs in Part B, Section 0 should be given in column 1
** Use also code numbers according to Annex I of Regulation (EU) No 396/2005
*** F: professional field use, Fn: non-professional field use, Fpn: professional and non-professional field use, G: professional greenhouse use, Gn: non-professional greenhouse use, Gpn: professional and non-professional greenhouse use, I: indoor application
NEU= Northern Europe; SEU= Southern Europe
NR= not relevant

New studies on the magnitude of residues are submitted by the applicant in the framework of this application.

In this document, the critical GAP used for setting the EU MRL is compared to critical GAP for the formulated product.

Table 6.3.3- 2: Amidosulfuron: summary of critical GAPs used for setting the EU MRL on pastures/meadows

Crop	F, G or I*	Region	Growth stage	Maximum Number of Applications	Maximum Rate (g a.s./ha)	PHI (days)
Flax/linseed	F	N-EU and S-EU	49	1	30	90

* F Field; G Greenhouse; I Indoor.

The Residue Trial Tables can be found in the document below. They include the supplementary trials presented in this dossier in support of the formulated product that were not already presented and evaluated during the Annex I inclusion of amidosulfuron.

Supplementary residue trials
A total of 4 supplementary residue trials were conducted on flax with Amidosulfuron WG 75 formulation in Northern and Southern Europe. The trials were conducted during the 2003, 2010 and 2014 growing seasons. The purpose of the studies was to determine the residue levels of amidosulfuron after a spray application of 30 g a.s./ha at growth stage 49. These trials cover the use supported in this dossier.

Table 6.3.2- 3: Number of residue trials conducted on flax with Amidosulfuron WG 75 per geographical region and vegetation period

Crop	Region	Formulation	Number of Trials				Report-No.	Dossier Ref.
			Vegetation period			Total		
			2003	2010	2014			
Flax	N-EU	WG	2			2	RA-2691/03 10-2242 10-2095 14-2010	KCA 6.3.3/03-06
	S-EU	WG		2	2	4		

N-EU: Northern Europe S-EU: Southern Europe

Report: KCA 6.3.3/02 [redacted]; 2004; M-232229-01-1
Title: Determination of residues of amidosulfuron in / on flax following spray application of AE F075032 00 WG75 A1 (75WG) in the field in Great Britain and Germany
Report No.: C042237
Document No.: M-232229-01-1
Guideline(s): EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, section A, point 8. Residues in or on Treated Products, Food and Feed.
Guideline deviation(s): none
GLP/GEP: **yes**

Material and methods
Two field trials were conducted in/on flax during the 2003 growing season in northern Europe. The trials were located in Great Britain (1) and in Germany (1).

The formulation WG 75, a wettable granule formulation containing 750 g/kg of amidosulfuron, was applied once at growth stage BBCH 32-38 with an application rate of 30 g/ha amidosulfuron.

For data gathering purposes the samples taken from these trials were analysed for parent amidosulfuron using method 00815/M001(Report M-226888-01-1 in Baseline dossier) with a limit of quantification of 0.01 mg/kg for seed and 0.05 mg/kg for shoot and straw. Please refer to MCA section 4.

Findings:
- Mean concurrent recoveries were within the acceptable range of 70-110 %, RSD <20% as shown in Table below. They validate the study results.

Table 6.3.3- 4: Concurrent recoveries in/on flax for amidosulfuron

Sample Material	FL [mg/kg]	Single Values [%]	Mean Value [%]	RSD [%]	LOQ [mg/kg]
Seed	0.01	85 82	84	-	0.01
	0.10	86 79	83	-	
	Overall Recovery (n = 4)		**83**	**3.8**	
Whole plant without root	0.05	95 96	96	-	0.05
	0.50	98 96	97	-	
	Overall Recovery (n = 4)		**96**	**1.3**	

FL = Fortification Level, RSD = Relative Standard Deviation, LOQ = Practical Limit of Quantification.

- Storage period of samples:
The longest period of time for which samples from these trials were stored prior to analysis is given in **Table 6.3.3- 5**. For the whole plant without root, the maximum storage period of samples is covered by the storage stability data.

Table 6.3.3- 5: Maximum storage period of samples from trials from RA-2691/03

Compound	Crop	Sample material	Maximum storage period (days)	Duration Covered (days)
Amidosulfuron	Flax	Whole plant without root	277	720
		Seed	209	720

- Residue results:
No residues above the LOQ of 0.05 mg/kg (shoot) or 0.01 mg/kg (seed) could be detected in any of the control samples.

At harvest, residues of parent amidosulfuron in all flax commodities were always less than the limit of quantification (0.01 mg/kg for grain and 0.05 mg/kg for shoot).

Residue levels in treated samples are summarised in **Table 6.3.3- 6**.

Table 6.3.3- 6: Residues of amidosulfuron from residue trials conducted on flax

Study Trial No. Trial SubID GLP Year	Crop Variety	Country	Application					Residues		
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	Amidosulfuron (mg/kg)
RA-2691/03 R 2003 0504 4 0504-03 GLP yes 2003	Flax/Linseed Juliette	United Kingdom GB-IP23 8HH, Thornham, Suffolk Europe, North	75 WG	1	0.030	0.00975	38	whole plant without roots	0	0.36
								seed	88	<0.01
RA-2691/03 R 2003 0604 0 0604-03 GLP yes 2003	Flax/Linseed Taurus	Germany D-86368 Gersthofen Europe, North	75 WG	1	0.030	0.00975	32	whole plant without roots	0	0.40
								seed	72	<0.01

Conclusion
Two supervised field trials on flax were conducted in northern Europe during the 2003 growing season, under similar application conditions than the intended use pattern with the product Amidosulfuron WG 75 product.

The formulation was applied at growth stage BBCH 32-38 with an application rate of 30 g/ha amidosulfuron.

All trials were conducted according to GLP.

As shown in the table, at harvest, the residues of amidosulfuron in linseed were below the LOQ of 0.01 mg/kg in linseed.

Report: KCA 6.3.3/03 [redacted]; [redacted]; 2011; M-417527-01-1
Title: Determination of the residues of amidosulfuron, iodosulfuron-methyl-sodium and mefenpyr-diethyl in/on flax/linseed after spraying of AMS & IMS & MPR WG 26.25 in the field in Spain
Report No.: 10-2242
Document No.: M-417527-01-1
Guideline(s): EU-Ref: Council Directive 91/414/EEC of July 15, 1991,
Annex II, part A, section 6 and Annex III, part A, section 8
Residues in or on Treated Products, Food and Feed
EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s): none
GLP/GEP: **yes**

Material and methods:
One field trial was conducted in/on flax during the 2010 growing season in Southern Europe. The trial was located in Spain.

AMS & IMS & MPR WG 26.25 is a water dispersible granule, containing 1.25% iodosulfuron-methyl-sodium, 12.5% amidosulfuron and 12.5% mefenpyr-diethyl. The product was used with an application rate of 0.25 kg/ha and 317 L water per ha, corresponding to 0.032 kg/ha amidosulfuron. The application was carried out at growth stage BBCH 49.

The samples taken from these trials were analysed for parent amidosulfuron and its metabolite AE F101630 using method BE-002-P09-02 (01325) with a limit of quantification of 0.01 mg/kg for green material and seed. Please refer to the Section 4 CA 4.1.2.

Findings:
- Mean concurrent recoveries were within the acceptable range of 70-110 %, RSD <20% as shown in the Tables below. They validate the study results.

Table 6.3.3- 7: Recovery data for amidosulfuron

Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
flax/linseed / green material	0.01	97; 98; 99; 101; 102	99	2.1	0.01
	0.10	91; 92; 92; 99; 99	95	4.3	
	1.0	78	78	-	
		Overall recovery (n = 11)	**95**	**7.2**	
flax/linseed / seed	0.01	100; 101; 101; 102; 102	101	0.8	0.01
	0.10	86; 89; 91; 93; 103	92	7.0	
	1.0	79	79	-	
		Overall recovery (n = 11)	**95**	**8.5**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with amidosulfuron, determined as amidosulfuron and calculated as amidosulfuron
These recoveries were performed during the conduct of the study/studies 10-2095 and 10-2242.

