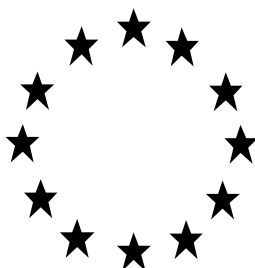


Draft Renewal Assessment Report
under Regulation (EC) 1107/2009



FORAMSULFURON

Volume 3 – B.5 (AS)

Rapporteur Member State: Finland
Co-Rapporteur Member State: Slovakia

March 2015

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2015/March	First Draft RAR

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B.5. METHODS OF ANALYSIS

Comments with respect to the Annex I renewal of approval process

This dossier contains study reports already submitted by Bayer CropScience for the Annex I inclusion of foramsulfuron, as well as new data, not yet evaluated at EU level and that was considered by the applicant to be necessary for the renewal of approval of foramsulfuron. In order to distinguish these, reference to studies in the original dossier are depicted in grey.

For summaries of studies submitted during the frame of the first Annex I inclusion please refer to the corresponding section in the Monograph. Copies of the study reports are provided in the baseline dossier (D-008962-01) provided by Bayer CropScience. Additional studies which were not submitted during the Annex I inclusion process are summarized in this document.

In this process the new studies submitted are:

- An analytical method (01360) and its ILV for the enforcement of foramsulfuron in plant material
- Modification 001 of the analytical method DGM F 03/98-0, developed for the determination of residues of foramsulfuron and its metabolite AE F153745 in/on corn green material with LC-MS/MS at a reduced limit of quantitation (LOQ) of 0.01 mg/kg.
- A HPLC-MS/MS method and its ILV validated for the determination of foramsulfuron with an LOQ of 0.01 mg/kg in milk, meat, fat, liver, kidney and eggs.
- An actual HPLC-MS/MS enforcement method (01115) and its validation for the monitoring of foramsulfuron in soil with a limit of quantification (LOQ) of 0.1 µg/kg soil.
- An actual HPLC-MS/MS enforcement method (01058) and its validation for the monitoring of foramsulfuron in surface and drinking water with a limit of quantification (LOQ) of 0.05 µg/L.
- A modified HPLC-MS/MS enforcement method (01387) for the monitoring of foramsulfuron in drinking and surface water preparing the associated ILV, with a limit of quantification (LOQ) of 0.05 µg/L.

On request, the following samples can be provided:

- Analytical standards of the purified active substance;
- Samples of the active substance as manufactured;
- Analytical standards of relevant metabolites and all other compounds included in all monitoring residue definitions; and
- Samples of reference substances for the relevant impurities.

B.5.1. METHODS USED FOR THE GENERATION OF PRE-AUTHORISATION DATA

B.5.1.1. Methods for the analysis of the active substance as manufactured

B.5.1.1.1 Determination of the pure active substance content in the active substance as manufactured and specified in the dossier submitted in support of approval under Regulation (EC) No 1107/2009

The following methods have been accessed in the previous EU review process:

Report:	KCA 4.1.1 /01;Cichy, M.;1998;M-183402-01
Title:	Determination of AE F130360 in the technical and pure active substance by HPLC (Analytical method) Code: AE F130360
Report No:	C001812
Document No:	M-183402-01-1
Guidelines:	USEPA (=EPA): OPPTS 830.1700;Deviation not specified
GLP/GEP:	no

Report:	KCA, 4.1.1/02; Cichy, M. - Kraus, S. 2000; M-194720-01-1; CHE 2000-1000
Title:	Validation of the analytical methods to determine AE F130360, ethyl acetate, toluene, cyclohexanone, tri-n-butylamine, formic acid, chloride and sulfate in technical Foramsulfuron.
Report No:	PA99/020; C006839
Document No:	M-194720-01-1
Guidelines:	EPA Product Properties Guideline; OPPTS 830.1800; EC 91/414/EEC ANNEX II 4.1
GLP/GEP:	Yes (GLP, unpublished)

The HPLC- method AM038813FP1 is used for the determination of Foramsulfuron in technical grade active substance.

Report:	KCA 4.1.1/10;Selzer, J.; Eichelmann, C.;2013;M-464431-01
Title:	Analytical method - Determination of foramsulfuron (AE F130360) in technical grade and pure active substance by high performance liquid chromatography (HPLC)
Report No:	AM038813FP1
Document No:	M-464431-01-1
Guidelines:	EPA Product Properties Guideline; OCSPP 830.1800; Reg (EC) 1107/2009 and Reg (EU) 283/2013;not specified
GLP/GEP:	no

Principle of the method: The method AM038813FP1 is a high performance liquid chromatographic method (HPLC) with UV detection. The results were obtained by comparison of the peak areas found in a sample with the peak areas from a standard containing certified reference items. The determination is performed using an external standard technique.

Analytical conditions:

Column:	50 x 4.6 mm, stainless steel
Stationary phase:	Ascentis Express RP-Amide 2.7 µm
Column temperature:	40 °C
Wavelength:	233 nm
Flow rate:	3.0 ml/min
Injection volume:	3 µl
Mobile phase:	gradient: A) acetonitrile / B) ammonium acetate c = 0.05 mol/l + acetic acid 0.05%

Approx. 100 mg of the test item (foramsulfuron, AE F130360) is weighed into a 50 ml volumetric flask, dissolved in 15 ml acetonitrile and 30 ml triethylamine (c = 0.01 mol/l), sonicated and after equilibration to room temperature make up to volume with triethylamine (c = 0.01 mol/l).

Report:	KCA 4.1.1/11;Selzer, J.; Eichelmann, C.;2013;M-464439-01
Title:	Validation of the HPLC analytical method AM038813FP1 - Determination of foramsulfuron (AE F130360) in technical grade and pure active substance by high performance liquid chromatography (HPLC)
Report No:	PA13/054
Document No:	M-464439-01-1
Guidelines:	EPA Product Properties Guideline; OCSPP 830.1800; Reg (EC) 1107/2009 and Reg (EU) 283/2013;not specified
GLP/GEP:	yes

The HPLC method AM038813FP1 for the determination of Foramsulfuron in technical grade active substance has been completely validated by checking the parameters linearity, precision/repeatability, accuracy/recovery, specificity and interference.

Linearity	6 concentrations with double measurements; range: 19.755 mg/100 ml - 395.1 mg/100 ml (98.775 g/kg - 1975.5 g/kg of the nominal concentration) of the test item; correlation coefficient r_K : 0.99988; equation: $y = 1.16365e-01 x + 1.04066$; Chromatograms (blank solution-acetonitrile/triethylamine $c = 0.01 \text{ mol/l}$, reference item solution and test item solution) are given; the function is linear in the operating range.
Precision/Repeatability	5 weighted samples (double measurements) are measure; no outliers are reported, mean repeatability: 984 g/kg; RSD: 0.18 %; acceptable according to the Horwitz equation.
Accuracy/Recovery	5 synthetic samples are measured; mean recovery: 100.0 %; RSD: 0.37 %.
Specificity/Interference	The LC/MS-spectra of reference substance and analyte in the sample show no spectral difference; the retention times are identical. No interferences are found.

The method AM038813FP1 for the determination of Foramsulfuron in technical grade active substance is found to be valid.

B.5.1.1.2 Applicability of existing CIPAC methods

Up to now there is no CIPAC method available for the determination of Foramsulfuron in technical grade active substance.

B.5.1.1.3 Determination of significant and relevant impurities and additives (such as stabilisers) in the active substance as manufactured

B.5.1.1.3 a, Methods for the analysis of significant impurities in the active substance as manufactured

For details refer to the file of confidential information in Volume 4.

B.5.1.1.3 b, Methods for the analysis of relevant impurities in the active substance as manufactured

Not relevant as there is no relevant impurity in the technical Foramsulfuron.

B.5.1.1.3 c, Methods for the analysis of additives in the active substance as manufactured

Not relevant as there is no additive in the technical Foramsulfuron.

B.5.1.2. Methods for risk assessment

B.5.1.2.1 Methods in or on plants, plant products, processed food commodities, food of plant and animal origin, feed and any additional matrices used in support of residues studies

Analytical methods for data generation in plant products

In the scope of the original submission in 2000, an analytical method was provided for the determination of foramsulfuron (AE F130360) in plant products. This method, DGM F03/98-0 (M-187207-01-1; KCA 4.1.2/01), was used for data generation for residues of foramsulfuron and its metabolite AE F153745 in corn matrices in Europe and included the determination of foramsulfuron via HPLC-MS/MS.

Report:	KCA 4.1.2 /01;Wrede, A.;1999;M-187207-01
Title:	Validation and analytical method for the determination of residues of AE F130360, iodosulfuron-methyl-sodium (AE F115008), and the metabolites AE F153745 and AE F075736 in maize by LC/MS/MS Code: AE F130360, AE F115008
Report No:	C004020
Document No(s):	Report includes Trial Nos.: CR98/022 M-187207-01-1
Guidelines:	Deviation not specified
GLP/GEP:	no

Table 5.1.2.1-1: EU conclusions: Analytical methods for residues of foramsulfuron (AE F130360) and metabolite AE F153745 in plants

Method No.	Matrix	Analytes	LOQ (mg/kg)	Technique	Doc. No	Reference
Data generation method						
DGM F03/98-0 (also used as enforcement)	Maize grain	AE F130360 AE F153745	0.01	LC-MS/MS	M-187207-01-1	DAR (01 April 2001); KCA 4.1.2/01
	Maize shoot and cob		0.05			

The limit of quantification (LOQ) was 0.01 mg/kg in corn grain and 0.05 mg/kg in corn shoot and cob. This method was considered appropriate for the determination of foramsulfuron residues.

The modification 001 of the analytical method DGMF 03/98-0 was developed for the determination of residues of Metsulfuron-methyl, Iodosulfuron-methyl-sodium, Foramsulfuron and its metabolite AE F153745 in/on corn green material with HPLC-MS/MS at a reduced limit of quantitation (LOQ) of 0.01 mg/kg.

Report:	KCA 4.1.2 /02;Stuke, S.;2013;M-461902-01-1
Title:	Modification M001 of the residue analytical method DGM F 03/98-0 for the determination of metsulfuron-methyl (AE F075736), iodosulfuron-methyl-sodium (AE F115008), foramsulfuron (AE F130360), AE F153745 in corn (green material) by HPLC-MS/MS at a LOQ of 0.01 mg/kg
Report No:	MR-13/047
Document No:	M-461902-01-1
Guidelines:	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 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 rev. 4, 11/07/00 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;not specified
GLP/GEP:	yes

Principle of the method

The sample preparation and essential parts of the instrumental analysis are conducted as described in the original method, DGM F 03/98-0, (A. Wrede, 1999; [M-187207-01-1](#); KCA 4.1.2/01). Minor adaptations in the instrumental analysis reflect the different instrumentation like modern sub-2µ HPLC fused-core-columns and more sensitive mass spectrometers. All residue results are expressed as the individual analytes.

10 g of the homogenised sample of corn, green material are twice extracted with 30 mL acetonitrile / triethylamine 0.02 mol/L (8:2, v/v) using an Ultraturrax. After centrifugation and decantation the combined

extracts are liquid-liquid partitioned with n-hexane. The volume of the separated acetonitrile/water phase is reduced on a vacuum rotary evaporator and made up to a defined volume. After filtration this solution is subjected to HPLC-MS/MS.

Validation of the method

Samples were measured by two fortification levels 0.01 mg/kg and 0.1 mg/kg in quintuplet, three untreated control samples of different origin were examined.

Two MRM transitions were monitored for each analyte and each matrix tested.

	Foramsulfuron	AE F153745
Linearity	Detector response was linear for matrix standards ranging from 0.4 µg/L to 20 µg/L. The correlation coefficient was 0.9970.	Detector response was linear for matrix standards ranging from 0.4 µg/L to 30 µg/L. The correlation coefficient was 0.9981.
Precision/Repeatability	Relative standard deviations were below 20% for all analytes and sample materials.	
Accuracy/Recovery	Mean recoveries for each fortification level and the overall mean recovery were within the range 70 - 110%.	
Specificity/Interferences	Apparent residues in control samples were below $0.3 \times \text{LOQ}$, m/z 453→272 for quantitation and m/z 453→139 for confirmation	Apparent residues in control samples were below $0.3 \times \text{LOQ}$, m/z 272→255 for quantitation and m/z 272→136 for confirmation
LOQ and LOD	LOQ = 0.01 mg/kg for each single analyte in corn green material, LOD ranged from 0.0009 to 0.0012 mg/kg	

Table 5.1.2.1-2 Recovery efficiency of analysis of foramsulfuron in corn green material - Quantifier Mass Transition 453→272

Sample material	Analyte	Fortification Level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual					Mean		
Corn green material	Foramsulfuron	0.01	73	80	79	74	79	77	4.2	5
		0.10	71	71	71	73	76	72	3.0	5
	Overall							75	4.8	10

RSD = relative standard deviation

Table 5.1.2.1-3 Recovery efficiency of analysis of foramsulfuron in corn green material - Qualifier Mass Transition 453→139

Sample material	Analyte	Fortification Level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual					Mean		
Corn green material	Foramsulfuron	0.01	77	83	82	72	90	81	8.4	5
		0.10	76	70	77	78	78	76	4.4	5
	Overall							78	7.3	10

RSD = relative standard deviation

Table 5.1.2.1-4 Recovery efficiency of analysis of AE F153745 in corn green material - Quantifier Mass Transition 272→255

Sample material	Analyte	Fortification Level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual					Mean		
Corn green material	AE F153745	0.01	82	86	87	83	88	85	3.0	5
		0.10	85	86	84	84	89	86	2.4	5
	Overall							85	2.6	10

RSD = relative standard deviation

Table 5.1.2.1-5 Recovery efficiency of analysis of AE F153745 in corn green material - Qualifier Mass Transition 272→136

Sample material	Analyte	Fortification Level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual					Mean		
Corn green material	AE F153745	0.01	87	93	95	87	87	90	4.3	5
		0.10	81	84	83	81	88	83	3.5	5
	Overall							87	5.4	10

RSD = relative standard deviation

Conclusion

The method modification meets all guideline criteria to determine residues of foramsulfuron and AE F153745 in/on samples from corn green material at a LOQ of 0.01 mg/kg. However, no ILV has been performed, which is considered as a data gap.

