FINAL REGISTRATION REPORT

Part B

Section 5

Analytical Methods

Detailed summary of the risk assessment

Product code: SHA 0724 A

Product name: COREY

Chemical active substances:

Rimsulfuron, 150 g/kg

Nicosulfuron, 300 g/kg

Central Zone

Zonal Rapporteur Member State: Poland

CORE ASSESSMENT

Applicant: SHARDA Cropchem España S.L.

Submission date: February 2020

Update date: January 2022

Finalisation date: 12.2020, 02.2022

Version history

When	What
December 2020	RMS assessment
January 2022	Applicant update
February 2022	Final vesion after applicant update

Table of Contents

5 Analytical methods

5.1 Conclusion and summary of assessment

Sufficiently sensitive and selective analytical methods are available for the active substance in the plant protection product.

Noticed data gaps are: none

Sufficiently sensitive and selective analytical methods are available for all analytes included in the residue definitions.
Noticed data gaps are:

- ILV for Nicosulfuron for drinking water.

Commodity/crop	Supported/ Not supported
Dry commodities / Maize	Supported

5.2 Methods used for the generation of pre-authorization data (KCP 5.1)

5.2.1 Analysis of the plant protection product (KCP 5.1.1)

5.2.1.1 Determination of active substance and/or variant in the plant protection product (KCP 5.1.1)

An overview on the acceptable methods and possible data gaps for analysis of Rimsulfuron and Nicosulfuron in plant protection product is provided as follows:

Reference:	KCP 5.1.1
Report	Rimsulfuron 15% + Nicosulfuron 30% WG: Method development and validation for the determination of active substances content in the formulation, Institute of Industrial Organic Chemistry Analytical department, Warsaw, Poland, report no. BA-37/16, report no. BA-37/16
Guideline(s):	SANCO/3030/99 rev4
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods

The analytical determination of Rimsulfuron and Nicosulfuron was performed by HPLC technique with UV/Vis detector using reversed phase column.

Preparation of solutions

Standard solution
Appropriate amounts of active substances standards were weighed (with the accuracy of 0.01 mg) into two 10 mL flask with a screw cap and acetonitrile was added up to the volume. The flask was put into the ultrasonic bath for 10 min.

Calibration curve
Into five 5 mL volumetric flasks the following amounts of standards were pipetted:

Solution No	**1**	**2**	**3**	**4**	**5**
Rimsulfuron	1.1 ml	1.3 ml	1.5 ml	1.7 ml	2.2 ml
Nicosulfuron	0.7 ml	0.9 ml	1.1 ml	1.3 ml	1.5 ml

Acetonitrile was added to the mark and the solutions were stirred.

Specimen solution
About 30 mg of examined specimen was weighed (with the accuracy of 0.01 mg) into a 10 mL flask with a screw cap, 2 mL of water and 5 mL of acetonitrile was added. The flask was put into the ultrasonic bath for 10 min. After cooling acetonitrile was added up to the volume. Six samples were prepared to assess the repeatability.

Validation - Results and discussions

The analytical method for determination of Rimsulfuron and Nicosulfuron active ingredient content in Rimsulfuron 15% + Nicosulfuron 30% WG was validated. The validation covered the aspects namely: Specificity, Linearity, Instrument precision, Repeatability and Accuracy.

Specificity
The chromatograms of placebo, solvent, solution of standard mixture and the examined specimen solution were performed and superimposed. There are no interferences between the analytes and other components of the specimen. There are no interferences between the analytes and other components of the specimen.

Linearity
The linearity of the detector response was assessed using five mixtures of standard solutions at the concentration range of rimsulfuron from 0.2929 mg/mL to 0.6277mg/mL, which corresponds to the concentration range from 65% to 140% of rimsulfuron content in the preparation and nicosulfuron from 0.5489 mg/mL to 1.0978 mg/mL, which corresponds to the concentration range from 62% to 125% of nicosulfuron content in the preparation. All solutions were analysed twice. Correlation coefficient should be $R2 \geq 0.99$.