Table 6.3.3- 8: Recovery data for AE F101630

Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
flax/linseed / green material	0.01	97; 107; 107; 110; 111	106	5.2	0.01
	0.10	92; 104; 108; 114; 114	106	8.6	
	1.0	80	80	-	
		Overall recovery (n = 11)	**104**	**10.0**	
flax/linseed / seed	0.01	95; 103; 104; 109; 111	104	6.0	0.01
	0.10	93; 100; 101; 103; 112	102	6.7	
		Overall recovery (n = 10)	**103**	**6.1**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with AE F101630, determined as AE F101630 and calculated as AE F101630
These recoveries were performed during the conduct of the study/studies 10-2095 and 10-2242.

- Storage period of samples:
The maximum storage periods of deep-frozen samples for amidosulfuron and its metabolite AE F101630 ranged between 266 and 324 days. All the maximum storage periods of samples are covered by the storage stability data.

Table 6.3.3- 9: Maximum storage period of samples from trials from 10-2242

Crop	Compound	Sample material	Maximum storage period (days)	Duration Covered (days)
Flax	Amidosulfuron and metabolite AE F101630	Green material	324	714
		Seed	266	720

- Residue results:
No residues of amidosulfuron and AE F101630 above the LOQ of 0.01 mg/kg (green material and seed) could be detected in any of the control samples.

At harvest, residues of parent amidosulfuron and its metabolite AE F101630 in flax/linseed were less than the limit of quantification (0.01 mg/kg for grain).

Residue levels in treated samples are summarised in Table 6.3.3- 10.

Table 6.3.3- 10: Analytical results of treated samples for amidosulfuron and its metabolite AE F101630 in/on flax/linseed

Study Trial No. Trial SubID GLP Year	Crop Variety	Country	Application					Residues			
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	Amido-sulfuron (mg/kg)	AE F101630 [mg/kg]
10-2242 10-2242-01 GLP yes 2010	Flax/ Linseed Border	Spain Maro, Neria 29001 Malaga Europe, South	26.25 WG	1	0.032	0.01	49	Green material	0	0.73	0.06
								seed	58	<0.01	< 0.01

DALT = Days after last treatment

Conclusion
One supervised field trial on flax was conducted in Southern Europe during the 2010 growing season, at the slightly higher dose rate (within ±25%) compared to the use pattern with the product Amidosulfuron WG 75. The formulation WG 26.25 was applied with an application rate of 32 g/ha amidosulfuron at growth stage 49.

This trial was conducted according to GLP.

As shown in the table, at harvest, the residues of amidosulfuron in linseed were below the LOQ of 0.01 mg/kg in linseed.

Report: KCA 6.3.3/04 [redacted]; [redacted]; 2012; M-424441-01-1
Title: Determination of the residues of amidosulfuron, iodosulfuron-methyl-sodium and mefenpyr-diethyl in/on flax/linseed after spraying of AMS & IMS & MPR WG 26.25 in the field in southern France
Report No.: 10-2095
Document No.: M-424441-01-1
Guideline(s): EU-Ref: Council Directive 91/414/EEC of July 15, 1991, Annex II, part A, section 6 and Annex III, part A, section 8
Residues in or on Treated Products, Food and Feed EC guidance working document 7029/VI/95 rev. 5 (1997-07-22)
Guideline deviation(s): none
GLP/GEP: **yes**

Material and methods:
One field trial was conducted in/on flax during the 2010 growing season in Southern Europe. The trial was located in France.

The formulation AMS & IMS & MPR WG 26.25 is a water dispersible granule, containing 1.25% iodosulfuron-methyl-sodium, 12.5% amidosulfuron and 12.5% mefenpyr-diethyl. The product was

used with an application rate of 0.24 kg/ha and 300 L water per ha, corresponding to 0.030 kg/ha amidosulfuron. The application was carried out at growth stage BBCH 36.

The samples taken from these trials were analysed for parent amidosulfuron and its metabolite AE F101630 using method BE-002-P09-02 (01325) with a limit of quantification of 0.01 mg/kg for green material and seed. Please refer to Section 4 CA 4.1.2.

Findings:
- Mean concurrent recoveries were within the acceptable range of 70-110 %, RSD ≤ 20% as shown in **Table 6.3.3- 11** and **Table 6.3.3- 12**. They validate the study results.

Table 6.3.3- 11: Recovery data for amidosulfuron

Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
flax/linseed / green material	0.01	97; 98; 99; 101; 102	99	2.1	0.01
	0.10	91; 92; 92; 99; 99	95	4.3	
	1.0	78	-	-	
		Overall recovery (n = 11)	**95**	**7.2**	
flax/linseed / seed	0.01	100; 101; 101; 102; 102	101	0.8	0.01
	0.10	86; 89; 91; 93; 103	92	7.0	
	1.0	79	-	-	
		Overall recovery (n = 11)	**95**	**8.5**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with amidosulfuron, determined as amidosulfuron and calculated as amidosulfuron
These recoveries were performed during the conduct of the study/studies 10-2095 and 10-2242.

Table 6.3.3- 12: Recovery data for AE F101630

Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
flax/linseed green material	0.01	97; 107; 107; 110; 111	106	5.2	0.01
	0.10	92; 104; 108; 114; 114	106	8.6	
	1.0	80	-	-	
		Overall recovery (n = 11)	**104**	**10.0**	
flax/linseed seed	0.01	95; 103; 104; 109; 111	104	6.0	0.01
	0.10	93; 100; 101; 103; 112	102	6.7	
		Overall recovery (n = 10)	**103**	**6.1**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with AE F101630, determined as AE F101630 and calculated as AE F101630
These recoveries were performed during the conduct of the study/studies 10-2095 and 10-2242.

- Storage period of samples:
The maximum storage periods of deep-frozen samples for amidosulfuron and its metabolite AE F101630 ranged between 328 and 422 days. All the maximum storage periods of samples are covered by the storage stability studies (cf. MCA Point 6.1.).

Table 6.3.3- 13: Maximum storage period of samples from trials from 10-2095

Crop	Compound	Sample material	Maximum storage period (days)	Duration Covered (days)
Flax	Amidosulfuron and metabolite AE F101630	Green material	422	714
		Seed	328	720

- Residue results:
No residues of amidosulfuron and AE F101630 above the LOQ of 0.01 mg/kg (green material and seed) could be detected in any of the control samples.

At harvest, residues of parent amidosulfuron and its metabolite AE F101630 in flax/linseed were less than the limit of quantification (0.01 mg/kg for grain).

Residue levels in treated samples are summarised in Table 6.3.3- 14.

Table 6.3.3- 14: Analytical results of treated samples for amidosulfuron and its metabolite AE F101630 in/on flax/linseed

Study Trial No. Trial SubID GLP Year	Crop Variety	Country	Application					Residues			
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	GS	Portion analysed	DALT (days)	Amido-sulfuron (mg/kg)	AE F101630 [mg/kg]
10-2095 10-2095-01 GLP yes 2010	Flax/ Linseed Banquise	France F-81800 Rabastens Europe, South	26.25 WG	1	0.030	0.01	36	Green material	0	0.71	0.01
								seed	94	<0.01	< 0.01

DALT = Days after last treatment

Conclusion
One supervised field trial on flax was conducted in Southern Europe during the 2010 growing season, at the corresponding use pattern with the product Amidosulfuron WG 75. The formulation WG 26.25 was applied with an application rate of 30 g/ha amidosulfuron at growth stage 36.

This trial was conducted according to GLP.

As shown in the table at harvest, the residues of amidosulfuron in linseed were below the LOQ of 0.01 mg/kg in linseed.

Table 6.3.3- 15: Residues on flax/linseed after application of AMS & IMS& MPR WG 26.25

Analyte	Crop	No of trials	Sample material	DALT	Residue [mg/kg]
Amidosulfuron	Flax/ Linseed	1	Green material	0	0.71
			Seed	94	< 0.01
AE F101630			Green material	0	0.01
			Seed	94	< 0.01

DALT = Days after last treatment

Supplementary residues trials conducted in 2014 in Southern zone

Report:	KCA 6.3.3/05[redacted]X;[redacted]; 2015; M-534553-01-1
Title:	Determination of the residues of amidosulfuron in/on flax/linseed after spray application of amidosulfuron WG 75 in Spain and Italy
Report No.:	14-2010
Document No.:	M-534553-01-1
Guideline(s):	- REGULATION (EC) No 1107/2009 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 21 October 2009 concerning the placing of plant protection products on the market - OECD Guideline for the Testing of Chemicals on Crop Field Trials (TG 509 published in September 2009) - US EPA OCSPP Guideline No. 860.1500 on Crop Field Trial
Guideline deviation(s):	none
GLP/GEP:	**yes**

Material and methods

Two residue trials were done with the formulation Amidosulfuron WG 75, a WG (Water dispersible granules) formulation containing 75 % amidosulfuron during the 2014 growing seasons. The residue trials were carried out in the field in Spain and Italy for Southern Europe.