Analytical methods for data generation in food of animal origin products

According to the Guidance document on residue analytical methods (SANCO 825/00 rev. 6, 20- June 2000), the submission of an analytical method for the determination of residues in food of animal origin was not necessary because no metabolism study in animals was required according to Directive 96/68/EC.

B.5.1.2.2 Methods in soil, water, sediment, air and any additional matrices used in support of environmental fate studies

Data generation methods used in the relevant studies on the fate of the active substances and its metabolites in soil, water, sediment and air are described in detail in the corresponding reports. Please refer to dRAR 9 Vol 3CA B7

B.5.1.2.3 Methods in soil, water and any additional matrices used in support of efficacy studies

No specific methods for the support of efficacy studies were developed.

B.5.1.2.4 Methods in soil, water, sediment, feed and any additional matrices used in support of ecotoxicology studies

The methods of analysis for ecotoxicology studies are specific for the respective eco-toxicity study. Please refer to dRAR 10 Vol 3CA B8.

B.5.1.2.5 Methods in feed, body fluids and tissues, air and any additional matrices used in support of toxicological studies

The methods of analysis for feed are specific for the respective toxicity study. Please refer to dRAR 8 Vol 3CA B6

B.5.1.2.6 Methods in body fluids, air and any additional matrices used in support of operator, worker, resident and bystander exposure studies

The risk evaluation for operators, workers, bystanders and residents demonstrates that experimental exposure studies in support of risk assessment are not necessary. Therefore, methods for body fluids and tissues are not required.

B.5.1.2.7 Methods in water, buffer solutions, organic solvents and any additional matrices resulting from the physical and chemical properties tests

The methods of analysis used for physical and chemical properties tests are described in the respective phys.-chem. study. Please refer to dRAR 4 Vol 3CA B2.

B.5.2. METHODS FOR POST-APPROVAL CONTROL AND MONITORING PURPOSES

(Methods for the determination of all components included in the monitoring residue definition as submitted in accordance with the provision of point 6.7.1 in order to enable Member States to determine compliance with established maximum residue levels (MRLs); they shall cover residues in or on food and feed of plant and animal origin)

B.5.2.1. Description of analytical methods for the determination of residues in plant products

In the scope of the original submission, in 2000, several analytical methods were provided for the monitoring of foramsulfuron (AE F130360) in plant products. The data generation method (Wrede, 1999, [M-187207-01-1](#); KCA 4.2/01) was also used as enforcement and it was validated by an independent laboratory validation (Reichert, 2000; [M-198922-01-1](#); KCA 4.2/02). This method was considered adequate during the evaluation.

Residues of foramsulfuron can be determined by HPLC-UV according to the method CF/01/00 (Williams, 2000; [M-238559-01-1](#); KCA 4.2/03). This was proposed as enforcement method, although during the method validation and ILV some difficulties occurred (Campbell, 2000; [M-196608-01-1](#); KCA 4.2/04).

Table 5.2.1- 1: Analytical methods for residues of foramsulfuron (AE F130360) and metabolite AE F153745 in plants

F153745 in plants							
Method no.	Matrix	Analytes	LOQ (mg/kg)	Technique	Doc. No.	References	
Plant monitoring method							
DGM F03/98 (also used as enforcement)	Maize grain Maize shoot and cob	AE F130360 AE F153745	0.01	LC-MS/MS	M-187207-01-1	KCA 4.2/01	DAR (01 April 2001)
	Maize shoot and cob		0.05				
ILV	Maize grain	AE F130360	0.01	LC-MS/MS	M-198922-01-1	KCA 4.2/02	DAR (01 April 2001)
CF/01/00	Maize grain	AE F130360	0.05	LC-UV	M-238559-01-1	KCA 4.2/03	
Statement	-	-	-	-	M-196608-01-1	KCA 4.2/04	

The following plant monitoring methods were submitted and evaluated for the first inclusion of foramsulfuron at European level:

The cited methods:

Report:	KCA 4.2./01;Wrede, A.;1999;M-187207-01-1
Title:	Validation and analytical method for the determination of residues of AE F130360, iodosulfuron-methyl-sodium (AE F115008), and the metabolites AE F153745 and AE F075736 in maize by LC/MS/MS Code: AE F130360, AE F115008
Report No:	C004020
Document No(s):	Report includes Trial Nos.: CR98/022 M-187207-01-1
Guidelines:	Deviation not specified
GLP/GEP:	no

Report:	KCA 4.2 /02;Reichert, N.;2000;M-198922-01-1
Title:	Independent laboratory validation of a method of analysis for the determination of AE F130360 in corn grain
Report No:	C010383
Document No:	M-198922-01-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

Report:	KCA 4.2 /03;Williams, L. E.;2000;M-238559-01-1
Title:	Analytical method for the determination of residues of AE F130360 in corn grain by high performance liquid chromatography using UV detection: AE F130360
Report No:	B002828
Document No:	M-238559-01-1
Guidelines:	Deviation not specified
GLP/GEP:	no

Report:	KCA 4.2/04;Campbell, J. K.;2000;M-196608-01-1
Title:	A statement on the analytical method for the determination of residues of AE F130360 in corn grain by high performance liquid chromatography using UV detection Code: AE F130360
Report No:	C007766
Document No:	M-196608-01-1
Guidelines:	Deviation not specified
GLP/GEP:	no

Subsequent to the original dossier, additional plant monitoring methods have been developed and validated on different commodity groups.

Table 5.2.1- 2 : Supplementary analytical methods for residues of foramsulfuron (AE F130360) in plants

Method no.	Matrix	Analytes	LOQ (mg/kg)	Technique	Doc. No.	References
Plant monitoring methods						
EM F08/99-0 (renamed 00815)	Wheat grain	AE F130360	0.01	LC-MS/MS	M-194528-01-1 M-194531-01-1	KCA 4.2./15 and 16
	Wheat straw and shoot		0.05			
	Lemon Tomato Maize kernel	AE F130360	0.01	LC-MS/MS	M-212674-01-1	KCA 4.2./17
00815/M001 supplement to monitoring method	Wheat grain, Flax grain, pomace wet, oil	AE F130360	0.01	HPLC-MS/MS	M-226888-01-1	KCA 4.2./18
	Wheat straw and shoot, Flax plant without root		0.05			
EM F08/99-0 (renamed 00815) ILV	Lemon Tomato	AE F130360	0.01	LC-MS/MS	M-215456-01-1	KCA 4.2./19

Report:	KCA 4.2 /15;Wrede, A.;2000;M-194528-01-1
Title:	Enforcement Method for Cereal Grain, Straw and Shoot by LC-MS/MS Amidosulfuron (AE F075032) Metsulfuron-methyl (AE F075736) Iodosulfuron-methyl-sodium (AE F115008) AE F130060 AE F130360
Report No:	C006734
Document No:	M-194528-01-1
Guidelines:	not specified;not specified
GLP/GEP:	no

Report:	KCA 4.2 /16;Wrede, A.;2000;M-194531-01-1
Title:	Validation of the Enforcement Method EM F08/99-0 of cereal grain, straw and shoot by LC-MS/MS - Amidosulfuron (AE F075032) - Metsulfuron-methyl (AE F075736) Iodosulfuron-methyl-sodium (AE F115008) - AE F130060 - AE F130360
Report No:	C006735
Document No:	M-194531-01-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

Report:	KCA 4.2 /17;Wrede, A.;2002;M-212674-01-1
Title:	Validation of the enforcement method EM F08/99-0 for lemon, tomato and maize kernel by LC-MS/MS - Amidosulfuron (AE F075032) - Iodosulfuron-methyl-sodium (AE F115008) - Mesosulfuron-methyl (AE F130060) - Foramsulfuron (AE F130360)
Report No:	C022220
Document No:	M-212674-01-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

The LC-MS/MS method EM F 08/99-0, suitable for both data generation and enforcement, was validated in wheat (shoot, straw and grain), lemon, tomato, and maize kernel. This residue analytical method was validated for five sulfonylureas: amidosulfuron (AE F075032), iodosulfuron-methyl-sodium (AE F115008), mesosulfuron-methyl (AE F130060), foramsulfuron (AE F130360) and metsulfuron-methyl (AE F075736). For clarity reasons, only results of foramsulfuron are reported below.

Principle of the method

Samples were extracted with acetonitrile/ aqueous triethylamine 0.02 mol/L (4:1, v/v). After clean-up by liquid/liquid extraction with hexane and acetonitrile/triethylamine 0.02 mol/L (4:1, v/v) and a second liquid/liquid extraction with ethyl acetate/diluted formic acid (0.01 mol/L), foramsulfuron was determined by LC-MS/MS. The following mass transition was used for quantification of foramsulfuron $m/z = 453.2(\text{MH}^+) \rightarrow m/z = 182.0$ (cleavage alpha of the carbonyl).

Validation of the method

The determination of the residues was done with matrix-matched standards.

Samples were measured by two fortification levels 0.01 mg/kg and 0.1 mg/kg for wheat grain and 0.05 mg/kg and 0.5 mg/kg for wheat straw and shoot in quintuplet.

Linearity	Wheat straw and shoot: Detector response was quadratic for matrix standards ranging from 10 µg/L to 500 µg/L (n = 6; analysed in duplicate). The correlation coefficient was higher than 0.99. Wheat grain, lemon, tomato and maize kernel: Detector response was quadratic for matrix standard ranging from 5 µg/L to 200 µg/L (n = 6; analysed in duplicate). The correlation coefficient was higher than 0.99.
Precision/Repeatability	Relative standard deviations were below 20% for all analytes and sample materials.
Accuracy/Recovery	Mean recoveries for each fortification level and the overall mean recovery were within the range 70 - 110%. Except tomato where mean recovery ranged from 115% to 120%.
Specificity/Interferences	No interference was observed. All control samples of cereal (grain, straw, and shoot), lemon, tomatoes, and maize kernels showed no residues higher than 30% of the LOQ
LOQ	LOQ was determined as 0.01 mg/kg for foramsulfuron in wheat grain, lemon, tomato and maize kernel. LOQ was determined as 0.05 mg/kg for foramsulfuron in wheat straw and shoot.

Table 5.2.1.-3 : Recovery efficiency of analysis of foramsulfuron in wheat (shoot, straw, grain), lemon tomato and maize kernel

Sample material	Fortification level [mg/kg]	Recoveries [%]						RSD [%]	n
		Individual					Mean		
Wheat grain	0.01	87	92	86	79	73	83	9	5
	0.10	95	101	85	105	92	96	8	5
Wheat straw	0.05	72	75	80	81	89	79	8	5
	0.50	80	87	87	94	72	84	10	5
Wheat shoot	0.05	74	78	82	79	78	78	4	5
	0.50	88	82	77	79	79	81	5	5
lemon*	0.01	108	83	106	112	96	101	12	5
	0.10	86	83	104	95	101	94	10	5
lemon*	0.01	90	106	91	100	88	95	8	5
	0.10	77	82	89	66	63	75	14	5
lemon*	0.01	94	93	103	96	109	99	7	5
	0.10	103	96	96	116	108	104	8	5
tomato	0.01	115	107	111	123	120	115	6	5
	0.10	119	123	117	113	128	120	5	5
maize kernel	0.01	88	79	111	103	110	98	14	5
	0.10	96	99	104	93	98	98	4	5

RSD: relative standard deviation

* lemon from three different origins were used for the validation

Based on the revision of the guideline (SANCO 825/00 rev.8.1), LC-MS/MS methods are not highly selective and specific when only one mass transition has been validated so a confirmatory method is required.

Report:	KCA 4.2 /18;Heinemann, O.;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(s):	Report includes Trial Nos.: P602033000 M-226888-01-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

This analytical method describes the determination of residues of amidosulfuron, iodosulfuron-methyl-sodium, foramsulfuron, metsulfuron-methyl, and mesosulfuron-methyl in several matrices of plant origin. In addition to cereal commodities (wheat shoot, grain and straw), the procedure was also validated for oily commodities (flax seed and oil). In the context of this AIR dossier, only the results for foramsulfuron will be discussed.

Principle of the Method

The residues were extracted by high-speed maceration with an acetonitrile/triethylamine mixture (0.2 mol/L (4:1, v/v)). After filtration the extract was purified by liquid-liquid extraction with *n*-hexane and acetonitrile/triethylamine mixture (0.2 mol/L (4:1, v/v)). The residues were then partitioned on a Chromabond XTR column with ethyl acetate and the final extract was measured by LC-MS/MS

The following transition ion was used for quantification of foramsulfuron $m/z = 453 \rightarrow m/z = 182$.

The modification is an addition of fatty products to the validation and modification of the extraction and

clean up procedures. Moreover, study of stability was also performed.

These changes are not considered as relevant, this modification cannot be considered as a confirmatory method.

Validation of the method

The determination of the residues was done with matrix-matched standards.

Samples were measured by two fortification levels 0.01 mg/kg and 0.1 mg/kg for flax (grain, pomace wet, oil), wheat (grain) and 0.05 mg/kg and 0.5 mg/kg for flax (plant without root), wheat (green material, straw) in quintuplet.

Linearity	Detector response was linear for matrix standards ranging from 0.5 µg/L to 500 µg/L (n = 7; analysed in triplicate). The correlation coefficient was higher than 0.99.
Precision/Repeatability	The overall mean RSDs ranged from 2.6% to 10.7% in wheat matrices and between 3.7% and 6.4% in flax matrices.
Accuracy/Recovery	The overall mean recovery of foramsulfuron ranged between 81% and 93% in wheat matrices and between 79% and 99% in flax matrices.
Specificity/Interferences	No interference was observed. All control samples showed no residues higher than 30% of the LOQ.
LOQ	The LOQ for foramsulfuron was 0.01 mg/kg in wheat grain, flax (grain, pomace wet, oil) and 0.05 mg/kg in wheat green material, straw and flax (plant without root).