Repeatability
The method repeatability was assessed on the basis of six independent determinations of active substances content in Rimsulfuron 15% + Nicosulfuron 30% WG preparation.
The obtained result for Rimsulfuron (RSDr = RSD * 0.67 = 0.62 %) is acceptable.
The obtained result for Nicosulfuron (RSDr = RSD * 0.67 = 0.64 %) is acceptable.

Precision of the system

Mixture of standard solution of active substances at concentration of rimsulfuron 0.4603 mg/mL and nicosulfuron 0.7485 mg/mL was injected six times into the chromatographic column and detector responses were recorded.

Accuracy
Accuracy of active substance determination in Rimsulfuron 15% + Nicosulfuron 30% WG was assessed by recovery value at two levels of concentration. The concentration of analytes in each solution was calculated from the equation of the calibration curve. Obtained final concentrations were examined and the nominal and calculated contents were compared.
For the main ingredient at concentration of > 10 % the average recovery value should be 100 ± 2 %. The obtained result of 99.67% for Rimsulfuron is acceptable.
For the main ingredient at concentration of > 10 % the average recovery value should be 100 ± 2 %. The obtained result of 99.22% for Nicosulfuron is acceptable

Table 5.2-1: Methods suitable for the determination of Rimsulfuron and Nicosulfuron in plant protection product Rimsulfuron 15% + Nicosulfuron 30% WG

	Rimsulfuron	**Nicosulfuron**
Author(s), year	Małgorzata Wołoszynowska MSc; 2017	
Principle of method	HPLC with UV/Vis detector	
Linearity (linear between mg/L / % range of the declared content) (correlation coefficient, expressed as r)	Linearity range: 0.2929 – 0.6277 mg/mL Calibration equotion y = 6801745x + 1567291 Correlation coefficient R^2 = 0.9979	Linearity range: 0.5489 – 1.0978 mg/ml Calibration equotion y = 5816690x + 3450231 Correlation coefficient R^2 = 0.9950
Precision – Repeatability Mean n = 6 (%RSD)	RSD = 0.92% Modified Horwitz - % RSDr = RSD × 0.67 = 0.62 RSDr = 1.78	RSD = 0.95% Modified Horwitz - % RSDr = RSD × 0.67 = 0.64 RSDr = 1.61
Accuracy n = 6 (% Total Recovery)	99.67%	99.22%
Interference/ Specificity	No interference	No interference
RMS Comment	According the requirements SANCO 3030/99 rev. 5. the Horrat ratio can be added (0.92/1.78 = 0,52 so this is <1)	According the requirements SANCO 3030/99 rev. 5. the Horrat ratio can be added (0.95/1.64 = 0,58 so this is <1)

Conclusion

The method for the determination of Rimsulfuron and Nicosulfuron in Rimsulfuron 15% + Nicosulfuron 30% WG is acceptable and validated according the requirements SANCO 3030/99 rev. 4.

5.2.1.2 Description of analytical methods for the determination of relevant impurities (KCP 5.1.1)

Not relevant.

5.2.1.3 Description of analytical methods for the determination of formulants (KCP

5.1.1)

Not relevant.

5.2.1.4 Applicability of existing CIPAC methods (KCP 5.1.1)

A CIPAC method No. 716 is available for Rimsulfuron.
A CIPAC method No. 709 is available for Nicosulfuron.

5.2.2 Methods for the determination of residues (KCP 5.1.2)

Please refer to post-registration methods.

5.3 Methods for post-authorization control and monitoring purposes (KCP 5.2)

5.3.1 Analysis of the plant protection product (KCP 5.2)

Analytical methods for the determination of the active substance and relevant impurities in the plant protection product shall be submitted, unless the applicant shows that these methods already submitted in accordance with the requirements set out in point 5.2.1 can be applied.