The formulation was applied once at growth stage 49 with an application rate of 0.04 kg/ha and 364-393 L water per ha, corresponding to a spray concentration of 0.011 % and 0.03 kg/ha of amidosulfuron.

Green material and seeds of flax/linseed were taken at days 0 and 54-60 after the last application (DALT). Residues of amidosulfuron and its metabolite AE F101630 in these matrices were determined according to method BE-002-P09-02 (BCS internal no. 01325).

Findings

- Method performance: The average recoveries were performed at fortification levels between 0.01 and 1 mg/kg per analyte for amidosulfuron and its metabolite AE F101630 and were within the acceptable range of 70 – 110% and the RSD values were below 20% as shown in **Table 6.3.3- 16** and **Table 6.3.3- 17**.

Table 6.3.3- 16: Recovery data for amidosulfuron

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
flax/linseed / green material	0.01	96; 97; 98	97	1.0	0.01
	0.10	96; 98; 100; 103	99	3.0	
	1.0	97	-	-	
	Overall recovery (n = 8)		**98**	**2.4**	
flax/linseed / seed	0.01	82; 84; 85	84	1.8	0.01
	0.10	80; 81; 84	82	2.5	
	Overall recovery (n = 6)		**83**	**2.4**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with amidosulfuron, determined as amidosulfuron and calculated as amidosulfuron
All recoveries were performed during the conduct of the study 14-2010.

Table 6.3.3- 17: Recovery data for AE F101630

Crop / Sample material	FL [mg/kg]	Single values [%]	Mean value [%]	RSD [%]	LOQ [mg/kg]
flax/linseed / green material	0.01	93; 101; 104	99	5.7	0.01
	0.10	102; 103; 107; 117	107	6.4	
	1.0	98	-	-	
		Overall recovery (n = 8)	**103**	**6.8**	
flax/linseed / seed	0.01	91; 96; 100	96	4.7	0.01
	0.10	93; 98; 98	96	3.0	
		Overall recovery (n = 6)	**96**	**3.5**	

FL = Fortification level, RSD = Relative standard deviation, LOQ = Practical limit of quantification
Fortified with AE F101630, determined as AE F101630 and calculated as AE F101630
All recoveries were performed during the conduct of the study 14-2010.

- Storage stability: The maximum storage periods of deep-frozen samples for amidosulfuron and its metabolite AE F101630 ranged between 263 and 354 days and are covered by the storage stability studies (cf. MCA Point 6.1.).

Table 6.3.3- 18: amidosulfuron and its metabolite AE F101630 in/on flax/linseed – maximum storage periods of samples

Study No.	Trial SubID	Targets crop	Sample Material	Substance	Storage Period [Max. days]	Dates of Sampling to Analysis
Southern Europe						
14-2010	14-2010-01	flax/linseed	green material	amidosulfuron and AE F101630	354	2014-06-26 to 2015-06-15
			seed		289	2014-08-25 to 2015-06-10
	14-2010-02	flax/linseed	green material	amidosulfuron and AE F101630	322	2014-07-28 to 2015-06-15
			seed		263	2014-09-20 to 2015-06-10

Use patterns for the trials conducted on flax/linseed are listed in **Table 6.3.3- 19**. Residues of the parent compound amidosulfuron and its metabolite AE F101630 are summarised in **Table 6.3.3- 20**.

- Residue results:
The apparent residues in the control samples were below 30% of the LOQ. Recoveries were not corrected for apparent residues in the control samples used for these recoveries.

Table 6.3.3- 19: Actual use pattern of residue trials conducted in/on flax/linseed with the formulation amidosulfuron WG 75

Study, Trial No., Trial SubID, GLP, Year	Crop Variety	Country	Application						
			FL	No	kg/ha (a.s.)	kg/hL (a.s.)	Water rate (L/ha)	Spray interval (days)	GS
Southern Europe									
14-2010 14-2010-01 GLP: yes 2014	flax/linseed Dorado	Spain	WG 75	1	0.03	0.0083*	364	-	49
14-2010 14-2010-02 GLP: yes 2014	flax/linseed Antares	Italy	WG 75	1	0.03	0.0076*	393	-	49

FL: Formulation No: number of applications a.s.: active substance
GS: growth stage (BBCH code) at application
* For the calculation of concentration of amidosulfuron a.s. in the spray liquid (kg/hL), rounded values were used. Therefore, minor deviations may occur between the value shown above and the one presented in the Field Phase Report (Appendix 10), which was calculated using unrounded values.

Table 6.3.3- 20: Residues of amidosulfuron and its metabolite AE F101630 in/on flax/linseed, treated with the formulation amidosulfuron WG 75

Trial No. Country, GLP, Year	Sample material	Growth stage (BBCH) at sampling	DALT (days)	Residues [mg/kg]	
				amidosulfuron	AE F101630
Southern Europe					
14-2010 14-2010-01 GLP: yes 2014	green material	49	0*	0.55*	0.087*
	seed	89	60	< 0.01	< 0.01
14-2010 14-2010-02 GLP: yes 2014	green material	49	0*	0.51*	0.94*
	seed	89	54	< 0.01	< 0.01

DALT = Days after last treatment * Days before last treatment
* Mean values. Treated samples of green material were analysed twice to confirm the first results.

Analyte:	Final determination as:	Residues calculated as:
amidosulfuron	amidosulfuron	amidosulfuron
AE F101630	AE F101630	AE F101630

Conclusion
To support the use of Amidosulfuron WG 75 formulation in/on flax/ linseed, 2 residue trials were conducted in Southern Europe during the 2014 growing season, at the corresponding use pattern. The formulation WG 75 was applied with an application rate of 30 g/ha amidosulfuron at growth stage 49. As shown in the table, at harvest, the residues of amidosulfuron in linseed were below the LOQ of 0.01 mg/kg.

The residue levels determined in the treated samples from the 2 trial sites of the study are summarised in the table below:

Table 6.3.3- 21: Residues on flax/linseed after application of Amidosulfuron WG 75

Application	Analyte	Crop	No of trials	Sample material	DALT	Residue [mg/kg] min - max
30 g amidosulfuron/Ha at GS 49	amidosulfuron	flax/linseed	2	green material	0	0.51 - 0.55
				seed	54 - 60	< 0.01
	AE F101630		2	green material	0	0.087 - 0.94
				seed	54 - 60	< 0.01

DALT = Days after last treatment

Analyte:	Final determination as:	Residues calculated as:
amidosulfuron	amidosulfuron	amidosulfuron
AE F101630	AE F101630	AE F101630

Summary of all supplementary residue trials conducted on flax in Southern and Northern Europe

As shown in the table, at harvest, the residues of amidosulfuron in linseed were below the LOQ of 0.01 mg/kg in linseed. The current MRL of 0.01 mg/kg in seed is supported by all supplementary trials.

Table 6.3.3- 22: Summary of amidosulfuron residue levels on flax/linseed (supplementary trials)

Application	Analyte	Crop	No of trials	Sample material	DALT	Residue [mg/kg] min - max
30 g amidosulfuron/Ha at GS 32-49	amidosulfuron	flax/linseed	6	green material (whole plant without roots)	0	0.36 - 0.73
				seed	54 - 94	< 0.01
	AE F101630	flax/linseed	4	green material (whole plant without roots)	0	0.01 - 0.94
				seed	54 - 94	< 0.01

DALT = Days after last treatment

CA 6.4 Feeding studies

Original Annex II dossier

No livestock feeding study was submitted during the original Annex II dossier.

"AIR process" - new studies submitted

A ruminant feeding study was conducted in 2007 where milk and edible tissues were analysed for amidosulfuron and metabolite AE F101630. It was submitted to the former RMS (Austria) in 2008 and in December 2010 (refer to Addendum to monograph prepared in the context of post Annex I procedure (new Annex II data) - rev.1 February 2011).