Table 5.2.1.-4 : Recovery efficiency of analysis of foramsulfuron in wheat (shoot, straw, grain) and flax (grain, oil, pomace wet, plant without root)

Sample material	Analyte	Fortification level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual values					Mean		
Wheat green material	Foramsulfuron	0.05	85	84	91	88	89	87	3.3	5
		0.50	89	90	88	89	91	89	1.3	5
Wheat straw	Foramsulfuron	0.05	93	100	88	95	89	93	5.2	5
		0.50	97	97	87	91	88	92	5.2	5
Wheat grain	Foramsulfuron	0.01	80	83	94	96	78	86	9.6	5
		0.10	83	70	70	78	77	76	7.4	5
Flax grain	Foramsulfuron	0.01	79	83	78	76	71	77	5.7	5
		0.10	81	85	80	74	78	80	5.1	5
Flax oil	Foramsulfuron	0.01	79	83	74	88	79	81	6.5	5
		0.10	85	86	90	89	89	88	2.5	5
Flax pomace wet	Foramsulfuron	0.01	93	84	84	84	86	86	4.5	5
		0.10	83	84	80	80	76	81	3.9	5
Flax plant without root	Foramsulfuron	0.05	102	96	104	104	94	100	4.7	5
		0.50	99	94	98	99	98	98	2.1	5

RSD: relative standard deviation

Stability

The stability was tested after storage in a refrigerator (4°C) during two months for solvents and standards and during two weeks for samples and found acceptable.

Conclusion:

The proposed analytical method was previously fully validated for the determination of foramsulfuron in dry products, matrices with high water content and acidic matrices with LOQ = 0.01 mg/kg. This method has also been validated in fatty products with LOQ = 0.01 mg/kg. Nevertheless, this method is not highly specific (only a mass transition was validated) so a confirmatory method is necessary.

Independent Laboratory Validation

Independent laboratory validations of the original method EMF08/99-0 are available in the monograph and cover cereals and dry matrices. Nevertheless, an ILV of the method supplement 00815/M001 is provided below.

Report:	KCA 4.2 /19;Reichert, N.; Klimmek, S.;2002;M-215456-01-1
Title:	Independent laboratory validation of the analytical method EM F08/99-0 for the residue analysis of Amidosulfuron (AE F075032), Iodosulfuron-methyl-sodium (AE F115008), Mesosulfuron-methyl (AE F130060), Foramsulfuron (AE F130360) in tomato and citrus
Report No:	C023679
Document No:	M-215456-01-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

Principle of the ILV

The analytical method EM F08/99-0 (Wrede, A.; 2000; [M-194528-01-1](#); KCA4.2/15) was followed for lemon and tomato. The following transition ion was used for quantification for foramsulfuron $m/z = 453.27 \rightarrow m/z = 182.17$.

Validation of the method

The determination of the residues was done with matrix-matched standards.

Samples were measured by two fortification levels 0.01 mg/kg and 0.1 mg/kg in quintuplet.

Linearity	Detector response was quadratic for matrix standards ranging from 5 µg/L to 80 µg/L (n = 6). The correlation coefficient was higher than 0.99.
Precision/Repeatability	The overall mean RSDs ranged from 2% to 8%.
Accuracy/Recovery	The overall mean recovery of foramsulfuron ranged between 88% and 99%.
Specificity/Interferences	No interference was observed. All control samples showed no residues higher than 30% of the LOQ.
LOQ	The limit of quantification was established at 0.01 mg/kg for foramsulfuron in tomato and lemon.

Table 5.2.1.-5 : Recovery efficiency of analysis of foramsulfuron in lemon and tomato

Sample material	Analyte	Fortification level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual values					Mean		
Lemon	Foramsulfuron	0.01	98	99	84	99	88	94	8	5
		0.10	89	83	88	91	89	88	3	5
Tomato	Foramsulfuron	0.01	93	89	93	97	97	94	4	5
		0.10	101	97	98	98	100	99	2	5

RSD: relative standard deviation

Conclusion:

The proposed ILV is fully validated for the determination of foramsulfuron in matrices with high water content and in acidic matrices with a LOQ = 0.01 mg/kg.

During the peer review under Directive 91/414/EEC, an analytical method using HPLC-MS/MS (Wrede, A.;1999; [M-187207-01-1](#); KCA 4.2/01) and its ILV (Reichert, 2000; [M-198922-01-1](#); KCA 4.2/02) were evaluated and validated for determination of parent foramsulfuron in plant matrices with an LOQ of 0.01 mg/kg in dry commodities (maize grain) and an LOQ of 0.05 mg/kg in maize cob and straw. Nevertheless, a confirmatory method was missing. Hence, EFSA concluded that parent foramsulfuron can be enforced in food of plant origin with an LOQ of 0.01 mg/kg in dry commodities and 0.05 mg/kg maize cob and straw but a confirmatory method was required (EFSA Journal 2012;10(11):2962; [M-466418-01-1](#); KCA 6/01).

Available plant enforcement methods for sulfonylureas are not highly specific. Consequently a new enforcement method, 01360, has been developed in 2013 for the determination of 5 sulfonylureas in plant matrices and it has been validated for their determination with a LOQ of 0.01 mg/kg for each single analyte in all matrices tested (Stuke, S.; Ballmann, C. 2013; [M-455564-01-1](#); KCA 4.2/20). The 5 crop groups are represented by sugar beet body (high starch content), sugar beet leaf (high water content), lemon fruit (high acid content), oilseed rape (high oil/fat content) and wheat straw (high protein content, dry commodities). This method and its Independent Lab Validation (ILV) are summarised below.

Table 5.2.1- 6: Summary of the new residue analytical method and its ILV

Method n°	Matrix	Analytes	LOQ (mg/kg)	Technique	Doc. No.	Ref.
Plant monitoring methods						
01360	Sugar beet body Sugar beet leaf Lemon Oilseed rape Wheat straw	AE F130360	0.01	HPLC-MS/MS	M-455564-01-1	KCA 4.2./20
ILV	Sugar beet body Sugar beet leaf Lemon Oilseed rape Wheat straw	AE F130360	0.01	HPLC-MS/MS	M-470160-01-1	KCA 4.2./21

Report:	KCA 4.2 /20;Stuke, S.; Ballmann, C.;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
Guidelines:	<p>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</p> <p>Guidance document on residue analytical methods, SANCO/825/00/rev. 8.1, European Commission, Directorate General Health and Consumer Protection 16/11/2010</p> <p>US EPA Residue Chemistry Test Guideline OCSPP 860.1340: Residue Analytical Method</p> <p>OECD Guideline, ENV/JM/MONO (2007) 17, Aug 13, 2007;not applicable</p>
GLP/GEP:	yes

Principle of the method

The sample preparation is based on the official QuEChERS method (unbuffered). Residues of these analytes were extracted from the plant samples with a mixture of acetonitrile : water 1:1. Additional water was added to oilseed rape and straw samples to adjust the water content of the extraction solvent. The extraction was conducted using an automated over-head shaker (shaking time 15 minutes considering the latest recommendations of M. Anastassiades, publisher of the QuEChERS method at the LVUA Stuttgart, Germany. Magnesium sulfate and sodium chloride were added and shaken again for 1 minute. After centrifugation the supernatant was diluted with Milli-Q water/acetonitrile and triethylamine (for oilseed rape, no acetonitrile was used). After filtration an aliquot of the extract was injected into a high performance liquid chromatograph and subjected to reversed phased chromatography coupled with tandem mass spectrometry (MS/MS). Two MRM transitions were monitored for each analyte and each matrix tested; for foramsulfuron m/z 451→296 for quantitation and m/z 451→268 for confirmation.

Valication of the method

The determination of the residues was done with matrix-matched standards.

Samples were measured by two fortification levels 0.01 mg/kg and 0.1 mg/kg in quintuplet.

Linearity	Detector response was linear for matrix standards ranging from 0.2 µg/L to 25 µg/L (or 50 µg/L in each matrix). The correlation coefficient ranged from 0.9945 to 0.9999.
Precision/Repeatability	Relative standard deviations were below 20% for all analytes and sample materials.
Accuracy/Recovery	Mean recoveries for each fortification level and the overall mean recovery were within the range 70 - 110%.
Specificity/Interferences	Apparent residues in control samples were below $0.3 \times \text{LOQ}$. The recoveries were not corrected for interferences. Two MRM transitions were monitored for each analyte and each matrix tested, therefore, the HPLC-MS/MS method is highly specific and an additional confirmatory method is not necessary.
LOQ adn LOD	The limit of quantitation (LOQ) for each single analyte is 0.01 mg/kg in all matrices tested. The limit of detection (LOD) ranged from 0.0007 to 0.0023 µg/kg.

Table 5.2.1-7 Recovery efficiency of analysis of foramsulfuron in sugar beet (body, leaf), lemon, oilseed rape and wheat (straw)-1st MRM (m/z 451→296)

Sample material	Analyte	Fortification level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual values					Mean		
Sugar beet, body	Foramsulfuron	0.01	96	96	98	103	102	99	3.4	5
		0.10	101	92	94	100	99	97	4.1	5
	Overall							98	3.6	10
Sugar beet, leaf	Foramsulfuron	0.01	81	94	86	83	81	85	6.4	5
		0.10	84	89	89	93	89	89	3.6	5
	Overall							87	5.4	10
Lemon, fruit	Foramsulfuron	0.01	94	94	89	92	92	92	2.2	5
		0.10	97	91	92	88	93	92	3.5	5
	Overall							92	2.8	10
Oilseed rape	Foramsulfuron	0.01	90	91	88	85	98	90	5.3	5
		0.10	96	97	100	95	96	97	2.0	5
	Overall							94	5.2	10
Cereals straw	Foramsulfuron	0.01	76	78	72	71	69	73	5.1	5
		0.10	78	77	76	72	74	75	3.2	5
	Overall							74	4.3	10

RSD = relative standard deviation

Table 5.2.1-8 : Recovery efficiency of analysis of foramsulfuron in sugar beet (body, leaf), lemon, oilseed rape and wheat (straw)-2nd MRM (m/z 451→268)

Sample material	Analyte	Fortification level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual values					Mean		
Sugar beet, body	Foramsulfuron	0.01	93	94	97	106	101	98	5.5	5
		0.10	93	96	88	98	93	94	4.0	5
	Overall							96	5.2	10
Sugar beet, leaf	Foramsulfuron	0.01	89	88	94	91	95	91	3.3	5
		0.10	87	84	87	87	90	87	2.4	5
	Overall							89	3.8	10
Lemon, fruit	Foramsulfuron	0.01	93	97	90	92	96	94	3.1	5
		0.10	95	96	92	89	97	94	3.5	5
	Overall							94	3.1	10
Oilseed rape	Foramsulfuron	0.01	91	94	91	99	97	94	3.8	5
		0.10	96	98	102	99	96	98	2.5	5

	Overall							96	3.7	10
Cereals straw	Foramsulfuron	0.01	75	74	74	81	79	77	4.2	5
		0.10	79	72	74	75	74	75	3.5	5
	Overall							76	3.8	10

RSD = relative standard deviation

Stability of sample extracts

The stability in final plant extracts was checked for the tested sample materials over a period of 16 to 43 days at $4^{\circ}\text{C} \pm 3^{\circ}\text{C}$ under dark conditions. foramsulfuron was stable for all matrices at the given conditions.

The results suggest that samples should be analysed as soon as possible after preparation, because not all analytes are stable in final plant extracts. This is not surprising when considering the hydrolytical data of sulfonyl ureas.

Table 5.2.1.- 9: Stability of Foramsulfuron in Plant Extracts

Sample Material	Fortification Level [mg/kg]		Recovery Rates [%]					Mean
Sugar beet, body	0.1	Day 0 (initial analysis)	101	92	94	100	99	
		43 days reanalysis	97	92	90	94	95	
		deviation day 0/43 days	4.0	0.0	4.3	6.0	4.0	3.7
Sugar beet, leaf	0.1	Day 0 (initial analysis)	84	89	89	93	89	
		43 days reanalysis	84	81	87	87	82	
		deviation day 0/43 days	0.0	9.0	2.2	6.5	7.9	5.1
Lemon, fruit	0.1	Day 0 (initial analysis)	97	91	92	88	93	
		16 days reanalysis	105	104	93	94	97	
		deviation day 0/16 days	8.2	14.3	1.1	6.8	4.3	6.9
Oilseed Rape	0.1	Day 0 (initial analysis)	96	97	100	95	96	
		38 days reanalysis	81	79	80	79	81	
		deviation day 0/38 days	15.6	18.6	20.0	16.8	15.6	17.3
Cereals Straw	0.1	Day 0 (initial analysis)	78	77	76	72	74	
		30 days reanalysis	103	105	105	98	96	
		deviation day 0/30 days	32.1	36.4	38.2	36.1	29.7	34.5

Conclusion

The method is suitable as an enforcement method for the determination of foramsulfuron in/on samples from plant origin at a LOQ of 0.01 mg/kg.

An independent lab validation (ILV) of the analytical BCS method 01360 for the determination of Amidosulfuron, Metsulfuron-methyl, Iodosulfuron-methyl, Mesosulfuron-methyl and Foramsulfuron residues in/on plant materials was conducted. The five crop groups are represented by sugar beet body (high starch content), sugar beet leaf (high water content), lemon fruit (high acid content), oilseed rape (high oil/fat content) and wheat straw (high protein content, dry commodities).