5.3.2 Description of analytical methods for the determination of residues of Rimsulfuron (KCP 5.2)

5.3.2.1 Overview of residue definitions and levels for which compliance is required

Compared to the residue definition proposed in the Draft Assessment Report (incl. its addenda) the current legal residue definition is identical.

Table 5.3-1: Relevant residue definitions for monitoring/enforcement and levels for which compliance is required

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Plant, high water content	Rimsulfuron	0.01 mg/kg	Regulation No. 617/2014
Plant, high acid content		0.01 mg/kg	Regulation No. 617/2014
Plant, high protein/high starch content (dry commodities)		0.01 mg/kg	Regulation No. 617/2014
Plant, high oil content		0.01 mg/kg	Regulation No. 617/2014
Plant, difficult matrices (hops, spices, tea)		0.05 mg/kg	Regulation No. 617/2014
Muscle	Rimsulfuron	0.02 mg/kg	Regulation No. 617/2014
Milk		0.02 mg/kg	Regulation No. 617/2014
Eggs		0.02 mg/kg	Regulation No. 617/2014
Fat		0.02 mg/kg	Regulation No. 617/2014

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Liver, kidney		0.02 mg/kg	Regulation No. 617/2014
Soil (Ecotoxicology)	Rimsulfuron	0.05 mg/kg	common limit
Drinking water (Human toxicology)	Rimsulfuron	0.1 µg/L	general limit for drinking water
Surface water (Ecotoxicology)	Rimsulfuron	4.6 µg/L	Lowest EC_{50} from *L. minor* study
Air	Rimsulfuron	21 µg/m^3	AOEL sys: 0.07 mg/kg bw/d
Tissue (meat or liver)		not required	not classified as T / T+
Body fluids		not required	not classified as T / T+

5.3.2.2 Description of analytical methods for the determination of residues in plant matrices (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Rimsulfuron in plant matrices is given in the following tables (please refer to the DAR of Rimsulfuron, July 2005).

Table 5.3-2: Validated methods for food and feed of plant origin (required for all matrix types, "difficult" matrix only when indicated by intended GAP)

Component of residue definition: Rimsulfuron				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
High water content	Primary	0.05 mg/kg 0.05 mg/kg	HPLC-UV HPLC-UV	LaRochelle, 1989 Amoo, 1996
	ILV	0.05 mg/kg	HPLC-UV	Clayton, 2001
	Confirmatory (if required)	0.01 mg/kg	LC-MS/MS	Fulton, 2001
High acid content	Primary	-	-	-
	ILV	-	-	-
	Confirmatory (if required)	-	-	-
High oil content	Primary	-	-	-
	ILV	-	-	-
	Confirmatory (if required)	-	-	-
High protein/high starch content (dry)	Primary	0.05 mg/kg 0.05 mg/kg	HPLC-UV HPLC-UV	LaRochelle, 1989 Amoo, 1996
	ILV	0.05 mg/kg	HPLC-UV	Clayton, 2001
	Confirmatory	0.01mg/kg	LC-MS/MS	Fulton, 2001

Component of residue definition: Rimsulfuron				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
	(if required)			
High protein/high starch content (dry)	Primary	0.01 mg/kg	LC-MS/MS	KCP 5.2.1. A. Markowicz, 2019 Report No. 19/FSL/15/1A
	ILV	0.01 mg/kg	LC-MS	KCP 5.2.1.1 M. Rubino, 2019 Report No. 19.500341.0001
	Confirmatory (if required)	-	-	The applied LC-MS/MS is highly selective method and 2 transitions were monitored, therefore no other confirmatory method is required.