Dietary burden

Amidosulfuron is authorised on cereals, grass and flax which might be fed to livestock. The median and maximum dietary burden for livestock was determined using the same approach as in the EFSA reasoned opinion on the review of the existing MRLs for amidosulfuron (EFSA Journal 2014; 12(3):3614). The OECD model from 2015 is used for this calculation.

Table 6.4-1: Input values for the dietary burden calculation

Commodity	Median dietary burden		Maximum dietary burden	
	Input value (mg/kg)	Comment	Input value (mg/kg)	Comment
Risk assessment residue definition: amidosulfuron				
Linseed meal	0.01	Median residue*CF	0.01	Median residue*CF
Wheat, barley, rye, oats grain	0.01	Median residue*CF	0.01	Median residue*CF
Barley bran	0.01	Median residue*CF	0.01	Median residue*CF
Wheat milled by-products	0.01	Median residue*CF	0.01	Median residue*CF
Risk assessment residue definition: sum of amidosulfuron and desmethyl amidosulfuron, expressed as amidosulfuron				
Wheat, rye straw*	0.1	Median residue	0.1	Highest residue
Risk assessment residue definition: sum of amidosulfuron, desmethyl amidosulfuron and amidosulfuron guanidine, expressed as amidosulfuron				
Grass, fresh	0.235	Median residue (PHI 7 d)	3.35	Highest residue (PHI 7 d)
Grass, hay	0.20	Median residue (PHI 21 d)	1.80	Highest residue (PHI 21 d)

* Worst case LOQ considered =0.05 mg/kg in cereal straw despite 2014 residue trials analysed with LOQ=0.01 mg/kg

The results of the dietary burden calculations are summarised in Table 6.4-2 for a PHI of 7 days on fresh grass.

Table 6.4-2: Results of the dietary burden calculation

Animals	**Median burden** (mg/kg bw)	**Maximum burden** (mg/kg bw)	**Above 0.004 mg/kg bw**	**Maximum burden** (mg/kg DM)	**Highest contributing commodities**
Beef cattle	0,0114	0,161	Yes	6.71	Grass forage (fresh)
Dairy cattle	0,0219	0,309	Yes	8.04	Grass forage (fresh)
Ram/Ewe	0,0298	0,424	Yes	12.73	Grass forage (fresh)
Lamb	0,0202	0,285	Yes	6.71	Grass forage (fresh)
Pig (breeding)	0,005	0,062	Yes	2.69	Grass forage (fresh)
Pig (finishing)	0,000	0,000	No	0,01	Barley grain
Poultry broiler	0,001	0,001	No	0,01	Barley grain
Poultry layer	0,001	0,001	No	0,02	Wheat straw
Turkey	0,001	0,001	No	0,01	Rye grain

These results indicate that fresh grass may represent the main contributing commodity for ruminants and for pigs. The trigger value was not exceeded in the case of poultry.

CA 6.4.1 Poultry

No study was performed. Poultry is not expected to be exposed to significant levels of amidosulfuron residues. Results of the dietary burden confirm that a feeding study is not required for poultry.

CA 6.4.2 Ruminants

"AIR" process – New study submitted:

The following study was submitted to the former RMS (Austria) in 2008 and in December 2010 (post Annex I review of amidosulfuron containing formulations). The conclusions of the former RMS are compiled in the DAR Addendum Volume 3, Annex B (Addendum to monograph prepared in the context of post Annex I procedure (new annex II data) – rev. 1 February 2011).

In the EFSA Scientific Report (2007) 116 of 14 November 2007, it was stated: "In order to obtain appropriate information for MRL setting a feeding study in lactating cows should be conducted, preferably with simultaneous administration of amidosulfuron and metabolite AE F101630 at a 1 to 1 ratio, reflecting the exposure resulting from consumption of grass or hay under worst case conditions." After this, it was agreed to perform a livestock feeding study with parent amidosulfuron alone; please refer to document [redacted]; 2007; Rationale for the design of a livestock feeding study with amidosufuron - Code: AE F075032; M-297410-01-1.

Report:	KCA 6.4.2/01[redacted], 2007, M-297410-01-1
Title:	Rationale for the design of a livestock feeding study with amidosufuron - Code: AE F075032
Report No.:	M-297410-01-1
Document No.:	M-297410-01-1
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

This is because the toxicity profile of metabolite AE F101630 is covered by the toxicological data package of parent amidosulfuron and the dietary burden was calculated taking into account the highest residue (HR) found in grass at PHI 7 days, for both amidosulfuron and '630 (sum expressed in amidosulfuron = 1.84 mg). These conditions represent the worst-case scenario.

On top of this, both amidosulfuron and AE F101630 were analysed in the cow tissues and milk.

Report:	KCA 6.4.2/02[redacted]E; 2008; M-298770-01-1
Title:	Amidosulfuron - Magnitude of the residue in lactating cows
Report No.:	RABER002
Document No.:	M-298770-01-1
Guideline(s):	OPPTS 860.1480 - Meat/Milk/Poultry/Eggs DACO 7.5.1 – Livestock Feeding Study OECD Test guidelines Residue in Livestock 505, adopted: 8 January 2007 OECD Guidance Doc Overview Residue Chemistry Studies. ENV/JM/MONO(2006)32, adopted 10 October 2006
Guideline deviation(s):	1. Analysis of the potable water for the test system was not conducted under GLPs. 2. Dry matter analysis of the feed components was not conducted under GLPs. 3. Some of the test substance properties described in table 2 were not performed under GLPs. 4. Standard storage conditions were added to the raw data as late entries.
GLP/GEP:	**yes**

The purpose of this study was to determine the magnitude of the residue of amidosulfuron and its metabolite desmethyl amidosulfuron (AE F101630) residue that may be expected in meat and milk from animals that have been fed feedstuffs containing residue of amidosulfuron. These residue values

will be used to establish a registered tolerance or maximum residue level (MRL) for amidosulfuron and support OECD registration for the use of amidosulfuron on crops that may be fed to livestock.

Material and methods

The magnitude of the residue of amidosulfuron has been studied in lactating dairy cows. Ten lactating Holstein dairy cows (three cows/treatment group and one control cow) were dosed orally, *via* capsule, for 29 consecutive days with amidosulfuron at different target dose rates. These levels were based on field residue data and were approximately 1X, 3X and 10X the anticipated maximum dietary burden. These were based on total amidosulfuron residues found on grasses at the 7-day re-entry period for livestock into pastures. The highest residues found in cattle feed items obtained from field trials with a 7-day PHI for total amidosulfuron residues are presented in the table below. Using these values in conjunction with the percent of livestock diet values, animal body weights and daily feed intakes found in Annex 4 of the OECD Guidance Document on Overview of Residue Chemistry Studies for beef cattle and dairy cattle in US/Canada and Europe, a maximum dietary burden and dose level in mg/kg bw was calculated as follows:

$$\text{Dietary Burden} = \frac{\text{Residue Level}}{\text{\% Dry Matter}} \times \text{\% of Diet}$$

$$\text{Dose (mg/kg bw)} = \frac{\text{Dietary Burden} \times \text{Daily Intake}}{\text{Body Wt}}$$

The maximum anticipated dietary burden (ppm: mg pesticide per kg of feed) and dose (mg/kg bw) calculated below were obtained using the percent of livestock diet values, body weight (650 kg), and daily intake (25 kg; dry weight) for dairy cattle in Europe.

Feedstuff	% of Diet	% Dry Matter	Residue Level (ppm)	Corrected Residue Level (ppm)[1]	Dietary Burden (ppm)	Dose in mg/kg body weight
Grass Forage	60	25	1.64	6.560	3.936	0.1514
Wheat Grain	25	89	0.01	0.011	0.003	0.0001
Flaxseed Meal	15	88	0.05	0.057	0.009	0.0003
Total	100				3.95	**0.152**

[1] Corrected residue level = residue level ÷ % dry matter

As determined in the above calculations, the maximum dietary burden of amidosulfuron for dairy cattle was calculated to be 0.152 mg/kg bw. Therefore, the cattle were dosed at the following levels:

Dose Group	Target Dose (mg/kg bw)	No. of Animals
1X Dose	0.152	3
3X Dose	0.456	3
10X Dose	1.52	3
Control	Placebo	1

Milk was collected twice daily during the dosing period. Milk samples from all dose groups were analysed for total amidosulfuron residue on study days 0, 1, 3, 7, 10, 14, 17, 21, 24, 26, and 28. Additionally, a portion of the 28 day milk sample from the control and 10X dose group was separated into milk fat (cream) and whey (skimmed milk), and each was analysed. On day 29, the animals were sacrificed and liver, kidney, composite muscle, and composite fat were collected for analysis.