Report:	KCA 4.2 /21;Konrad, S.;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

Guidelines:	<p>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.</p> <p>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.</p> <p>Guidance document on residue analytical methods; SANCO/825/00 rev. 8.1, European Commission, Directorate General Health and Consumer Protection; 2010-11-16.</p> <p>OECD Guidance Document on Pesticide Residue analytical Methods; ENV/JM/Mono (2007); 2007-08-13</p> <p>US EPA Residue Chemistry Test Guideline OCSPP 860.1340: Residue Analytical Method;not applicable</p>
GLP/GEP:	yes

Principle of the ILV

The analytical method 01360 ([Stuke, S.; Ballmann, C.;2013;M-455564-01-1, KCA 4.2 /20](#)) was followed for sugar beet body (high starch content), sugar beet leaf (high water content), lemon fruit (high acid content), oilseed rape (high oil/fat content) and wheat straw (high protein content, dry commodities). Two MRM transitions were monitored for each analyte and each matrix tested; for foramsulfuron m/z 451→296 for quantitation and m/z 451→268 for confirmation.

Validation of the method

The determination of the residues was done with matrix-matched standards.

Samples were measured by two fortification levels 0.01 mg/kg and 0.1 mg/kg in quintuplet.

Linearity	Detector response was linear for matrix standards ranging from 0.2 µg/L to 25 µg/L (expressed as parent equivalents). The correlation coefficients were higher than 0.99.
Precision/Repeatability	Relative standard deviations were below 20% for all analytes and sample materials.
Accuracy/Recovery	Mean recoveries for each fortification level and the overall mean recovery were within the range 70 - 110%.
Specificity/Interferences	Apparent residues in control samples were below $0.3 \times \text{LOQ}$. The recoveries were not corrected for interferences. Two MRM transitions were monitored for each analyte and each matrix tested, therefore, the HPLC-MS/MS method is highly specific and an additional confirmatory method is not necessary. Matrix effects were observed for all matrices. It was decided to use matrix-matched standards for all matrices in order to compensate all possible matrix effects.
LOQ adn LOD	For all matrices the limit of quantitation (LOQ) for each single analyte is 0.01 mg/kg and the limit of detection (LOD) is 0.003 mg/kg.

Table 5.2.1-10: Recovery efficiency of analysis of foramsulfuron in sugar beet (body, leaf), lemon , oilseed rape and wheat (straw)-1st MRM (m/z 451→296)

Sample material	Analyte	Fortification level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual values					Mean		
Sugar beet, body	Foramsulfuron	0.01	94	98	88	96	93	94	4.0	5
		0.10	102	100	103	96	99	100	2.7	5
	Overall							97	4.6	10
Sugar beet, leaf	Foramsulfuron	0.01	89	89	89	87	92	89	1.7	5
		0.10	90	100	96	89	93	94	4.7	5
	Overall							92	4.3	10
Lemon, fruit	Foramsulfuron	0.01	70	81	75	76	80	76	5.5	5
		0.10	94	88	88	86	87	88	3.9	5
	Overall							82	8.9	10
Oilseed rape	Foramsulfuron	0.01	100	106	103	87	107	101	7.9	5
		0.10	106	101	103	103	104	103	1.8	5
	Overall							102	5.5	10
Cereals straw	Foramsulfuron	0.01	86	84	74	71	72	77	9.4	5
		0.10	77	79	78	80	73	77	3.2	5
	Overall							77	6.6	10

RSD = relative standard deviation

Table 5.2.1-11: Recovery efficiency of analysis of foramsulfuron in sugar beet (body, leaf), lemon, oilseed rape and wheat (straw)-2nd MRM (m/z 451→268)

Sample material	Analyte	Fortification level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual values					Mean		
Sugar beet, body	Foramsulfuron	0.01	95	91	87	98	98	94	5.0	5
		0.10	102	99	98	87	96	97	5.7	5
	Overall							95	5.3	10
Sugar beet, leaf	Foramsulfuron	0.01	98	100	85	90	98	94	6.8	5
		0.10	94	107	101	92	95	98	6.2	5
	Overall							96	6.5	10
Lemon, fruit	Foramsulfuron	0.01	78	82	77	70	79	77	5.6	5
		0.10	103	92	91	77	87	90	10.3	5
	Overall							84	11.4	10
Oilseed rape	Foramsulfuron	0.01	99	101	102	101	103	101	1.5	5
		0.10	105	100	102	103	102	102	1.9	5
	Overall							102	1.7	10
Cereals straw	Foramsulfuron	0.01	78	71	76	71	88	77	8.9	5
		0.10	71	76	70	75	72	73	3.3	5
	Overall							72	7.0	10

RSD = relative standard deviation

Stability of Analytes

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. 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.

Table 5.2.1.- 12: Stability of foramsulfuron 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	10.30	9.60	9.90	-2
		2013-09-10	9.56	9.55	9.94	
Sugar beet, leaf	0.1	2013-08-29	9.63	8.92	9.32	-23
		2013-09-09	7.23	7.06	7.09	
Lemon, fruit	0.1	2013-09-06	8.76	8.55	8.65	-77
		2013-09-09	2.07	1.90	1.94	
Oilseed Rape	0.1	2013-09-02	10.30	10.30	10.40	-69
		2013-09-09	3.32	3.25	3.18	
Cereals Straw	0.1	2013-09-04	7.80	7.96	7.33	11
		2013-09-09	8.65	8.74	8.18	

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

Conclusion

The method is suitable as an enforcement method for the determination of foramsulfuron in/on samples from plant origin at a LOQ of 0.01 mg/kg. Validation of this method was successfully carried out in wheat (straw) as well as in sugar beet (body, leaf), lemon and oilseed rape.

B.5.2.2. Description of analytical methods for the determination of residues in animal products

In the scope of the original submission in 2000, no analytical methods were provided for the monitoring of foramsulfuron (AE F130360) in animal products, as no animal metabolism data was required at the time of the first EU dossier submission. As no MRLs have been set in foodstuff of animal origin, no analytical methods were required for the determination of residues in foodstuff of animal origin.

An enforcement method (EM F07/00-0; KCA 4.2/22 and 23) was afterwards developed in hen egg, bovine milk and bovine tissues (liver, fat, muscle and kidney) but this included only a validation for the liver (KCA 4.2/24).

Analytical method 01208/M001 was validated for all matrices and can be used as an enforcement method.

After the Annex I inclusion of foramsulfuron, the RMS Italy evaluated a HPLC-MS/MS method (KCA 4.2/25) which was validated for the determination of foramsulfuron with an LOQ of 0.01 mg/kg in milk, meat, fat, liver, kidney and eggs. An ILV is also available (KCA 4.2/26) and the method has been confirmed by the validation for two different mass transitions.

Hence it is concluded, that foramsulfuron can be enforced in food of animal origin with an LOQ of 0.01 mg/kg in milk, meat, fat, liver, kidney and eggs (EFSA Journal 2012;10(11):2962; [M-466418-01-1](#); KCA 6/01).

The mentioned methods are summarised below.

Table 5.2.1- 13: Analytical methods for the determination of residues in animal matrices

Method n°	Matrix	Analytes	LOQ (mg/kg)	Technique	Doc. No.	Ref.
Animal monitoring methods						
EM F07/00-0 monitoring method	Egg Milk Muscle Liver Kidney Fat	AE F130360	0.01	LC-MS/MS	M-199702-02-1 M-200439-01-1	KCA 4.2./22 and 23
EM F07/00-0 ILV	Liver	AE F130360	0.01	LC-MS/MS	M-240268-01-1	KCA 4.2./24
01208/M001 monitoring method	Egg Milk Muscle Liver Kidney Fat	AE F130360	0.01	LC-MS/MS	M-389788-03-1	KCA 4.2./25
01208/M001 ILV	Egg Milk Muscle Liver Kidney Fat	AE F130360	0.01	LC-MS/MS	M-398300-01-1	KCA 4.2./26

Report:	KCA 4.2 /22;Wrede, A.;2000;M-199702-02-1; Amended: 2001-02-07
Title:	Enforcement Method for Animal tissue, Milk and Egg by LC-MS/MS Amidosulfuron (AE F075032) Metsulfuron-methyl (AE F075736) Iodosulfuron-methyl-sodium (AE F115008) Mesosulfuron-methyl (AE F130060) Foramsulfuron (AE F130360)
Report No:	EMF07/00-0
Document No:	M-199702-02-1
Guidelines:	Deviation not specified
GLP/GEP:	no

Report:	KCA 4.2 /23;Wrede, A.;2001;M-200439-01-1
Title:	Validation of the Enforcement Method EM F07/00-0 for Animal tissue, Milk and Egg by LC-MS/MS-Amidosulfuron (AE F075032)-Metsulfuron-methyl (AE F075736)-Iodosulfuron-methyl-sodium (AE F115008)-Mesosulfuron-methyl (AE F130060)-Foramsulfuron (AE F130360)
Report No:	C011226
Document No:	M-200439-01-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

Principle of the method

Samples were extracted from all substrates with acetone. After sequential clean-up steps, involving liquid/liquid extraction with hexane and aqueous triethylamine 0.02 mol/L and liquid/liquid extraction with ethyl acetate / diluted formic acid, foramsulfuron was determined by LC-MS/MS.

The following transition ion was used for quantification of foramsulfuron $m/z = 453.2 \rightarrow m/z = 182.0$ ionisation mode: positive electrospray (MRM mode).

Validation of the method

Samples were measured by two fortification levels 0.01 mg/kg and 0.1 mg/kg in quintuplet for each matrix.

Linearity	Detector response was quadratic for matrix standards or pure solvent standards ranging from 10 µg/L to 500 µg/L (n=7 ; in duplicate). The correlation coefficients were higher than 0.99.
Precision/Repeatability	Relative standard deviations were below 20% for all analytes and sample materials.
Accuracy/Recovery	Mean recoveries for each fortification level and the overall mean recovery were within the range 70 - 110% for all sample materials
Specificity/Interferences	No interference was observed. All control samples showed no residues higher than 30% of the LOQ. Matrix effects were noticed in all matrices. It is therefore recommended to use matrix-matched standards.
LOQ	The limit of quantification for foramsulfuron in hen egg, bovine milk, and bovine tissues (muscle, fat, liver and kidney) was established at 0.01 mg/kg.

Table 5.2.2-1: Recovery efficiency of analysis of foramsulfuron in hen egg, bovine milk and bovine tissues (muscle, liver, fat and kidney).

Sample material	Analyte	Fortification level [mg/kg]	Recoveries [%]						RSD [%]	n
			Individual values					Mean		
Hen egg	Foramsulfuron	0.01	67	76	78	78	80	76	7	5
		0.10	61	74	78	65	83	72	13	5
Bovine milk	Foramsulfuron	0.01	98	102	87	90	84	92	8	5
		0.10	105	106	102	103	108	105	2	5
Bovine muscle	Foramsulfuron	0.01	93	100	117	137	82	106	20	5
		0.10	77	81	99	90	88	87	10	5
Bovine fat	Foramsulfuron	0.01	128	103	93	94	117	107	14	5
		0.10	102	106	113	99	99	104	6	5
Bovine liver	Foramsulfuron	0.01	96	96	95	87	96	94	4	5
		0.10	89	91	87	96	97	92	5	5
Bovine kidney	Foramsulfuron	0.01	98	100	90	96	84	94	7	5
		0.10	86	92	89	87	90	89	3	5

RSD: relative standard deviation

(a) Calculations were done with matrix-matched standards.

Conclusion:

The proposed analytical method is fully validated for the determination of foramsulfuron in foodstuff of animal origin with LOQ = 0.01 mg/kg in liver, kidney, meat, fat, eggs and milk. Nevertheless, this method is not highly specific (only a mass transition was validated).

Independent Laboratory Validation

An independent laboratory validation (ILV) of the analytical method EM F07/00-0 ([M-199702-02-1](#); KCA 4.2/22 and [M-200439-01-1](#); KCA 4.2/23) was carried out in bovine liver.

Report:	KCA 4.2/24;Randolph, R.:2004;M-240268-01-1
Title:	Independent Laboratory Validation for Aventis CropScience GmbH Analytical method No. EM/F07/00-0, Enforcement method for animal Tissue, Milk and Egg by LC-MS/MS
Report No:	B004802
Document No(s):	Report includes Trial Nos.: RAMMY004 M-240268-01-1
Guidelines:	USEPA (=EPA): 860.1340;Deviation not specified
GLP/GEP:	yes

Principle of the ILV

The analytical method EM F07/00-0 was followed. The following transition ion was used for quantification of foramsulfuron: $m/z = 453.0 \rightarrow m/z = 182.0$

Validation of the method

Samples were measured by three fortification levels 0.01 mg/kg, 0.02 mg/kg and 0.1 mg/kg in quintuplet.

Linearity	Detector response was quadratic for matrix standards or pure solvent standards ranging from 10 µg/L to 500 µg/L. The correlation coefficients were higher than 0.99.
Precision/Repeatability	Relative standard deviations were below 20% for all analytes and sample materials.
Accuracy/Recovery	Mean recoveries for each fortification level and the overall mean recovery were within the range 70 - 110% for all sample materials
Specificity/Interferences	No interference was observed. All control samples showed no residues higher than 30% of the LOQ. Matrix effects were noticed in all matrices. It is therefore recommended to use matrix-matched standards.
LOQ	The limit of quantification for foramsulfuron in bovine liver was established at 0.01 mg/kg.

Table 5.2.2.- 2: Recovery efficiency of analysis of foramsulfuron in bovine liver

Sample material	Analyte	Fortification Level [mg/kg]	Recoveries* [%]					RSD [%]	n	
			Individual values				Mean			
Bovine Liver	Foramsulfuron	0.01	82	85	92	86	92	87	5.2	5
		0.02	80	86	90	81	91	85	5.9	5
		0.10	87	83	92	87	93	89	4.4	5

RSD: relative standard deviation

*Values were rounded

Conclusion:

The proposed ILV is fully validated for the determination of foramsulfuron in liver with LOQ = 0.01 mg/kg. According to the Guidance document SANCO 825/00, ILV should be performed on at least two matrices, therefore complementary data is necessary.