Table 5.3-3: Statement on extraction efficiency

	Method for products of plant origin
Required, available from:	Extraction efficiency was not investigated in this study as a solvent system similar to that used in metabolism studies reported in the DAR from the previous EU Review for rimsulfuron was used for extraction (Volume 3, Annex B, B.7, July 2005. Brown and Young (1989) AMR 1222-88 (maize); Brown (1990) AMR 1444-89 (potato) and Zhang et al. (1996) AMR 3520-95 (tomato foliage)).
Not required, because:	-

Study Comments: IIIA 5.3.2.2	~~Adequate method exists to monitor Rimsulfuron residues in high protein/high starch content (dry). The analytical methods presented by the applicant are active substances data, which were reviewed in the Assessment Report for Rimsulfuron and were considered adequate.~~ ~~Additional methods for the purpose of the evaluation are not required.~~ None of the methods mentioned in Table 5.3 2 were considered still acceptable in the renewal assessment report of rimsulfuron, since they do "*not meet the current guideline SANCO/825/00*" (RAR, Slovenia, 10/2017). Additionally, the LOQ of 0.05 mg/kg validated in these methods (except Fulton, 2001), does not cover the lowered MRLs from Regulation (EU) No 617/2014 of 0.01 mg/kg for all matrix groups, including maize. Consequently, monitoring of residues of rimsulfuron at 0.01 mg/kg cannot be ensured. The Applicant provided new studies for determination of residues of rimsulfuron in maize: primary method (LC-MS/MS, LOQ = 0.01 mg/kg) and ILV (LC-MS, LOQ = 0.01 mg/kg). The analytical method meets the criteria of specificity, linearity, repeatability and accuracy. The method is acceptable and is suitable for determination of rimsulfuron in maize.

Agreed end-point: IIIA 5.3.2.2	~~Residues of Rimsulfuron in commodities with high protein/high starch content (dry):~~ ~~HPLC-UV~~ ~~LOQ = 0.05 mg/kg~~ ~~LC-MS/MS~~ ~~LOQ = 0.01 mg/kg~~ LC-MS LOQ = 0.01 mg/kg

5.3.2.3 Description of analytical methods for the determination of residues in animal matrices (KCP 5.2)

Not relevant, no residue definition is proposed.

Study Comments: IIIA 5.3.2.3	The explanation provided by the applicant is accepted.
Agreed end-point: IIIA 5.3.2.3	As no residue definition was proposed, the analytical method for the determination of residues of Rimsulfuron in animal matrices is not required.

5.3.2.4 Description of methods for the analysis of soil (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Rimsulfuron in soil is given in the following tables (please refer to the DAR of Rimsulfuron, July 2005).

Table 5.3-4: Validated methods for soil (if appropriate)

Component of residue definition: Rimsulfuron, IN-70912, IN-70941, IN-J0290 and IN-E9260			
Method type	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary (Rimsulfuron)	0.2 µg/kg 0.05 µg/kg	LC-MS/MS LC-MS/MS	Connolly, 2001 (This method is specific, validated on two mass transitions, so confirmatory is not required) Hill and Stry, 2001 (This method is specific, validated on two mass transitions, so confirmatory is not required)
Confirmatory (Rimsulfuron)	-	-	-
Primary (IN-70942, IN-70941, IN-J0290, IN-E9260 metabolites)	0.2 µg/kg	LC-MS/MS	Connolly, 2001 (This method is specific, validated on two mass transitions, so confirmatory is not required)
Confirmatory (IN-70942, IN-70941,	-	-	-

Component of residue definition: Rimsulfuron, IN-70912, IN-70941, IN-J0290 and IN-E9260			
Method type	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
IN-J0290, IN-E9260 metabolites)			

*IN-70942, IN-70941, IN-J0290, IN-E9260 metabolites are not component of residue definition.

Study Comments: IIIA 5.3.2.4	Adequate method exists to monitor Rimsulfuron residues in soil. The analytical methods presented by the applicant are active substances data, which were reviewed in the Assessment Report for Rimsulfuron and were considered adequate. Additional methods for the purpose of the evaluation are not required.
Agreed end-point: IIIA 5.3.2.4	Residues of Rimsulfuron in soil: LC-MS/MS LOQ = 0.2 µg/kg

5.3.2.5 Description of methods for the analysis of water (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Rimsulfuron in surface and drinking water is given in the following tables (please refer to the DAR of Rimsulfuron, July 2005).