Residues of amidosulfuron and its metabolite desmethyl amidosulfuron (AE F101630) in tissue and milk samples were quantitated by high performance liquid chromatography/electrospray ionization/tandem mass spectrometry (LC/MS/MS) using method BE-001-A07-01 (please refer to M-298517-01-1 and M-298519-01-1).

Method validation was performed prior to sample analysis and concurrent recoveries were performed during sample analysis to demonstrate acceptable method performance. The limit of quantification (LOQ) was 0.005 ppm for each analyte in milk matrices and was 0.010 ppm for each analyte in the tissue matrices.

Findings

Milk

Maximum amidosulfuron residues in the milk samples from the 10X, 3X, and 1X feeding levels were 0.138 ppm, 0.021 ppm, and 0.007 ppm, respectively. Amidosulfuron residue reached a plateau by the tenth day of dosing for all feeding levels and declined slightly through study day 28. Desmethyl amidosulfuron (AE F101630) residues were not found in any milk samples above the LOQ. The concentration factors for amidosulfuron residue in milk were determined to be 1.09 and 1.06 for the whey and milk fat, respectively.

The results are summarised in the table below.

Table 6.4.2- 1: Residue Levels (ppm) of amidosulfuron and AE F101630 in milk and milk products

Analyte	Matrix	Feeding Level (ppm)	Residue Levels (ppm)					
			n	Min	Max	Median	Mean	Std. Dev.
Amidosulfuron	Milk	(10X)	30	0.038	0.138	0.050	0.066	0.031
		(3X)	30	<0.005	0.021	0.014	0.014	0.004
		(1X)	30	<0.005	0.007	0.005	0.005	0.000
	Milk Fat	(10X)	3	0.038	0.080	0.049	0.056	0.022
	Whey	(10X)	3	0.041	0.084	0.049	0.058	0.023
AE F101630	Milk	(10X)	30	<0.005	<0.005	<0.005	<0.005	-
		(3X)	30	<0.005	<0.005	<0.005	<0.005	-
		(1X)	30	<0.005	<0.005	<0.005	<0.005	-
	Milk Fat	(10X)	3	<0.005	<0.005	<0.005	<0.005	-
	Whey	(10X)	3	<0.005	<0.005	<0.005	<0.005	-

Tissues

The highest amidosulfuron residues were found in the excretory organs of the kidney and liver. In kidney tissue, the maximum residues found were 0.180 ppm in the 10X dose group, 0.094 ppm in the 3X dose group, and 0.019 ppm in the 1X dose group.

In liver tissue, the maximum amidosulfuron residue found was 0.020 ppm in the 10X dose group, 0.012 ppm in the 3X dose group, and < 0.010 ppm in the 1X dose group.

Desmethyl amidosulfuron (AE F101630) residues were not found in any of the kidney or liver samples.

In muscle tissue, amidosulfuron residue was <0.010 ppm in all animals in the 10X dose group. Muscle samples from the 1X and 3X dose groups were not analysed.

In fat tissue, two of three animals in the 10X dose group had amidosulfuron residue below the LOQ. The maximum residue found was 0.015 ppm. No animal in the 3X dose group had residue above the LOQ in fat tissue. Fat samples from the 1X dose group were not analysed. Desmethyl amidosulfuron (AE F101630) residues were not found in any of the muscle or fat samples. Amidosulfuron residue in all tissues showed evidence of a dose response, although the coefficients of determination (R2) were not high.

The results are summarised in the table below.

Table 6.4.2- 2: Residue Levels (ppm) of amidosulfuron and AE F101630 in beef tissues

Analyte	Matrix	Feeding Level (ppm)	Residue Levels (ppm)					
			n	Min	Max	Median	Mean	Std. Dev.
Amidosulfuron	Fat	(3X)	3	<0.010	<0.010	0.002	0.003	0.001
		(10X)	3	<0.010	0.015	0.003	0.007	0.007
	Kidney	(1X)	3	0.015	0.019	0.019	0.018	0.002
		(3X)	3	0.041	0.094	0.050	0.062	0.028
		(10X)	3	0.116	0.180	0.156	0.151	0.032
	Muscle	(10X)	3	<0.010	<0.010	<0.010	<0.010	0.001
	Liver	(1X)	3	<0.010	<0.010	<0.010	<0.010	0.000
		(3X)	3	<0.010	0.012	<0.010	<0.010	0.004
		(10X)	3	0.013	0.020	0.016	0.016	0.004
AE F101630	Fat	(3X)	3	<0.010	<0.010	<0.010	<0.010	-
		(10X)	3	<0.010	<0.010	<0.010	<0.010	-
	Kidney	(1X)	3	<0.010	<0.010	<0.010	<0.010	-
		(3X)	3	<0.010	<0.010	<0.010	<0.010	-
		(10X)	3	<0.010	<0.010	<0.010	<0.010	-
	Muscle	(10X)	3	<0.010	<0.010	<0.010	<0.010	-
	Liver	(1X)	3	<0.010	<0.010	<0.010	<0.010	-
		(3X)	3	<0.010	<0.010	<0.010	<0.010	-
		(10X)	3	<0.010	<0.010	<0.010	<0.010	-

The analytical method was validated for the analysis of residues of amidosulfuron and its metabolite desmethyl amidosulfuron (AE F101630) in milk, milk fat (cream), whey (skimmed milk), fat, kidney, liver, and muscle prior to treated sample analyses. Recoveries of amidosulfuron and AE F101630 were also measured concurrently with each set of samples to verify method performance.

Concurrent recoveries were performed during the analysis of the study samples. In all matrices and at all fortifications levels, the recovery means ranged between 70-110% with relative standard deviations (RSD) lower than 20% thus validating the study results.

The results from concurrent recoveries are summarised in the table below.

Table 6.4.2- 3: Summary of Concurrent Recoveries of amidosulfuron and Desmethyl Amidosulfuron (AE F101630) from Milk, Milk Fat, Whey, Fat, Kidney, Muscle, and Liver

Matrix	Analyte	Spike Level (ppm)	Sample Size (n)	Recoveries (%)	Mean ± Std. Dev.
Milk	Amidosulfuron	0.005	13	87, 84, 99, 93, 96, 88, 91, 95, 90, 104, 86, 89, 89	91.6 ± 5.6
		0.100	11	96, 100, 105, 91, 89, 93, 87, 101, 86, 87, 93	93.5 ± 6.4
		0.200	3	88, 84, 87	86.3 ± 2.1
	AE F101630	0.005	13	83, 90, 101, 95, 95, 98, 97, 99, 103, 102, 87, 100, 92	95.5 ± 6.1
		0.100	11	90, 90, 105, 102, 94, 99, 89, 103, 87, 91, 94	94.9 ± 6.3
		0.200	3	93, 91, 95	93.0 ± 2.0
Milk Fat	Amidosulfuron	0.005	1	104	104
		0.100	1	109	109
	AE F101630	0.005	1	100	100
		0.100	1	101	101
Whey	Amidosulfuron	0.005	1	97	97
		0.100	1	94	94
	AE F101630	0.005	1	97	97
		0.100	1	94	94
Fat	Amidosulfuron	0.010	2	94, 84	89 ± 7.1
		0.100	2	87, 91	89 ± 2.8
	AE F101630	0.010	2	89, 91	90 ± 1.4
		0.100	2	87, 99	93 ± 8.5
Kidney	Amidosulfuron	0.010	2	96, 84	90 ± 8.5
		0.100	2	100, 90	95 ± 7.1
		0.200	3	87, 88, 84	86 ± 2.1
	AE F101630	0.010	2	98, 86	92 ± 8.5
		0.100	2	92, 88	90 ± 2.8
		0.200	3	88, 88, 87	88 ± 0.6
Muscle	Amidosulfuron	0.010	1	85	85
		0.100	1	86	86
	AE F101630	0.010	1	90	90
		0.100	1	94	94
Liver	Amidosulfuron	0.010	2	97, 85	91 ± 8.5
		0.100	2	107, 86	97 ± 14.8
	AE F101630	0.010	2	95, 89	92 ± 4.2
		0.100	2	94, 99	97 ± 3.5

The tissue and milk samples in this study were analysed within 19 days of collection; therefore, freezer storage stability studies on beef tissue and milk matrices are not required.