The modification M001 of the Residue Analytical Method 01208 (EM F07/00-0) of the determination of foramsulfuron by LC-MS/MS was validated for animal tissues (meat, fat, liver, kidney) egg and milk. The objective of this study was to increase the selectivity of the method by adding a second MRM- transition and to adapt the method to the requirements of SOP 1540.

Report:	KCA 4.2 /25;Schmeer, K.; Philipowski, C.;2010;M-389788-03-1; Amended: 2011-01-03
Title:	Modification M001 of the residue analytical method 01208 for the determination of amidosulfuron (AE F075032), metsulfuron-methyl (AE F075736), iodosulfuron-methyl-sodium (AE F115008), mesosulfuron-methyl (AE F130060), foramsulfuron (AE F130360) in animal tissues (meat, fat, liver, kidney), egg, and milk by HPLC-MS/MS
Report No:	01208/M001
Document No:	M-389788-03-1
Guidelines:	91/414/EEC, 96/68/EC 91/414, SANCO/3029/99 SANCO/825/00 rev. 7, OPPTS 860.5340;not specified
GLP/GEP:	yes

Principle of the method

The employed extraction procedure was taken from method 01208 (EM F07/00-0), and facilitated by reducing clean-up steps. The homogenised samples were extracted with acetone, centrifuged, and the supernatant brought to volume. An aliquot was evaporated to dryness, taken up with acetonitrile/water (with 0.02 mM tri-ethylamine) 2:8 and analysed by LC-MS/MS.

The following MRM transitions were used for quantification and confirmation of foramsulfuron $m/z = 453.1 \rightarrow m/z = 182.2$ (quantification) and $m/z = 453.1 \rightarrow m/z = 139.2$ (confirmation).

Validation of the method

The determination of the residues was done with matrix-matched standards.

Samples were measured by two fortification levels 0.01 mg/kg and 0.1 mg/kg in quintuplet for all matrices.

Linearity	Detector response was linear for matrix standards ranging from 2 µg/L to 50 µg/L. The correlation coefficients were higher than 0.99.
Precision/Repeatability	Relative standard deviations were below 20% for all analytes and sample materials.
Accuracy/Recovery	Mean recoveries for each fortification level and the overall mean recovery were within the range 70 - 110% for all sample materials
Specificity/Interferences	No residues/interferences were found in control samples used for recovery experiments.
LOQ	The limit of quantification for foramsulfuron in all matrices was established at 0.01 mg/kg.

Table 5.2.2.-3: Recovery efficiency of analysis of foramsulfuron in animal matrices

Analyte	Sample material	Fortification level [mg/kg]	Recoveries* [%]						RSD [%]	n
			Individual values					Mean		
Foramsulfuron $m/z = 453.1 \rightarrow 182.2$ (quantification)	Egg	0.01	73	77	75	78	74	75	2.8	5
		0.10	75	72	79	88	79	79	7.7	5
	Overall							77	5.9	10
	Fat	0.01	86	88	88	85	80	85	3.8	5
		0.10	89	83	85	79	76	82	6.2	5
	Overall							84	5.2	10
	Kidney	0.01	82	85	83	81	84	83	1.9	5
		0.10	88	86	87	90	87	88	1.7	5
	Overall							85	3.3	10
	Liver	0.01	84	90	90	94	89	89	4.0	5
		0.10	90	92	93	89	92	91	1.8	5
	Overall							90	3.1	10
	Meat	0.01	90	83	88	90	86	87	3.4	5
		0.10	83	87	82	82	91	85	4.6	5
	Overall							86	4.1	10
Foramsulfuron $m/z = 453.1 \rightarrow 139.2$ (confirmation)	Egg	0.01	73	80	81	81	80	79	4.3	5
		0.10	75	71	82	83	78	78	6.4	5
	Overall							78	5.2	10
	Fat	0.01	88	84	82	87	77	84	5.3	5
		0.10	85	77	87	84	77	82	5.7	5
	Overall							83	5.3	10
	Kidney	0.01	86	98	85	85	87	88	6.3	5
		0.10	86	86	87	93	87	88	3.4	5
	Overall							88	4.8	10
	Liver	0.01	87	88	91	88	90	89	1.9	5

		0.10	88	91	94	90	92	91	2.5	5
	Overall							90	2.4	10
	Meat	0.01	86	81	78	98	88	86	8.9	5
		0.10	81	84	84	81	86	83	2.6	5
	Overall							85	6.6	10
	Milk	0.01	86	97	89	82	85	88	6.5	5
		0.10	80	88	83	86	89	85	4.3	5
	Overall							87	5.5	10

Confirmatory method

Two MRM transitions were monitored for each analyte. Further on, the peak-area ratios of both MRMs (being a compound specific constant under given mass spectrometric conditions) were calculated. Therefore, the HPLC-MS/MS method is highly specific and an additional confirmatory method is not necessary.

Conclusion

Method 01208/M001 is suitable as an enforcement method for the determination of foramsulfuron. Validation of this method was successfully carried out in egg, milk and bovine tissues (muscle, fat, liver, and kidney).

Independent Laboratory Validation

An independent laboratory validation of the method was performed (Moore, S.; 2010; [M-398300-01- 1](#); KCA 4.2/26), and is summarised below.

Report:	KCA 4.2 /26;Moore, S.;2010;M-398300-01-1
Title:	Independent laboratory validation of an analytical method 01208/M001 for the determination of amidosulfuron (AE F075032), metsulfuron-methyl (AE F075736), iodosulfuron-methyl-sodium (AE F115008), mesosulfuron-methyl (AE F130060), foramsulfuron (AE F130360) in animal tissues (meat, fat, liver, kidney), egg, and milk by HPLC-MS/MS
Report No:	RAMML014
Document No:	M-398300-01-1
Guidelines:	Residue Chemistry Test Guidelines: OPPTS 860.1340 Residue Analytical Method European Commission Guidelines: SANCO/825/00 rev. 7 Guidance Document on Residue Analytical Methods Residue Chemistry Guidelines: PMRA Regulatory Directive Dir98-02 DACO 7.2.3 Inter-laboratory Analytical Methodology Validation;not specified
GLP/GEP:	yes

Principle of the ILV

Analytical method 01208/M001 was followed. The determination of the residues was done with matrix-matched standards. The following MRM transitions were used for quantification and confirmation of foramsulfuron $m/z = 453.03 \rightarrow m/z = 182.04$ (quantification) and $m/z = 453.03 \rightarrow m/z = 139.03$ (confirmation).

Validation of the method

The determination of the residues was done with matrix-matched standards. Samples were measured by two fortification levels 0.01 mg/kg and 0.1 mg/kg in quintuplet for all matrices.

Linearity	Detector response was linear for matrix standards ranging from 0.6 µg/L to 20 µg/L. The correlation coefficients were higher than 0.99.
Precision/Repeatability	Relative standard deviations were below 20% for all analytes and sample materials. The essay with fat had to be repeated at both concentration levels. The second attempt was successful.
Accuracy/Recovery	Mean recoveries for each fortification level and the overall mean recovery were within the range 70 - 110% for all sample materials. The essay with fat had to be repeated at both concentration levels. The second attempt was successful.
Specificity/Interferences	No residues/interferences were found in control samples used for recovery experiments. Therefore, the recoveries were not corrected for interferences.
LOQ	The limit of quantification for foramsulfuron in all matrices was established at 0.01 mg/kg.

Table 5.2.2.-4: Recovery efficiency of analysis of foramsulfuron in animal matrix (matrix matched standards).

Analyte	Sample material	Fortification level [mg/kg]	Recoveries* [%]						RSD [%]	n
			Individual values					Mean		
Foramsulfuron m/z = 453.03 → 182.04 (quantification)	Egg	0.01	85	85	81	83	80	83	2.8	5
		0.10	85	85	84	81	79	83	3.2	5
	Overall							83	2.8	10
	Fat	0.01	68	79	70	69	74	72	6.3	5
		0.10	69	66	74	68	78	71	6.9	5
	Overall							72	6.3	10
	Kidney	0.01	81	85	90	88	89	87	4.2	5
		0.10	88	88	88	94	85	89	3.7	5
	Overall							88	3.9	10
	Liver	0.01	88	79	84	83	84	84	3.8	5
		0.10	84	88	104	99	104	96	9.7	5
	Overall							90	10	10
	Muscle	0.01	84	86	88	96	88	88	5.2	5
		0.10	88	92	89	90	93	90	2.3	5
	Overall							89	3.9	10
	Milk	0.01	87	86	86	84	90	87	2.5	5
		0.10	85	87	87	90	85	87	2.4	5
	Overall							87	2.3	10
Foramsulfuron m/z = 453.03 → 139.03 (confirmation)	Egg	0.01	85	80	75	85	77	80	5.7	5
		0.10	86	83	81	78	80	82	3.7	5
	Overall							81	4.6	10
	Fat	0.01	69	76	71	73	75	73	3.9	5
		0.10	69	67	75	67	74	70	5.5	5
	Overall							72	4.8	10
	Kidney	0.01	87	83	88	89	92	88	3.7	5
		0.10	89	89	88	95	85	89	4.1	5
	Overall							89	3.8	10
	Liver	0.01	104	81	77	88	100	90	13	5
		0.10	82	93	102	101	109	97	11	5
	Overall							94	12	10
	Muscle	0.01	87	86	88	85	80	85	3.7	5
		0.10	85	92	91	92	88	90	3.4	5
	Overall							87	4.3	10
	Milk	0.01	88	82	83	82	83	84	3.0	5
		0.10	87	90	86	90	86	88	2.3	5
	Overall							86	3.6	10

Confirmatory method

Two MRM transitions were monitored for each analyte. Further on, the peak-area ratios of both MRMs (being a compound specific constant under given mass spectrometric conditions) were calculated. Therefore, this HPLC-MS/MS method is highly specific and an additional confirmatory method is not necessary.

Conclusion

Method 01208/M001 was successfully validated in egg, milk and bovine tissues (muscle, fat, liver, and kidney).

B.5.2.3. Analytical methods for monitoring purposes in soil

An enforcement method (KCA 4.2/08) and its ILV (KCA 4.2/09) were submitted and evaluated within the process of evaluation for Annex I inclusion, as published in the corresponding Monograph of RMS, Germany, (April 01, 2001) and its amendment. These methods will not be described in detail under the scope of the renewal of approval of foramsulfuron. A brief summary of the EU conclusions are provided below.

Report:	KCA 4.2/08;Wrede, A.;2000;M-193807-01-1
Title:	Enforcement Method for Soil by LC-MS/MS Metsulfuron-methyl (AE F075736) Iodosulfuron-methyl-sodium (AE F115008)
Report No:	EM F13/99-0 (C006394)
Document No(s):	M-193807-01-1
Guidelines:	Deviation not specified
GLP/GEP:	no

Report:	KCA 4.2/09;Wrede, A.;2000;M-194836-01-1
Title:	Validation of the enforcement method EM F13/99-0 in soil by LC-MS/MS - Code: AE F130360
Report No:	CR 99/035 (C006905)
Document No(s):	M-194836-01-1
Guidelines:	Deviation not specified
GLP/GEP:	no

Method EM F13/99-0 was developed as a typical soil enforcement method for the determination of residues of sulfonyl urea compounds (Wrede, A.; 2000; [M-193807-01-1](#), KCA 4.2 /08). The method was based on HPLC-MS/MS and had a limit of quantification (LOQ) of 0.0005 mg/kg. During the EU evaluation this method was considered appropriate for the determination of foramsulfuron residues.

Table 5.2.3.- 1: EU conclusions: Analytical method for residues of foramsulfuron (AE F130360) in soil

Method	Matrix	Analytes	LOQ (mg/kg)	Technique	Doc. No.	Reference
Enforcement method						
EM F13/99-0	Soil	Foramsulfuron	0.0005	HPLC-MS/MS	M-193807-01-1	DAR (01 April 2001)

Method EM F13/99-0 was validated for the determination of residues of foramsulfuron in soil (Wrede, A.; 2000; [M-194836-01-1](#); KCA 4.2 /09). The method was based on HPLC-MS/MS and had a limit of quantification (LOQ) of 0.0005 mg/kg.

Table 5.2.3.- 2: EU conclusions: Validated analytical method for residues of foramsulfuron (AE F130360) in soil

Method	Matrix	Analytes	LOQ (mg/kg)	Technique	Doc. No.	Reference
Enforcement method						
EM F13/99-0	soil	Foramsulfuron	0.0005	HPLC-MS/MS	M-194836-01-1	DAR (01 April 2001)

During the first approval it was concluded that the method was suitable for the determination of residues of foramsulfuron in soil for monitoring purposes.

The enforcement method for the determination of residues of foramsulfuron in soil has been updated following the latest data requirements. For example, the new study (KCA 4.2/27) has a lower LOQ (0.1 µg/kg) and has two MS/MS transitions.

Report:	KCA 4.2 /27;Freitag, T.;2008;M-310074-03-1; Amended: 2013-08-08
Title:	Amendment no. 0001 to report no.: MR-08/138 - Analytical Method 01115 for the determination of residues of amidosulfuron,iodosulfuron-methyl-sodium, metsulfuron-methyl, mesosulfuron-methyl and foramsulfuron in soil by HPLC-MS/MS
Report No:	M-310074-03-1
Document No:	M-310074-03-1
Guidelines:	91/414/EEC,96/68/EC 91/414,SANCO/3029/99 SANCO/825/00 rev. 7, OPPTS 860.1340: Residue Analytical Method;not specified
GLP/GEP:	yes

Principle of the Method

Soil samples of 20 g were extracted in a microwave extractor with 40 mL of a mixture of acetonitrile/water (2:8, v/v). Then a subsample was centrifuged to remove fine particles of soil. Identification and quantification of the test items was done by high performance liquid chromatography using MS/MS detection in the Multiple Reaction Monitoring mode. The method was validated using a silt soil (Höfchen) and a sandy loam soil (Laacher Hof).