Table 5.3-5: Validated methods for water (if appropriate)

Component of residue definition: Rimsulfuron				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Drinking water	Primary	0.1 µg/L 0.05 µg/L	HPLC-UV LC-MS/MS	Powley and de Bernard, 1996 Devine and Jin, 2001 (This method is specific, validated on two mass transitions, so confirmatory is not required)
	ILV	-	-	According to SANCO/825/00 rev. 8.1, ILV is not required.
	Confirmatory	0.1 µg/L	LC-MS/MS	Jin, 2001
Drinking water	Primary	0.05 µg/L	LC/MS	KCP 5.2.2 M. Rubino, 2019 Report No. 19.500341.0007
	ILV	0.05 µg/L	LC-MS/MS	KCP 5.2.2.1 M. Zarębska, 2020 Report No. 30/2020
	Confirmatory	-	-	The applied LC-MS is highly selective method and 2 transitions were monitored, therefore no other confirmatory method is required.
Surface water	Primary	0.1µg/L	HPLC-UV	Powley and de Bernard, 1996

Component of residue definition: Rimsulfuron				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
		0.05 µg/L	LC-MS/MS	Devine and Jin, 2001 (This method is specific, validated on two mass transitions, so confirmatory is not required)
	Confirmatory	0.1 µg/L	LC-MS/MS	Jin, 2001

Study Comments: IIIA 5.3.2.5	Adequate method exists to monitor Rimsulfuron residues in ~~drinking and~~ surface water. The analytical methods presented by the applicant are active substances data, which were reviewed in the Assessment Report for Rimsulfuron and were considered adequate. Additional methods for the purpose of the evaluation are not required. With regard to the drinking water: ILV for drinking water is required according to Regulation (EU) 284/2013, legally binding in contrast to guideline. The Applicant provided new studies for determination of residues of rimsulfuron in drinking water: primary method (LC-MS, LOQ = 0.05 µg/L) and ILV (LC-MS/MS, LOQ = 0.05 µg/L). The analytical method meets the criteria of specificity, linearity, repeatability and accuracy. The method is acceptable and is suitable for determination of rimsulfuron in drinking water.
Agreed end-point: IIIA 5.3.2.5	Residues of Rimsulfuron in surface water: HPLC-UV LOQ = 0.1 µg/L LC-MS/MS LOQ = 0.05 µg/L Residues of Rimsulfuron in drinking water: LC-MS LOQ = 0.05 µg/L

5.3.2.6 Description of methods for the analysis of air (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Rimsulfuron in air is given in the following tables (please refer to the DAR of Rimsulfuron, July 2005).

Table 5.3-6: Validated methods for air (if appropriate)

Component of residue definition: Rimsulfuron			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	3 µg/m^3 air	LC-MS/MS	Bacher, 2001 (This method is specific, validated on two mass

Component of residue definition: Rimsulfuron			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
			transitions, so confirmatory is not required)
Confirmatory	-	-	-

Study Comments: IIIA 5.3.2.6	Adequate LC-MS/MS method exists to monitor Rimsulfuron residues in air. The analytical methods presented by the applicant are active substances data, which were reviewed in the Assessment Report for Rimsulfuron and were considered adequate. Additional methods for the purpose of the evaluation are not required.
Agreed end-point: IIIA 5.3.2.6	Residues of Rimsulfuron in air: LC-MS/MS LOQ = 3 $\mu g/m^3$

5.3.2.7 Description of methods for the analysis of body fluids and tissues (KCP 5.2)

Not relevant, the active substance Rimsulfuron is not classified as toxic or very toxic, no residue method for body fluids and tissues is required.

5.3.2.8 Other studies/ information

No new or additional studies have been submitted.

5.3.3 Description of analytical methods for the determination of residues of Nicosulfuron (KCP 5.2)

5.3.3.1 Overview of residue definitions and levels for which compliance is required

Compared to the residue definition proposed in the Draft Assessment Report (incl. its addenda) the current legal residue definition is identical.