Conclusion
The cow feeding study showed, that, in the case of a treatment on pastures and with a re-entry period of 7 days, the residues of amidosulfuron in animal matrices are low and do not create any issues. Metabolite AE F101630 was not found in any tissues or milk.

CA 6.4.3 Pigs

Since the metabolic profile of amidosulfuron did not differ in ruminants and rat, a feeding study in pigs is not required.

CA 6.4.4 Fish

No study is needed as amidosulfuron is not fat soluble.

CA 6.5 Effects of processing

CA 6.5.1 Nature of the residue

"AIR" process – New study submitted:

A high temperature hydrolysis study was conducted on amidosulfuron and is presented below.

Report: KCA 6.5.1/01 [redacted]; 2014; M-505652-01-1
Title: Nature of the residues of [pyrimidyl-2-14C]amidosulfuron in processed commodities - High temperature hydrolysis
Report No.: EnSa-14-0425
Document No.: M-505652-01-1
Guideline(s): OECD Guideline for the Testing of Chemicals 507; Nature of the Pesticide Residues in Processed Commodities - High Temperature Hydrolysis, adopted 2007-10-16; Regulation (EC) No 1107/2009 amended by Commission Regulation (EU) No 283/2013 (Europe); US EPA OCSPP not applicable
Guideline deviation(s): not specified
GLP/GEP: **yes**

The hydrolytic degradation of amidosulfuron in buffered drinking water has been investigated in order to determine whether the nature of the residue found in processed agricultural commodities is likely to be different from that in raw agricultural commodities. The test substance was exposed to three sets of conditions each of them being representative for a typical food processing operation.

Material and Methods
Radiolabelled [pyrimidyl-2-^{14}C] amidosulfuron was dissolved at concentrations of approximately 1 mg/L in citrate buffers which were incubated at 90 °C for 20 min (pH 4.0), 100 °C for 60 min (pH 5.0) and 120 °C for 20 min (pH 6.0). After incubation the samples were analysed by liquid scintillation counting to establish recoveries of radioactivity and by HPLC for determination of concentrations of test substance and hydrolysis products. A set of control solutions was used to obtain zero-time values.

Findings
HPLC profiling of samples before and after incubation showed complete or almost complete degradation of Amidosulfuron. Trace amounts (0.7%) of the test compound were found under conditions representative for sterilisation (pH 6, 120°C, 20 min), whereas under all other tested conditions complete hydrolytic cleavage of Amidosulfuron was observed.

Two major hydrolysis products were observed under conditions representative for pasteurisation (pH 4, 90°C, 20 min): AE F092944 (79.6%) and BCS-AW41401 (19.6%).

Under all other tested conditions AE F092944 was by far the major or only degradation product (> 99%). Identification of the hydrolysis products was accomplished by HPLC co-chromatography with non-radiolabelled reference compounds and by LC-MS/MS spectroscopy. A trace degradation product (< 1%) was detected in the test solution after hydrolysis at pH 4 (90°C, 20 min), but was not further investigated.

Amounts of parent compound and hydrolysis products after processing are presented in the following table:

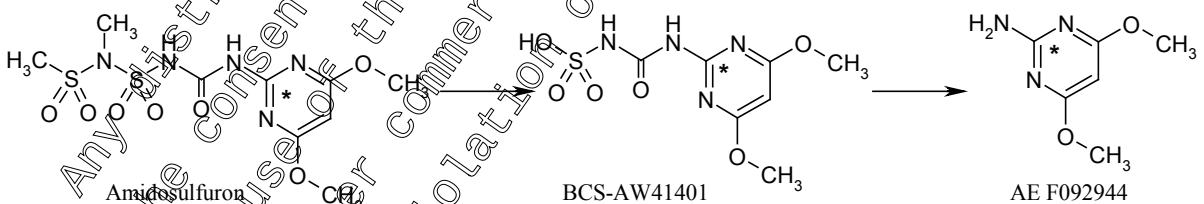
Table 6.5.1-1 Amounts of parent compound and hydrolysis products after processing

[pyrimidyl-2-14C] amidosulfuron	Processing conditions		
	pH4, 90 °C	pH5; 100 °C	pH6; 120 °C
	% Radioactivity	% Radioactivity	% Radioactivity
Parent	-	-	0.7
BCS-AW41401	19.6	0.5	-
AE F092944	79.6	99.5	99.3
Unknown	0.8	-	-

On the basis of these results, the hydrolysis pathway of [pyrimidyl-2-^{14}C] amidosulfuron was proposed. Cleavage of the parent compound at the central sulfonamide bond followed by hydrolysis of the carbamide bond was observed.

The proposed hydrolysis pathway of [pyrimidyl-2-^{14}C] amidosulfuron is presented below.

Figure 6.5.1-1: Hydrolytic degradation of [pyrimidyl-2-^{14}C] amidosulfuron under processing conditions



Conclusion
Under all tested conditions representative for food processing operations almost quantitative to complete hydrolysis of amidosulfuron was observed. The degradation reactions observed were cleavage of the parent compound at the central sulfonamide bond followed by hydrolysis of the carbamide bond. Under conditions representative for pasteurisation (pH 4, 90°C, 20 min) two major hydrolysis products (AE F092944 and BCS-AW41401) were observed, whereas under all other hydrolytic conditions AE F092944 was by far the major or only degradation product (> 99%).

Other degradation products were very minor (< 1%) under all tested conditions.

All material balances were in the range of 99.6 to 100.3% demonstrating that no volatile degradation products were formed.

CA 6.5.2 Distribution of the residue in inedible peel and pulp

Not relevant for the investigated representative uses (cereals, pasture, meadow and flax).

CA 6.5.3 Magnitude of residues in processed commodities

No studies were performed. Residues of amidosulfuron in grain from cereals and in linseeds are <0.01 mg/kg in the raw agricultural commodities. Therefore no significant residues (>0.1 mg/kg) can be expected in the plant products to be processed. Studies on the effect of processing on the nature of the residue are therefore not triggered.

CA 6.6 Residues in rotational crops

CA 6.6.1 Metabolism in rotational crops

Original Annex II dossier

The studies were presented in the Tier 2 summary document on the active substance, Section 4, Point 6.6. No supplementary metabolism studies in rotated crops are considered necessary for the AIR dossier.

In summary, confined crop rotation studies for amidosulfuron were performed using the 2-^{14}C-pyrimidyl-labelled active ingredient. The substance was applied to bare soil at a rate of up to 41 g a.s./ha, with wheat, carrots, white cabbage, potatoes and spinach being at three plant back intervals. The total radioactive residues in the edible part of all the plants that develop were extremely low (maximum of 0.003 mg/kg in wheat grain, 0.001 mg/kg in white cabbage, 0.0005 mg/kg in potato tuber, and < 0.001 mg/kg in carrot at the time of normal harvest for each crop). The residues in the non-edible parts of the plants were also low. The total residues in straw did not exceed 0.03 mg/kg (in wheat).

As a consequence, a specific residue definition for rotational crops was not deemed necessary due to the very low residue levels expected.

Furthermore, a reasoned opinion on the review of the existing maximum residue levels (MRLs) for amidosulfuron was published in EFSA Journal 014; 12(3):3614. EFSA concluded the following:

"Occurrence of amidosulfuron residues in rotational crops was also investigated and it was concluded that a specific residue definition for rotational crops is not deemed necessary and that residue levels in rotational commodities are not expected to exceed 0.01 mg/kg, provided that amidosulfuron is applied in compliance with the authorised European GAPs."

Specific plant back restrictions related to the use of amidosulfuron are therefore not required.