Two MRM transitions were used for quantification and confirmation of foramsulfuron $m/z = 453.1 \rightarrow 182.1$ (quantification) and $m/z = 451.0 \rightarrow 268.2$ (confirmation).

Table 5.2.3-3 : Soil Characteristics.

	Soil Hofchen	Soil Laacher Hof
Description	Plot 4011 ; 0-30 cm soil layer	Plot 712/718 ; 0-30 cm soil layer
pH (in CaCl ₂ solution)	6.7	6.8
pH (in H ₂ O)	7.4	7.4
Organic Carbon (%)	0.92	1.2
Organic Matter (%)*	1.58	2.06
Cation Exchange Capacity (meq/100 g dry soil)	12.4	9.8
max. Water Holding Capacity (g/100g dry soil)	39.5	37.9
Textural Description according to USDA (Fraction%)	Fraction (%)	Fraction (%)
Clay (<0.002 mm)	19.4	12.0
Silt (0.002-0.050 mm)	76.3	18.3
Sand (0.050-2.000 mm)	4.3	69.7
Soil type	Silt loam	Sandy loam

* : Organic matter = Organic carbon x 1.72

Validation of the method

The determination of the residues was done with matrix-matched standards.

Samples were measured by two fortification levels 0.1 µg/kg and 1 µg/kg in quintuplet for all matrices.

Linearity	Detector response was linear for matrix standards ranging from 0.1 µg/L to 5 µg/L. The correlation coefficients were higher than 0.99.
Precision/Repeatability	During method validation, the relative standard deviations at the individual fortification levels and the overall relative standard deviations ranged between 3.5 and 17.7%
Accuracy/Recovery	The mean recoveries of foramsulfuron for the individual types of soil and/or fortification levels ranged from 82 to 114%
Specificity/Interferences	LC/MS/MS using two characteristic MS/MS transitions for detection and quantification of foramsulfuron ensures a high level of specificity. In the control samples, residues were always < 30% LOQ.
LOQ and LOD	The limit of quantification (LOQ) for foramsulfuron is 0.1 µg/kg soil while the limit of detection (LOD) is 0.03 µg/kg.

Table 5.2.3.-4: Recoveries of foramsulfuron in soil

Compound	Soil	Fortification Level [µg/kg]	Recovery [%]						RSD [%]	n
			Individual					Mean		
Foramsulfuron m/z = 453.1 → 182.1	Höfchen	0.1	117	118	115	106	112	114	4.1	5
		1	88	86	80	81	76	82	6.0	5
		Overall						98	11.4	10
	Laacher Hof	0.1	94	90	89	89	81	89	5.6	5
		1	110	114	110	103	101	108	4.9	5
		Overall						98	17.7	10
Foramsulfuron m/z = 451.0 → 268.2	Höfchen	0.1	106	100	104	102	100	102	2.5	5
		1	102	97	98	109	100	101	4.6	5
		Overall						102	3.5	10
	Laacher Hof	0.1	93	91	80	99	101	93	8.9	5
		1	96	93	92	100	104	97	5.2	5
		Overall						95	7.1	10

RSD: relative standard deviation

Stability of the Analytical Extracts

In order to investigate the storage stability of residues in soil extracts, the extracts were stored refrigerated (at ≤ 6°C) for 11 days after the validation and measured a second time for the quantifier mass transition. The mean recoveries amounted to 91% for the silt loam soil, and 106% for the sandy loam soil for the quantifier mass transition of foramsulfuron. Therefore, the sample extracts were shown to be stable under refrigerated conditions for at least 11 days.

Confirmatory method

LC-MS/MS using two characteristic MS/MS transitions for detection and quantification ensures specificity of the method.

Conclusion

The LC-MS/MS method 01115 has been validated (LOQ = 0.1 µg/kg) for use as an enforcement method of foramsulfuron in soil. The HPLC-MS/MS method is highly specific (2 MRM transitions measured for each analyte and each matrix tested) and therefore an additional confirmatory method is not required.

B.5.2.4. Analytical methods for monitoring purposes in surface and drinking water

An enforcement method (KCA 4.2/10) and its ILV (KCA 4.2/11; 4.2/12) were submitted and evaluated within the process of evaluation for Annex I inclusion, as published in the corresponding Monograph of RMS, Germany, (April 01, 2001) and its amendments. These methods will not be described in detail under the scope of the renewal of approval of foramsulfuron. A brief summary of the EU conclusions are provided below.

Report:	KCA 4.2/10;Wrede, A.;2000;M-195026-01-1
Title:	Enforcement method for surface and drinking water by HPLC-UV Code: AE F130360
Report No:	EM F07/99-0 (C006998)
Document No(s):	M-195026-01-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

Report:	KCA 4.2/11;Wrede, A.;2000;M-195060-01-1
Title:	Validation of the enforcement method EM F07/99-0 for surface and drinking water by HPLC-UV - Code: AE F130360
Report No:	CR 99/020 (C007019)
Document No(s):	M-195060-01-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

Report:	KCA 4.2/12;Wrede, A.; Neuss, B.;2001;M-200406-01-1
Title:	Validation of the enforcement method EM F04/00-0 for surface and drinking water by LC-MS/MS-Amidosulfuron (AE 075032) -Metsulfuron-methyl (AE F075736)-Iodosulfuron-methyl-sodium (AE F115008)-Mesosulfuron-methyl (AE F130060)-Foramsulfuron (AE F130360)
Report No:	CR 00/027 (C011207)
Document No(s):	M-200406-01-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

Method EM F07/99-0 was provided as enforcement method for the determination of residues of foramsulfuron (AE F130360) in surface and drinking water (Wrede, A.; 2000; [M-195026-01-1](#); KCA 4.2 /10). The method was based on HPLC-UV to result in a limit of quantification (LOQ) of 0.1µg/L. The method was considered as appropriate. A validation of method EM F07/99-0 was provided (Wrede, A.; 2000; [M-195060-01-1](#); KCA 4.2 /11).

Table 5.2.4.-1: EU conclusions: Analytical method for foramsulfuron (AE F130360) in surface and drinking water

Method	Matrix	Analytes	LOQ (µg/L)	Technique	Doc. No.	Reference
Enforcement method						
EM F07/99-0	surface and drinking water	foramsulfuron	0.1	HPLC-UV	M-195026-01-1	DAR (01 April 2001)

During the Annex I listing process the monitoring method in surface and drinking water was updated. A new multi-residue method based on HPLC-MS/MS was submitted (method EM F04/00-0) (Wrede, A; 2001; M-200406-01-1; KCA 4.2 /12). In addition to foramsulfuron, the multi-residue analytical method (EM F04/00-0) covered the analytical targets amidosulfuron (AE F075032), metsulfuron- methyl (AE F075736), iodosulfuron-methyl-sodium (AE F115008) and mesosulfuron-methyl (AE F130060). The limit of quantification (LOQ) for each of the analytical targets was 0.05µg/L. The method was considered as appropriate.

Table 5.2.4.-2: EU conclusions: Validated analytical method for foramsulfuron (AE F130360) in surface and drinking water

Method	Matrix	Analytes	LOQ (µg/L)	Technique	Doc. No.	Reference
Enforcement method						
EM F04/00-0	surface and drinking water	foramsulfuron	0.05	HPLC-MS/MS	M-200406-01-1	Addendum to DAR (23 April 2002)

During the first approval it was concluded that the method was suitable for the determination of residues of foramsulfuron in surface and drinking water for monitoring purposes.

Since the EU approval of foramsulfuron (2002) the enforcement method for surface and drinking water has been updated in 2007 according to the latest data requirements (Krebber et al., 2007, M-291466-01-1; KCA 4.2/28) and then updated in 2013 for inter-laboratory validation (Krebber & Braune; [M-466732-01-1](#); KCA 4.2/29). The inter-laboratory validation report is provided (KCA 4.2/30; [M-468318-01-1](#)).

Report:	KCA 4.2 /28;Krebber, R.; Braune, M.;2007;M-291466-01-1
Title:	Analytical method 01058 for the determination of amidosulfuron, foramsulfuron, iodosulfuron-methyl-sodium, mesosulfuron-methyl and the metabolite metsulfuron-methyl (AE F075736) in drinking and surface water by HPLC-MS/MS
Report No:	01058
Document No:	M-291466-01-1
Guidelines:	SANCO/825/00 rev. 7; BBA Guideline: Residue Analytical Methods for Post-Registration Control Purposes of July 21, 1998; EU 96/46/EEC amending Council Directive 91/414/EEC;not specified
GLP/GEP:	yes

Principle of the Method

An aliquot of the sample solution was injected into the HPLC and subjected to reversed phase chromatography coupled with tandem mass spectrometry (MS/MS) with electrospray ionisation. The MS/MS instrument was operated in Multiple Reaction Monitoring mode (MRM). The following MRM transitions were used for quantification and confirmation of foramsulfuron $m/z = 453 \rightarrow 182$ (quantification) and $m/z = 453 \rightarrow 255$ (confirmation).

Table 5.2.4-3 : Characteristics of the Surface water from River Rhine, Sampled on 2004-09-10 in Leverkusen-Hitdorf (Germany)

Parameter	Value
Total organic carbon (TOC)	4 mg/L
Dissolved organic carbon (DOC)	< 2 mg/L
Conductivity	507 µS/cm
pH	7.6
Water hardness	10.1 °dH
Dry residue after filtration	54 mg/L

Validation of the method

The determination of the residues was done with matrix-matched standards.

Samples were measured by two fortification levels 0.05 µg/L and 0.5 µg/L in decuple for all matrices.

Linearity	Detector response was linear for matrix standards ranging from 0.04 µg/L to 10 µg/L. The correlation coefficients were higher than 0.99.
Precision/Repeatability	The relative standard deviations at the individual fortification levels and the overall relative standard deviations ranged between 2.7% and 5.1% for both MRM transitions
Accuracy/Recovery	Mean recoveries for each fortification level and for overall mean recoveries ranged from 93 to 100% for both MRM transitions.
Specificity/Interferences	Apparent concentrations in control samples were below $0.3 \times \text{LOQ}$. Two MRM transitions were monitored for foramsulfuron.
LOQ and LOD	The limit of quantification (LOQ) for foramsulfuron is 0.05 µg/L associated with a limit of detection (LOD) of 0.02 µg/L.

Table 5.2.4-4: Recoveries of foramsulfuron in surface water

Matrix	Analyte	Fortification Level [µg/L]	Recovery [%]						RSD [%]	n
			Individual					Mean		
Surface Water	Foramsulfuron m/z = 453 → 182 (quantification)	0.05	96 97	96 94	90 90	91 93	93 93	93	2.7	10
		0.5	96 93	103 103	92 95	103 99	93 96	97	4.4	10
		Overall						95	4.2	20
	Foramsulfuron m/z = 453 → 255 (confirmation)	0.05	108 102	98 98	93 107	95 97	105 100	100	5.1	10
		0.5	96 95	99 102	94 93	95 96	92 95	96	3.0	10
		Overall						98	4.7	20

Confirmatory method

LC-MS/MS using two characteristic MS/MS transitions for detection and quantification ensures the specificity of the method.

Conclusion

Analytical method 01058 is regarded to be straightforward since the water samples are directly measured by LC-MS/MS without further concentration or purification steps. The quantification by two distinct MS/MS transitions ensured the specificity of the method successfully validated at a limit of quantification (LOQ) of 0.05 µg/L. A validation for drinking water is not necessary since the limit of quantification for surface water of 0.05 µg/L is below the drinking water limit of 0.1 µg/L. Since drinking water is the less complex analytical matrix, the method is also applicable for drinking water.

The method has been demonstrated to be suitable as enforcement method for monitoring of foramsulfuron residues in drinking and surface water. However, no ILV has been given for the method 01058.

The analytical method 01387 presented below describes the determination of multiple active substances and selected metabolites in drinking and surface water by HPLC-MS/MS with two MRM transitions.

Report:	KCA 4.2 /29;Krebber, R.; Braune, M.;2013;M-466732-01-1
Title:	Analytical method 01387 for the determination of various pesticides in drinking and surface water by HPLC-MS/MS
Report No:	MR-13/085
Document No:	M-466732-01-1

Guidelines:	<p>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</p> <p>EC Guidance Document on Residue Analytical Methods, SANCO/825/00 rev. 8.1 of November 16, 2010</p> <p>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 rev. 4, July 11, 2000; Deviation not specified</p>
GLP/GEP:	yes

Principle of the Method

Foramsulfuron was determined by direct injection into the HPLC-MS/MS instrument using positive ion mode without further clean-up. An aliquot of the sample solution was injected into HPLC and subjected to reversed phase chromatography coupled with tandem mass spectrometry (MS/MS) with electrospray ionisation. The MS/MS instrument was operated in the Multiple Reaction Monitoring mode (MRM). The following MRM transitions were used for quantification and confirmation of foramsulfuron $m/z = 453 \rightarrow 182$ (quantification) and $m/z = 453 \rightarrow 139$ (confirmation)

Table 5.2.4-5 : Characteristics of the Surface Water River Rhine, Sampled on 2009-07-29 in Leverkusen-Hitdorf (Germany)

Parameter	Value
Total organic carbon (TOC)	2 mg/L
Dissolved organic carbon (DOC)	2 mg/L
Conductivity	448 $\mu\text{S}/\text{cm}$
pH	7.3
Water hardness	9.9dH
Filtrable solids	14 mg/L
Dry residue after filtration	290 mg/L

Validation of the method

Concentrations were quantified using external matrix-matched standard solutions.

Samples were measured by two fortification levels 0.05 $\mu\text{g}/\text{L}$ and 0.5 $\mu\text{g}/\text{L}$ in decuple for all matrices.