Table 5.3-7: Relevant residue definitions for monitoring/enforcement and levels for which compliance is required

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Plant, high water content	Nicosulfuron	0.01 mg/kg	Regulation No. 617/2014
Plant, high acid content		0.01 mg/kg	Regulation No. 617/2014

Matrix	Residue definition	MRL / limit	Reference for MRL/level Remarks
Plant, high protein/high starch content (dry commodities)		0.01 mg/kg	Regulation No. 617/2014
Plant, high oil content		0.01 mg/kg	Regulation No. 617/2014
Plant, difficult matrices (hops, spices, tea)		0.05 mg/kg	Regulation No. 617/2014
Muscle	Nicosulfuron	0.02 mg/kg	Regulation No. 617/2014
Milk		0.02 mg/kg	Regulation No. 617/2014
Eggs		0.02 mg/kg	Regulation No. 617/2014
Fat		0.02 mg/kg	Regulation No. 617/2014
Liver, kidney		0.02 mg/kg	Regulation No. 617/2014
Soil (Ecotoxicology)	Nicosulfuron	0.05 mg/kg	common limit
Drinking water (Human toxicology)	Nicosulfuron	0.1 µg/L	general limit for drinking water
Surface water (Ecotoxicology)	Nicosulfuron	1.7 µg/L	Lowest EC_{50} from *Lemna gibba* study
Air	Nicosulfuron	240 µg/m^3	AOEL sys: 0.8 mg/kg bw/d
Tissue (meat or liver)		Not required	not classified as T / T+
Body fluids		Not required	not classified as T / T+

5.3.3.2 Description of analytical methods for the determination of residues in plant matrices (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Nicosulfuron in plant matrices is given in the following tables (please refer to the DAR of Nicosulfuron, June 2006 and Addendum to the DAR, July 2007).

Table 5.3-8: Validated methods for food and feed of plant origin (required for all matrix types, "difficult" matrix only when indicated by intended GAP)

Component of residue definition: Nicosulfuron				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
High water content	Primary (Nicosulfuron)	0.01 mg/kg 0.01 mg/kg	HPLC-UV HPLC-MS/MS	Huber, 1996a Wolf, 2000
	ILV (Nicosulfuron)	0.01 mg/kg	HPLC-MS/MS	Ginzburg, 2000
	Confirmatory (if required) (Nicosulfuron)	0.025mg/kg	GC/MS, LC-MS	Mirbach, 1998

Component of residue definition: Nicosulfuron				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
	Primary (ADMP metabolite)*	0.04 mg/kg	HPLC-UV	Huber, 1996a
	ILV (ADMP metabolite)*	-	-	-
	Confirmatory (if required) (ADMP metabolite)*	-	-	-
	Primary (ASDM metabolite)*	0.06 mg/kg	HPLC-UV	Huber, 1996a
	ILV (ASDM metabolite)*	-	-	-
	Confirmatory (if required) (ASDM metabolite)*	-	-	-
High acid content	Primary	-	-	-
	ILV	-	-	-
	Confirmatory (if required)	-	-	-
High oil content	Primary	-	-	-
	ILV	-	-	-
	Confirmatory (if required)	-	-	-
High protein/high starch content (dry)	Primary (Nicosulfuron)	0.02 mg/kg 0.01 mg/kg	HPLC-UV HPLC-MS/MS	Huber, 1996a Wolf, 2000
	ILV (Nicosulfuron)	0.01 mg/kg	HPLC-MS/MS	Ginzburg, 2000
	Confirmatory (if required) (Nicosulfuron)	0.025mg/kg	GC/MS, LC-MS	Mirbach, 1998
	Primary (ADMP metabolite)*	0.04 mg/kg	HPLC-UV	Huber, 1996a
	ILV (ADMP metabolite)*	-	-	-
	Confirmatory (if required) (ADMP metabolite)*	-	-	-
	Primary (ASDM metabolite)*	0.02 mg/kg	HPLC-UV	Huber, 1996a
	ILV (ASDM metabolite)*	-	-	-
	Confirmatory (if required)	-	-	-

Component of residue definition: Nicosulfuron				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing / EU agreed**
	(ASDM metabolite)*			
Difficult (if required, depends on intended use)	Primary	-	-	-
	ILV	-	-	-
	Confirmatory (if required)	-	-	-

* ADMP and ASDM are not components of residue definition.