CA 6.6.2 Magnitude of residues in rotational crops

Studies submitted and evaluated for the first inclusion of amidosulfuron on Annex I:

Report:	KCA 6.6.1/01 [redacted]; 1991; M-130186-01-1
Title:	Hoe 075032-14C. Determination of the Nature and Level of Residues in Rotational Crops Sown 30 Days after Treatment of Soil
Report No.:	A46117
Document No.:	M-130186-01-1
Guideline(s):	BBA: IV, 3-10; USEPA (=EPA): § 165-1
Guideline deviation(s):	--
GLP/GEP:	**yes**

Report:	KCA 6.6.1/02 [redacted]; 1992; M-137893-01-1
Title:	Hoe 075032-14C, residue determination and metabolism in rotational crops sown 150 days after treatment of soil
Report No.:	A48798
Document No.:	M-137893-01-1
Guideline(s):	EPA Pesticide Assessment Guideline 165
Guideline deviation(s):	not specified
GLP/GEP:	**yes**

Report:	KCA 6.6.1/03 [redacted]; 1993; M-132745-01-1
Title:	Hoe 075032-14C, Residue Determination and Metabolism in Rotational Crops sown 365 Days after Treatment of Soil
Report No.:	A51844
Document No.:	M-132745-01-1
Guideline(s):	USEPA (=EPA): § 165-1 (1982)
Guideline deviation(s):	--
GLP/GEP:	**yes**

CA 6.7 Proposed residue definitions and maximum residue levels

CA 6.7.1 Proposed residue definitions

A reasoned opinion on the review of the existing maximum residue levels (MRLs) for amidosulfuron was published in EFSA Journal 2014, 12(3):3614.

Matrices	EFSA Journal 2014;12(3):3614
Cereal grain and linseed	Risk assessment: Amidosulfuron Monitoring: Amidosulfuron
Cereal straw	Risk assessment: Sum of amidosulfuron and desmethyl-amidosulfuron, expressed as amidosulfuron Monitoring: Amidosulfuron
Fresh grass	Risk assessment: Sum of amidosulfuron, desmethyl-amidosulfuron and amidosulfuron-guanidine, expressed as amidosulfuron Monitoring: Amidosulfuron
Food of animal origin	Risk assessment: Sum of amidosulfuron, desmethyl-amidosulfuron and amidosulfuron-guanidine, expressed as amidosulfuron Monitoring: Sum of amidosulfuron and desmethyl-amidosulfuron, expressed as amidosulfuron

CA 6.7.2 Proposed maximum residue levels (MRLs) and justification of the acceptability of the levels proposed

According to Article 12 of Regulation (EC) No 396/2005, the European Food Safety Authority (EFSA) has reviewed the Maximum Residue Levels (MRLs) currently established at European level for the pesticide active substance amidosulfuron. MRLs were published in the Official Journal of the European Union (Commission Regulation (EU) 2015/1200 of 22 July 2015).

CA 6.7.3 Proposed maximum residue levels (MRLs) and justification of the acceptability of the levels proposed for imported products (import tolerance)

"AIR3" process

According to the EFSA review, MRLs were proposed for linseed, cereal grain and animal commodities.

Table CA 6.7.3- 1: Current MRLs established by EFSA

Commodity	MRL (mg/kg)	Reference
Linseed	0.01*	EFSA Journal 2014; 12(3):3614 Commission Regulation (EU) 2015/1200 of 22 July 2015
Barley grain	0.01*	
Oats grain	0.01*	
Rye grain	0.01*	
Wheat grain	0.01*	
Swine kidney, edible offals	0.05	
Bovine, sheep, goat, and equine muscle, fat tissue	0.03	
Bovine, sheep, goat and equine liver	0.04	
Bovine, sheep, goat and equine kidney, edible offals	0.2	
Milk	0.07	

* indicates that the MRL is set at the limit of analytical quantification

Report: KCA 6.7.3/01 [redacted], 2016; M-555920-01-1
Title: Statement amidosulfuron on pastures
Report No.: M-555920-01-1
Document No.: M-555920-01-1
Guideline(s): --
Guideline deviation(s): --
GLP/GEP: **no**

The position paper (M-555920-01-1) presents an overview of all residue trials conducted on grass. Based on these exhaustive data, tentative MRLs proposed by EFSA on fresh grass in 2014 are discussed: MRL of 15 mg/kg in Northern Europe can be confirmed. For Southern Europe, the MRL proposed at 5 mg/kg could be reduced to 3 mg/kg.

Based on the livestock feeding study carried with amidosulfuron on dairy cows at dose levels representative to the exposure to the sum of amidosulfuron and its metabolites desmethyl amidosulfuron and amidosulfuron guanidine, it is possible to estimate residues of parent amidosulfuron and its metabolites in ruminant and pig tissues and in ruminant milk according to the EFSA model (animal model 2015a.xls). As no feeding study on sheeps or pigs was available, the

results of the cattle study were used to estimate by extrapolation the residue levels to sheep and pig tissues. MRLs for poultry products are not required because poultry is not expected to be exposed to significant levels of amidosulfuron residues.

New proposals for MRLs on animal commodities are also presented in the position paper.

Table CA 6.7.3- 2: MRL derived from the livestock feeding study based on the dietary burden calculation (intended PHI of 7 days for fresh grass)

Commodity	Dietary burden (mg/kg bw/d)		Feeding study			Calculated residue (mg/kg)		**MRL proposal (mg/kg), 2015 EFSA model**	EFSA MRL proposal, 2014 [(a)] (mg/kg)
	Median	Max	Dose level (mg/kg bw/d)	Mean residue (mg/kg)	Max residue (mg/kg)	Median	Max		
Beef muscle	0,011	0,161	0.152	ND	ND	< 0.02	< 0.02	-	0.03
			0.456	ND	ND				
			1.52	<0.02	<0.02				
Beef fat	0,011	0,161	0.152	ND	ND	< 0.02	< 0.02		0.03
			0.456	<0.02	<0.02				
			1.52	<0.02	0.025				
Beef liver	0,011	0,161	0.152	<0.02	<0.02	0.020	0.021	**0.030**	0.04
			0.456	<0.02	0.022				
			1.52	0.026	0.03				
Beef kidney	0,011	0,161	0.152	0.027	0.029	0.022	0.071	**0.070**	0.2
			0.456	0.071	0.104				
			1.52	0.16	0.19				
Cow milk	0,022	0,309	0.152	<0.01	0.012	0.003	0.015	**0.015**	0.07
			0.456	0.019	0.026				
			1.52	0.071	0.143				
Sheep muscle	0,030	0,424	Extrapolation from results of feeding study on dairy cows			< 0.02	< 0.02	-	0.03
Sheep fat	0,030	0,424	Extrapolation from results of feeding study on dairy cows			< 0.02	<0.02	-	0.03
Sheep liver	0,030	0,424	Extrapolation from results of feeding study on dairy cows			0.020	0.022	**0.030**	0.04
Sheep kidney	0,030	0,424	Extrapolation from results of feeding study on dairy cows			0.023	0.097	**0.100**	0.2
Sheep milk	0,030	0,424	Extrapolation from results of feeding study on dairy cows			0.003	0.020	**0.020**	0.07
Pig muscle	0,005	0,062	Extrapolation from results of feeding study on dairy cows			< 0.02	<0.02	-	0.02 *
Pig fat	0,005	0,062	Extrapolation from results of feeding study on dairy cows			< 0.02	<0.02	-	0.02 *
Pig liver	0,005	0,062	Extrapolation from results of feeding study on dairy cows			0.020	0.019	**0.020**	0.02 *
Pig kidney	0,005	0,062	Extrapolation from results of feeding study on dairy cows			0.021	0.038	**0.040**	0.05

(a) EU MRL proposed by EFSA (EFSA Journal 2014; 12(3): 3614) – These values are shown for comparison with the potential residues resulting from the intended use of amidosulfuron WG 75 in grass.

(*) Indicates that the MRL is set at the LOQ

ND= not detected

CA 6.8 Proposed safety intervals

Study submitted and evaluated for the first inclusion of amidosulfuron on Annex I:

Report:	KCA 6.8/01 [redacted].; 2006; M-275822-01-1
Title:	Rationale for a 7-day re entry interval after treatment of pastures with Amidosulfuron - Code: AE F075032
Report No.:	M-275822-01-1
Document No.:	M-275822-01-1
Guideline(s):	not specified
Guideline deviation(s):	not specified
GLP/GEP:	**no**

Grazing animals are to be brought to treated pasture not earlier than 7 days after application. Due to the very low toxicity of amidosulfuron this re-entry period will prevent livestock from unacceptable exposure.