Linearity	The correlation between the injected amount of substance and the detector response was linear (1/x weighted) for standard solutions in surface water/acetonitrile (800:200, v/v) ranging from 0.012 $\mu\text{g}/\text{L}$ to 4.0 $\mu\text{g}/\text{L}$. The correlation coefficient was ≥ 0.9991 for both MRM transitions
Precision/Repeatability	During method validation, the relative standard deviations at the individual fortification levels ranged between 1.0% and 3.0% for peak area and between 0.2 and 0.3 for retention time for both MRM transitions.
Specificity/Interferences	Apparent concentrations in control samples were below $0.3 \times \text{LOQ}$. Two MRM transitions were monitored for foramsulfuron.
LOQ	The limit of quantification (LOQ) for foramsulfuron is 0.05 $\mu\text{g}/\text{L}$ in surface water.

Table 5.2.4.-6: Method validation for foramsulfuron for the quantification ion (m/z = 453→182)

Matrix	Fortification Level [µg/L]	Peak Area (Area Counts)				RSD [%]	Retention Time		
		Single Values			Mean		Mean [min]	RSD [%]	
Surface water	0.05	30519	30013	29125	29614	29622	1.9	2.10	0.2
		29817	29330	28915	29644				
		28947	30299	-	-				
	0.5	278237	279480	274841	280804	277828	1.0	2.10	0.2
		277750	275951	282634	273963				
		275753	278871	-	-				

RSD: Relative Standard Deviation

Table 5.2.4.-7: Method validation for foramsulfuron for the quantification ion (m/z = 453→139)

Matrix	Fortification Level [µg/L]	Peak Area (Area Counts)				RSD [%]	Retention Time		
		Single Values					Mean	Mean [min]	RSD [%]
Surface water	0.05	5703	5689	5615	5868	5767	2.9	2.11	0.3
		5889	5700	5452	5862				
		6048	5842						
	0.5	54857	58759	54522	57653	57292	3.0	2.10	0.2
		59859	58087	58781	56017				
		57536	56852						

RSD: Relative Standard Deviation

Stability of analytes

The analytes were stable in surface water when stored in a freezer at ≤ -18 °C for a period of 22 days.

Confirmatory method

HPLC-MS/MS using two characteristic MS/MS transitions for detection and quantification of foramsulfuron ensures a high level of specificity.

Conclusion

The method was validated using surface water from the river Rhine. A validation for drinking water was not necessary since the method limit of quantification in surface water is below the one for drinking water of 0.1 µg/L.

The method meets all guideline criteria to determine concentrations in drinking and surface water of foramsulfuron at 0.05 µg/L.

Following new data requirements the inter-laboratory validation of the monitoring method of foramsulfuron in surface and drinking water is presented.

Report:	KCA 4.2 /30;Stanislawski, T.;2013;M-470714-02-1
Title:	Independent laboratory validation of BCS analytical methods 01333 and 01387 for determination of various pesticides in surface water by Di-HPLC-MS/MS
Report No:	P3117 G
Document No:	M-470714-02-1

Guidelines:	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, rev. 4, 11/07/00 Guidance document on Pesticide Residue Analytical Methods, SANCO/825/00/rev. 8.1, European Commission, Directorate General Health and Consumer Protection 16/11/2010 Commission Regulation (EU) No 283/2013 (section 4.2) of 1 March 2013 setting out the data requirements for active substances, in accordance with Regulation (EC) No 1107/2009 of the European Parliament and of the Council concerning the placing of plant protection products on the market
GLP/GEP:	yes

Principle of the ILV

The independent validation follows the methods 01333 and 01387 that was performed for 23 analytical target compounds (i.e. active substances and metabolites) at an LOQ of 0.05 µg/L in surface water. The following MRM transitions were used for quantification and confirmation of foramsulfuron $m/z = 453 \rightarrow 182$ (quantification) and $m/z = 453 \rightarrow 139$ (confirmation)

Table 5.2.4-8 : Characteristics of the Surface water from river Danube (non-GLP), sampled on 2013-09-09 in Ulm (Germany)

Parameter	Value
Total organic carbon (TOC) (EN 1484 :1997)	1.90 mg/L
Dissolved organic carbon (DOC) (EN 1484 :1997)	1.7 mg/L
Conductivity (EN 27888 :1993)	584 µS/cm
pH (DIN 38 404-C 5)	8.16
Water hardness (calculated)	3.20 mmol/L (17.9°d)
Filterable solids (EN 872 Whatman GF 6)	290 mg/L

Validation of the method

Concentrations were quantified using external matrix-matched standard solutions.

Samples were measured by two fortification levels 0.05 µg/L and 0.5 µg/L in quintuplet.

Linearity	The correlation between the injected amount of substance and the detector response was linear (1/x weighted) for standard solutions in surface water/acetonitrile (800:200, v/v) ranging from 0.012 µg/L to 0.8 µg/L. The correlation coefficient was ≥ 0.9987 for both MRM transitions
Precision/Repeatability	The relative standard deviations for the peak areas for all quantifications were higher than 5.5% at 0.05 µg/L and higher as 4.7% at 0.5 µg/L. The relative standard deviation for the retention time was higher than 0.9% for all analytes and MRM transitions.
Specificity/Interferences	Apparent concentrations in control samples were below $0.3 \times \text{LOQ}$. Two MRM transitions were monitored for foramsulfuron.
LOQ	The limit of quantification (LOQ) was 0.05 µg/L for each analytes in surface water.

Table 5.2.4.-9: Method validation for foramsulfuron for the quantification ion (m/z = 453→182)

Matrix	Fortification Level [µg/L]	Peak Area (Area Counts)				RSD [%]	Retention Time		
		Single Values			Mean		Mean [min]	RSD [%]	
Surface water	0.05	274023	270548	273662	274000	273165	0.6	4.04	0.1
		272451	-	-	-				
	0.5	612310	616750	614956	612724	618261	2.2	4.04	0.1
		598518	-	-	-				

RSD - relative standard deviation

Table 5.2.4.-10: Method validation for foramsulfuron for the confirmatory ion (m/z = 453→139)

Matrix	Fortification Level [µg/L]	Peak Area (Area Counts)				RSD [%]	Retention Time		
		Single Values			Mean		Mean [min]	RSD [%]	
Surface water	0.05	62459	63615	62249	62817	63077	0.9	4.04	0.1
		64040	-	-	-				
	0.5	146774	142250	142421	142330	142840	0.7	4.03	0.1
		142653	-	-	-				

RSD - relative standard deviation

Stability of analytes

The stability of solutions of each analytical target compound upon storage in a freezer at $\leq -18^{\circ}\text{C}$ was determined (storage stability: at least 22 days at $\leq -18^{\circ}\text{C}$).

Confirmatory method

HPLC-MS/MS using two characteristic MS/MS transitions for detection and quantification of each analytical target compound ensures a high level of specificity with no separate confirmatory method needed since this is represented by the second transition.

Conclusion

The method was validated using surface water taken from the river Danube. The method is regarded to be straightforward since the water samples are directly measured by LC- MS/MS without further concentration or purification steps. The quantification by two distinct MS/MS transitions ensures the specificity of the method to be successfully validated at a limit of quantification (LOQ) of 0.05 µg/L. A validation for drinking water is not necessary since the method limit of quantification in surface water was below the one for drinking water of 0.1 µg/L. While being the less complex matrix than surface water, the objective of the study is to confirm that the method is also applicable for the determination of residues of each analytical target in drinking water.

The study demonstrates the validity of the enforcement method for monitoring of residues of the analytical target compounds in drinking and surface water when being performed by an independent analytical laboratory.

B.5.2.5. Analytical method for monitoring purposes in air

Two enforcement methods (KCA 4.2/13& KCA 4.2/14) were submitted and evaluated within the process of evaluation for Annex I inclusion, as published in the corresponding Monograph of RMS, Germany, (April 01, 2001) and its amendments. These methods will not be described in detail under the scope of the renewal of approval of foramsulfuron. A brief summary of the EU conclusions are provided below.

Report:	KCA 4.2 /13;Sutton, A. L.;Everitt, S. L.;1999;M-188704-01-1
Title:	Enforcement method with validation for air by GC-MSD active substance Code: AE F130360
Report No:	EM C08/99-0 (C004845)
Document No(s):	M-188704-01-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

Report:	KCA 4.1.1 /14;Golka, I.; Makowski, S.;2013;M-465143-01-1
Title:	Development and validation of an analytical method for the determination of AE F130360 in air
Report No:	IF-100/21281-00 (C010595/C022475)
Document No(s):	M-227811-03-1
Guidelines:	Deviation not specified
GLP/GEP:	yes

The first enforcement method for the determination of residues of foramsulfuron (AE F130360) in air (Sutton, A. L.;Everitt, S. L.;1999; [M-188704-01-1](#); KCA 4.2 /13) was based on GC-MSD to result in a limit of quantification (LOQ) of 12µg/m³. The method was considered as appropriate and the EU conclusions are presented in the table below.

Table 5.2.5.-1: EU conclusions: Analytical method for residues of foramsulfuron (AE F130360) in air

Method	Matrix	Analytes	LOQ (µg/m ³)	Technique	Doc. No.	Reference
Enforcement method						
EM C08/99-0	air	Foramsulfuron	12	GC-MSD	M-188704-01-1	DAR (01 April 2001)

During the course of the Annex I Listing process the monitoring method in air was updated by a new method based on HPLC-UV (Reichert; [M-227811-03-1](#); KCA 4.2 /14). The limit of quantification (LOQ) was 12µg/m³ and thus the same as for the method submitted earlier. The method was considered as appropriate.

Table 5.2.5.-2: EU conclusions: Updated analytical method for residues of foramsulfuron (AE F130360) in air

Method	Matrix	Analytes	LOQ (µg/m ³)	Technique	Doc. No.	Reference
Enforcement method						
IF-100/21281-00	air	Foramsulfuron	12	HPLC-UV	M-227811-03-1	Addendum to DAR (13 August 2002)

No new methods are submitted for the renewal of the approval of foramsulfuron.

B.5.2.6. Methods for the analysis in body fluids and tissues for active substances and relevant metabolites

No methods are required for the analysis in body fluids and tissues.

B.5.3. REFERENCES RELIED ON

Study KCA 4.1.1/02 was added as relevant for the evaluation of the first evaluation of foramsulfuron in technical active substance.

Studies KCA 4.2/05-07 were excluded as they were not reviewed during the first inclusion of the active substance and they are not relevant for this renewal.

Data Point	Author(s)	Year	Title Compagny Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner	Previous evaluation
B.5.1.1.1. KCA 4.1.1 /01	Cichy, M	1998	Determination of AE F130360 in the technical and pure active substance by HPLC (Analytical method) Code: AE F130360 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Report No.: C001812, Edition Number: M-183402-01-1 EPA MRID No.: 45109406 Date: 1998-10-21 GLP/GEP: no, unpublished	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.1.1.1. KCA 4.1.1/02	Cichy, M.; Kraus, S.	2000	Validation of the analytical methods to determine AE F 130360, ethyl acetate, toluene, cyclohexanone, tri-n-butylamine, formic acid, chloride and sulphate in technical Foramsulfuron Code: AE F130360 Foramsulfuron (proposed ISO) (Technical Grade Active Ingridient) Hoechst Schering AgroEvo GmbH, Frankfurt am Main, Germany Report No.: PA99/020 Ecition Number: M-134720-01-1 EPA MRID No.:C006839 Date: 2000-2-1 GLP/GEP: yes	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.1.1.1 KCA 4.1.1 /10	Selzer, J.; Eichelmann, C.	2013	Analytical method-Determination of foramsulfuron (AE F130360) in technical grade and pure active substance by high performance liquid chromatography (HPLC) Bayer CropScience, Report No.: AM038813FP1, Edition Number:M-464431-01-1 Date: 2013-09-13 GLP/GEP: no, unpublished	N	N	Not relevant	Bayer CropScience	Submitted for the purpose of renewal
B.5.1.1.1 KCA 4.1.1 /11	Selzer, J.; Eichelmann, C.	2013	Validation of the HPLC analytical method AM038813FP1- Determination of foramsulfuron (AE F130360) in technical grade and pure active substance by high performance liquid chromatography (HPLC) Bayer CropScience, Report No.: PA13/054, Edition Number:M-464439-01-1	N	Y	Triggered by the new method	Bayer CropScience	Submitted for the purpose of renewal

			Date: 2013-09-13 GLP/GEP: yes, unpublished					
B.5.1.2.1. KCA 4.1.2 /01	Wrede, A.	1999	Validation and analytical method for the determination of residues of AE F130360, iodosulfuron-methyl-sodium (AE F115008), and the metabolites AE F153745 and AE F075736 in maize by LC/MS/MS Code: AE F130360, AE F115008 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer CropScience, Report No.: C004020, Report includes Trial Nos.: CR98/022 Edition Number: M-187207-01-1 EPA MRID No.: 45108501 Date: 1999-05-12 GLP/GEP: no, unpublished ...also filed: KCA 4.2 /01	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.1.2.1 KCA 4.1.2 /02	Stuke, S.	2013	Modification M001 of the residue analytical method DGM F03/98-0 for the determination of metsulfuron-methyl (AE F075736), iodosulfuron-methyl-sodium (AE F115008), foramsulfuron (AE F130360), AE F153745 in corn (green material) by HPLC-MS/MS at a LOQ of 0.01 mg/kg Bayer CropScience, Report No.: MR-13/047, Edition Number: M-461902-01-1 Method Report No.: MR-13/047 Date: 2013-08-05 GLP/GEP: yes, unpublished	N	Y	Validation of the data generation method for the determination of foramsulfuron in green material at a reduced limit of quantification (0.01 mg/kg)	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.1. KCA 4.2 /01	Wrede, A.	1999	Validation and analytical method for the determination of residues of AE F130360, iodosulfuron-methyl-sodium (AE F115008), and the metabolites AE F153745 and AE F075736 in maize by LC/MS/MS Code: AE F130360, AE F115008 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer CropScience, Report No.: C004020, Report includes Trial Nos.: CR98/022 Edition Number: M-187207-01-1 EPA MRID No.: 45108501 Date: 1999-05-12 GLP/GEP: no, unpublished ...also filed: KCA 4.1.2 /01	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.2.1. KCA 4.2 /02	Reichert, N.	2000	Independent laboratory validation of a method of analysis for the determination of AE F130360 in corn grain Institut Fresenius Chem.und Biolog. Lab. AG, Taunusstein, Germany Report No.: C010383, Edition Number: M-198922-01-1	N	N	Not relevant	Bayer CropScience	In DAR 2001

			Date: 2000-10-26 GLP/GEP: yes, unpublished					
B.5.2.1. KCA 4.2 /03	Williams, L. E.	2000	Analytical method for the determination of residues of AE F130360 in corn grain by high performance liquid chromatography using UV detection: AE F130360 Aventis CropScience USA LP, Residue Chemistry, Pikeville, NC, USA Report No.: B002828, Edition Number:M-238559-01-1 EPA MRID No.: 45109415 Date: 2000-03-21 GLP/GEP: no, unpublished	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.2.1. KCA 4.2 /04	Campbell, J. K.	2000	A statement on the analytical method for the determination of residues of AE F130360 in corn grain by high performany liquid chromatography using UV detection Code: AE F130360 Aventis CropScience USA LP, Residue Chemistry, Pikeville, NC, USA Report No.: C007766, Edition Number:M-196608-01-1 EPA MRID No.: 45109416 GLP/GEP: no, unpublished	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.2.3. KCA 4.2 /08	Wrede, A.	2000	Enforcement Method for Soil by LC-MS/MS Metsulfuron-methyl (AE F075736) Iodosulfuron-methyl-sodium (AE F115008) Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer CropScience, Report No.: C006394, Edition Number:M-193807-01-1 EPA MRID No.: 45108502 Date: 2000-02-04 GLP/GEP: no, unpublished	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.2.3. KCA 4.2 /09	Wrede, A.	2000	Validation of the enforcement method EM F13/99-0 in soil by LC-MS/MS-Code: AE F130360 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Report No.: C006905, Edition Number:M-194836-01-1 EPA MRID No.: 45109421 Date: 2000-02-04 GLP/GEP: yes, unpublished	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.2.4 KCA 4.2 /10	Wrede, A.	2000	Enforcement method for surface and drinking water by HPLC-UV Code: AE F130360 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Report No.: C006998, Edition Number:M-195026-01-1 EPA MRID No.: 45109422	N	N	Not relevant	Bayer CropScience	In DAR 2001

			Date: 2000-02-22 GLP/GEP: no, unpublished					
B.5.2.4 KCA 4.2 /11	Wrede, A.	2000	Validation of the enforcement method EM F07/99-0 for surface and drinking water by HPLC-UV-Code: AE F130360 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Report No.: C007019, Edition Number:M-195060-01-1 EPA MRID No.: 45109423 Date: 2000-02-22 GLP/GEP: yes, unpublished	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.2.4 KCA 4.2 /12	Wrede, A.; Neuss, B.	2001	Validation of the enforcement method EM F04/00-0 for surface and drinking water by LC-MS/MS-Amidosulfuron (AE 075032)-Metsulfuron-methyl (AE F075736) - Iodosulfuron-methyl-sodium (AE F115008) -Mesosulfuron-methyl (AE F130060) -Foramsulfuron (AE F 130360) Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer CropScience, Report No.: C011207, Edition Number:M-200406-01-1 Date: 2001-02-14 GLP/GEP: yes, unpublished	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.2.5 KCA 4.2 /13	Sutton, A. L.; Everitt, S. L.	1999	Enforcement method with validation for air by GC-MSD active substance Code: AE F130360 AgrEvo UK Crop Protection Ltd., Chesterford Park, United Kingdom Report No.: C004845, Report includes Trial Nos.: 246/01/001 Edition Number:M-188704-01-1 EPA MRID No.: 45109424 Date: 1999-12-03 GLP/GEP: no, unpublished	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.2.5 KCA 4.2 /14	Reichert, N.	2000	Development and validation of an analytical method for the determination of AE F130360 in air Institut Fresenius Chem.und Biolog. Lab. AG, Taunusstein, Germany Report No.: C010595, Edition Number:M-227811-03-1 Date: 2000-11-22 ...Amended: 2002-05-08 GLP/GEP: no, unpublished	N	N	Not relevant	Bayer CropScience	In DAR 2001
B.5.2.1 KCA 4.2 /15	Wrede, A.	2000	Enforcement Method for Cereal Grain, Straw and Shoot by LC-MS/MS Amidosulfuron (AE F075032) Metsulfuron-methyl (AE F075736) Iodosulfuron-methyl-sodium (AE F115008) AE F130060 AE F130360 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer CropScience,	N	N	Not relevant	Bayer CropScience	Submitted for the purpose of renewal

			Report No.: C006734, Edition Number:M-194528-01-1 Date: 2000-03-02 GLP/GEP: no, unpublished					
B.5.2.1 KCA 4.2 /16	Wrede, A.	2000	Validation of the Enforcement Method EM F08/99-0 of cereal grain, straw and shoot by LC-MS/MS-Amidosulfuron (AE F075032) - Metsulfuron-methyl (AE F075736) Iodosulfuron-methyl-sodium (AE F115008) - AE F130060 - AE F 130360 Hoechst Schering AgrEvo GmbH, Frankfurt am Main, Germany Bayer CropScience, Report No.: C006735, Edition Number:M-194531-01-1 Date: 2000-03-02 GLP/GEP: yes, unpublished	N	Y	An enforcement method and associated ILV was developed on different crop groups.	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.1 KCA 4.2 /17	Wrede, A.	2002	Validation of the enforcement method EM F08/99-0 for lemon, tomato and maize kernel by LC-MS/MS - Amidosulfuron (AE F075032) - Iodosulfuron-methyl-sodium (AE F115008) - Mesosulfuron-methyl (AE F130060) - Foramsulfuron (AE F130360) Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer CropScience, Report No.: C022220, Edition Number:M-212674-01-1 Date: 2002-04-30 GLP/GEP: yes, unpublished	N	Y	An enforcement method and associated ILV was developed on different crop groups.	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.1 KCA 4.2 /18	Heinemann, O.	2004	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 Bayer CropScience, Report No.: 00815/M001, Report includes Trial Nos.:P602033000 Edition Number:M-226888-01-1 Date: 2004-01-30 GLP/GEP: yes, unpublished	N	Y	An enforcement method and associated ILV was developed on different crop groups.	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.1 KCA 4.2 /19	Reichert, N.; Klimmek, S.	2002	Independent laboratory validation of the analytical method EM F08/99-0 for the residue analysis of Amidosulfuron (AE F075032), Iodosulfuron-methyl-sodium (AE F115008), Mesosulfuron-methyl (AE F130060), Foramsulfuron (AE F130360) in tomato a Institut Fresenius Chem.und Biolog. Lab. AG, Taunusstein, Germany Bayer CropScience, Report No.: C023679, Edition Number:M-215456-01-1 Date: 2002-06-06	N	Y	An enforcement method and associated ILV was developed on different crop groups.	Bayer CropScience	Submitted for the purpose of renewal

			GLP/GEP: yes, unpublished					
B.5.2.1 KCA 4.2 /20	Stuke, S.; Ballmann, C.	2013	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 Bayer CropScience, Report No.: MR-13/007, Edition Number:M-455564-01-1 MethodReport No.: MR-13/007 Date: 2013-05-28 GLP/GEP: yes, unpublished ...also filed: KCA 6.1 /03	N	Y	Plant enforcement methods for Sus are not highly specific (only one ion transition). A confirmatory method was requested for corn grain and forage (EFSA).	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.1 KCA 4.2 /21	Konrad, S.	2013	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 Currenta GmbH & Co. OHG, Leverkusen, Germany BCS, Report No.: 2013/0060/01, Edition Number:M-470160-01-1 Date: 2013-10-18 GLP/GEP: yes, unpublished ...also filed: KCA 6.1 /04	N	Y	Plant enforcement methods for Sus are not highly specific (only one ion transition). A confirmatory method was requested for corn grain and forage (EFSA).	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.2. KCA 4.2 /22	Wrede, A.	2000	Enforcement Method for Animal tissue, Milk and Egg by LC-MS/MS Amidosulfuron (AE F075032) Metsulfuron-methyl (AE F075736) Iodosulfuron-methyl-sodium (AE F115008) Mesosulfuron-methyl (AE F130060) Foramsulfuron (AE F130360) Aventis CropScience GmbH, Frankfurt am Main, Germany Bayer CropScience, Report No.: EMF07/00-0, Edition Number:M-199702-02-1 Date: 2000-12-21 ...Amended: 2001-02-07 GLP/GEP: no, unpublished	N	N	Not relevant	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.2. KCA 4.2 /23	Wrede, A.	2001	Validation of the Enforcement Method EM F07/00-0 for Animal tissue, Milk and Egg by LC-MS/MS- Amidosulfuron (AE F075032) - Metsulfuron-methyl (AE F075736) - Iodosulfuron-methyl-sodium (AE F115008) -Mesosulfuron-methyl (AE F130060) -Foramsulfuron (AE F130360) Aventis CropScience GmbH, Frankfurt a m Main, Germany Bayer CropScience, Report No.: C011226, Edition Number:M-200439-01-1 EPA MRID No.: 46229001	N	Y	Validation of the enforcement method required for animal tissue	Bayer CropScience	Submitted for the purpose of renewal

			Date: 2001-02-07 GLP/GEP: yes, unpublished					
B.5.2.2. KCA 4.2 /24	Randolph, R.	2004	Independent Laboratory Validation for Aventis CropScience GmbH Analytical method No. EM/F07/00-0, Enforcement method for animal Tissue, Milk and Egg by LC-MS/MS Pyxant Labs, Inc., Colorado Springs, CO, USA Bayer CropScience, Report No.: B004802, Report includes Trial Nos.:RAMMY004 Edition Number:M-240268-01-1 EPA MRID No.: 48080501 Date: 2004-10-11 GLP/GEP: yes, unpublished	N	Y	ILV of the enforcement method required for animal tissue	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.2. KCA 4.2 /25	Schmeer, K.; Philipowski, C.	2010	Modification M001 of the residue analytical method 01208 for the determination of amidosulfuron (AE F075032), metsulfuron-methyl (AE F075736), iodosulfuron-methyl-sodium (AE F115008), mesosulfuron-methyl (AE F130060), foramsulfuron (AE F130360) in animal tissues (meat, fat, liver, kidney), egg, and milk by HPLC-MS/MS Bayer CropScience, Report No.: 01208/M001, Edition Number:M-389788-03-1 Method Report No.: MR-10/103 Date: 2010-09-06 ...Amended: 2011-01-03 GLP/GEP: yes, unpublished	N	Y	In the original EU dossier animal methods were not required. Due to new enforcement method and ILV were performed in 2010/2011 on animal matrices. It include a validation for all matrices and for two different mass transitions.	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.2. KCA 4.2 /26	Moore, S.	2010	Independent laboratory validation of an analytical method 01208/M001 for the determination of amidosulfuron (AE F075032), metsulfuron-methyl (AE F075736), iodosulfuron-methyl-sodium (AE F115008), mesosulfuron-methyl (AE F130060), foramsulfuron (AE F130360) in animal tissues (meat, fat, liver, kidney), egg, and milk by HPLC-MS/MS Bayer CropScience LP, Stilwell, KS, USA Bayer CropScience, Report No.: RAMML014, Edition Number:M-398300-01-1 Date: 2010-12-22 GLP/GEP: yes, unpublished	N	Y	In the original EU dossier animal methods were not required. Due to new enforcement method and ILV were performed in 2010/2011 on animal matrices. It include a validation for all matrices and for two different mass transitions.	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.3. KCA 4.2 /27	Freitag, T.	2008	Amendment no. 0001 to report no.: MR-08/138-Analytical Method 01115 for the determination of residues of amidosulfuron,iodosulfuron-methyl-sodium, metsulfuron-methyl, mesosulfuron-methyl and foramsulfuron in soil by HPLC-MS/MS Bayer CropScience, Report No.:M-310074-03-1,	N	Y	Method developed to current requirements	Bayer CropScience	Submitted for the purpose of renewal

			Edition Number:M-310074-03-1 Method Report No.: MR-08/138 Date: 2008-10-27 ...Amended: 2013-08-08 GLP/GEP: yes, unpublished					
B.5.2.4. KCA 4.2 /28	Krebber, R.; Braune, M.	2007	Analytical method 01058 for the determination of amidosulfuron, foramsulfuron, iodosulfuron-methyl-sodium, mesosulfuron-methyl and the metabolite metsulfuron-methyl (AE F075736) in drinking and surface water by HPLC-MS/MS Bayer CropScience, Report No.: 01058, Edition Number:M-291466-01-1 Method Report No.: MR-07/292 Date: 2007-08-14 GLP/GEP: yes, unpublished	N	Y	This residue analytical method was validated as a multi-residue analytical method for the sulfonylureas according to current requirements using LC/MS-MS with two MRM transitions	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.4. KCA 4.2 /29	Krebber, R.; Braune, M.	2013	Analytical method 01387 for the determination of various pesticides in drinking and surface water by HPLC-MS/MS Bayer CropScience, Report No.: MR-13/085, Edition Number:M-466732-01-1 Method Report No.: MR-13/085 Date: 2013-10-09 GLP/GEP: yes, unpublished	N	Y	Enforcement analytical method for water meeting all guideline criteria New data requirement	Bayer CropScience	Submitted for the purpose of renewal
B.5.2.4. KCA 4.2 /30	Stanislowski, T.	2013	Indepent laboratory validation of BCS analytical methods 01333 and 01387 for determination of various pesticides in surface water by Di-HPLC-MS/MS PTRI Europe, Ulm, Germany Bayer CropScience, Report No.: P3117 G, Edition Number:M-470714-02-1 Date: 2013-10-01 GLP/GEP: no, unpublished	N	Y	New data requirement	Bayer CropScience	Submitted for the purpose of renewal