CA 6.9 Estimation of the potential and actual exposure through diet and other sources

Original Annex II dossier

Study submitted and evaluated for the first inclusion of amidosulfuron on Annex I:

Report:	KCA 6.9/01 [redacted].; 2002; M-210422-01-1
Title:	TMDI estimation of dietary intake of amidosulfuron (AE F075032) from residues in cereals and linseed (Statement) Code: AE F075032
Report No.:	C021005
Document No.:	M-210422-01-1
Guideline(s):	
Guideline deviation(s):	--
GLP/GEP:	**no**

Acceptable Daily Intake (ADI) and Dietary Exposure Calculation

Toxicological endpoints for amidosulfuron

Compound	Endpoint	Value (mg/kg bw)	Study	Safety factor	Reference
Amidosulfuron	Acceptable Daily Intake (ADI)	0.2	2 generation study	100	EFSA Scientific Report (2007) 116 of 14 November 2007
	Acute Reference Dose (ARfD)	No value proposed as amidosulfuron is not acutely toxic			

In order to evaluate the potential chronic exposure to amidosulfuron residues through the diet, the Theoretical Maximum Dietary Intakes (TMDI) were estimated using the EFSA PRIMo model (revision 2). For the evaluation of the chronic exposure the model uses 5 WHO diets relevant to the EU and 22 national diets from 13 different EU Member States.

Table 8.10.1- 3: input values used for TMDI calculation of amidosulfuron

Commodity	Chronic risk assessment		
	Input value (mg/kg)	Comment	Origin of the value
Cereal grain	0.01*	Median residue	EFSA Journal 2014; 12(3):3614
Linseed	0.01*	Median residue	
Beef, sheep muscle and fat	0.02*	Median residue	Derived from the livestock feeding study based on the dietary burden calculation
Beef, sheep liver	0.03	Median residue	
Beef kidney	0.07	Median residue	
Sheep kidney	0.10		
Milk	0.015	Median residue	
Sheep milk	0.02		
Pig liver	0.02	Median residue	
Pig kidney	0.04	Median residue	

* Indicates that the input value is proposed at the limit of analytical determination

As shown in **Table 8.10.1- 4**, the highest TMDI calculated for amidosulfuron represented less than 1% of the ADI, which denotes considerable margins of safety.

Table 8.10.1- 4: Highest TMDI calculated for iodosulfuron-methyl-sodium according to the EFSA model

Compound	EFSA model	Highest contributor	
	Highest TMDI (%ADI)	MS diet	Commodity / group of commodities
amidosulfuron	0.3	NL Child	Milk and milk products: cattle

Prepare workbook for refined calculations

Undo refined calculations

amidosulfuron

Status of the active substance:	CR 2015/1200	Code no.	
LOQ (mg/kg bw):		proposed LOQ:	
Toxicological end points			
ADI (mg/kg bw/day):	**0.2**	ARfD (mg/kg bw):	-
Source of ADI:		Source of ARfD:	
Year of evaluation:		Year of evaluation:	

Explain choice of toxicological reference values.

The risk assessment has been performed on the basis of the MRLs collected from Member States in April 2006. For each pesticide/commodity the highest national MRL was identified (proposed temporary MRL = pTMRL).
The pTMRLs have been submitted to EFSA in September 2006.

Chronic risk assessment

TMDI (range) in % of ADI
minimum - maximum

No of diets exceeding ADI: ---

Highest calculated TMDI values in % of ADI	MS Diet	contributor to MS diet (in % of ADI)	Commodity / group of commodities	2nd contributor to MS diet (in % of ADI)	Commodity / group of commodities	3rd contributor to MS diet (in % of ADI)	Commodity / group of commodities	pTMRLs at LOQ (in % of ADI)
0,263	NL child	0,22	Milk and milk products: Cattle	0,0	CEREALS	0,0	Bovine: Meat	
0,2	FR infant	0,2	Milk and milk products: Cattle	0,0	Bovine: Meat	0,0	CEREALS	
0,1	DE child	0,1	Milk and milk products: Cattle	0,0	CEREALS	0,0	Bovine: Meat	
0,1	ES child	0,1	Milk and milk products: Cattle	0,0	CEREALS	0,0	Bovine: Meat	
0,1	SE general population 90th percentile	0,1	Milk and milk products: Cattle	0,0	CEREALS		FRUIT (FRESH OR FROZEN)	
0,1	WHO Cluster diet B	0,1	CEREALS	0,0	Milk and milk products: Cattle	0,0	Bovine: Meat	
0,1	WHO cluster diet D	0,0	CEREALS	0,0	Milk and milk products: Cattle	0,0	Bovine: Meat	
0,1	NL general	0,0	Milk and milk products: Cattle	0,0	CEREALS	0,0	Bovine: Meat	
0,1	WHO Cluster diet F	0,0	Milk and milk products: Cattle	0,0	CEREALS	0,0	Bovine: Meat	
0,1	WHO regional European diet	0,0	Milk and milk products: Cattle	0,0	CEREALS	0,0	Bovine: Meat	
0,1	IE adult	0,0	CEREALS	0,0	Milk and milk products: Cattle	0,0	Bovine: Meat	
0,1	ES adult	0,0	Milk and milk products: Cattle	0,0	CEREALS	0,0	Bovine: Meat	
0,1	WHO cluster diet E	0,0	CEREALS	0,0	Milk and milk products: Cattle	0,0	Bovine: Meat	
0,1	DK child	0,1	CEREALS	0,0	Bovine: Liver		FRUIT (FRESH OR FROZEN)	
0,0	LT adult	0,0	Milk and milk products: Cattle	0,0	CEREALS	0,0	Bovine: Meat	
0,0	FR all population	0,0	Milk and milk products: Cattle	0,0	CEREALS	0,0	Bovine: Meat	
0,0	IT kids/toddler	0,0	CEREALS		FRUIT (FRESH OR FROZEN)		FRUIT (FRESH OR FROZEN)	
0,0	FR toddler	0,0	CEREALS	0,0	Bovine: Meat		FRUIT (FRESH OR FROZEN)	
0,0	PT General population	0,0	CEREALS		FRUIT (FRESH OR FROZEN)		FRUIT (FRESH OR FROZEN)	
0,0	IT adult	0,0	CEREALS		FRUIT (FRESH OR FROZEN)		FRUIT (FRESH OR FROZEN)	
0,0	UK Infant	0,0	CEREALS	0,0	Bovine: Liver	0,0	Bovine: Kidney	
0,0	UK Toddler	0,0	CEREALS	0,0	Bovine: Liver	0,0	Bovine: Kidney	
0,0	DK adult	0,0	CEREALS	0,0	Bovine: Meat	0,0	Bovine: Liver	
0,0	UK vegetarian	0,0	CEREALS		FRUIT (FRESH OR FROZEN)		FRUIT (FRESH OR FROZEN)	
0,0	UK Adult	0,0	CEREALS	0,0	Bovine: Liver	0,0	Bovine: Kidney	
0,0	FI adult	0,0	CEREALS		FRUIT (FRESH OR FROZEN)		FRUIT (FRESH OR FROZEN)	
0,0	PL general population	0,0	CEREALS		FRUIT (FRESH OR FROZEN)		FRUIT (FRESH OR FROZEN)	

Conclusion:
The estimated Theoretical Maximum Daily Intakes (TMDI), based on pTMRLs were below the ADI.
A long-term intake of residues of amidosulfuron is unlikely to present a public health concern.

Acute Reference Dose (ARfD) and Dietary Exposure Calculation

Acute exposure calculations were not carried out because an ARfD was not deemed necessary for amidosulfuron.

CA 6.10 Other studies

The summary for the active substance sufficiently addresses aspects of the residue situation. Therefore, other special studies are not needed.

CA 6.10.1 Effect on the residue level in pollen and bee products

Amidosulfuron is applied on cereals, pasture, meadow and flax early in the growing season and no residues are expected in pollen and bee products. Furthermore there is no guidance document indicating how to investigate the residues in pollen and bee products.