Table 5.3-9: Statement on extraction efficiency

	Method for products of plant origin
Required, available from:	DAR, 2005
Not required, because:	-

Study Comments: IIIA 5.3.3.2	Adequate method exists to monitor Nicosulfuron residues in high protein/high starch content (dry). The analytical methods presented by the applicant are active substances data, which were reviewed in the Assessment Report for Nicosulfuron and were considered adequate. Additional methods for the purpose of the evaluation are not required.
Agreed end-point: IIIA 5.3.3.2	Residues of Nicosulfuron in commodities with high protein/high starch content (dry): HPLC-UV LOQ = 0.02 mg/kg HPLC-MS/MS LOQ = 0.01 mg/kg

5.3.3.3 Description of analytical methods for the determination of residues in animal matrices (KCP 5.2)

An analytical method for food of animal origin is not required due to the fact that no residue definition is proposed (*EFSA Scientific Report (2007) 120, 1-91*).

Study Comments: IIIA 5.3.3.3	The explanation provided by the applicant is accepted.
Agreed end-point: IIIA 5.3.3.3	As no residue definition was proposed, the analytical method for the determination of Nicosulfuron residues in animal matrices is not required.

5.3.3.4 Description of methods for the analysis of soil (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Nicosulfuron in soil is given in the following tables (please refer to the DAR of Nicosulfuron, June 2006 and Addendum to the DAR, July 2007).

Table 5.3-10: Validated methods for soil (if appropriate)

Component of residue definition: Nicosulfuron			
Method type	**Method LOQ**	**Principle of method (*i.e.* GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary (Nicosulfuron)	0.005 mg/kg	HPLC-UV	Huber, 1996b
Confirmatory (Nicosulfuron)	0.05 µg/kg	LC-MS/MS	Wais, 2000a
Primary (ADMP and ASDM metabolites)	0.02 mg/kg	HPLC-UV	Huber, 1996b
Confirmatory (ADMP and ASDM metabolites)	-	-	-
Primary (AUSN and UCSN metabolites)	0.01 mg/kg	LC-MS/MS	Wolf, 2003 (This method is specific, validated on two mass transitions, so confirmatory is not required)
Confirmatory (AUSN and UCSN metabolites)	-	-	-

*ADMP, ASDM, AUSN and UCSN are not component of residue definition.

Study Comments: IIIA 5.3.3.4	Adequate method exists to monitor Nicosulfuron residues in soil. The analytical methods presented by the applicant are active substances data, which were reviewed in the Assessment Report for Nicosulfuron and were considered adequate. Additional methods for the purpose of the evaluation are not required.
Agreed end-point: IIIA 5.3.3.4	Residues of Nicosulfuron in soil: LC-MS/MS LOQ = 0.05 µg/kg

5.3.3.5 Description of methods for the analysis of water (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Nicosulfuron in surface and drinking water is given in the following tables (please refer to the DAR of Nicosulfuron, June 2006 and Addendum to the DAR, July 2007).

Table 5.3-11: Validated methods for water (if appropriate)

Component of residue definition: Nicosulfuron				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Drinking water (Nicosulfuron)	Primary	0.05 µg/L 0.05 µg/L	HPLC-UV HPLC-MS/MS	Schulz and Ullrich-Mitzel, 1995a Wolf, 2007 (This method is specific, validated on two mass transitions, so confirmatory is not required)
	ILV	-	-	According to SANCO/825/00 rev. 8.1,

Component of residue definition: Nicosulfuron				
Matrix type	**Method type**	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
				ILV is not required.
	Confirmatory	0.05 µg/L	LC-DAD	Wais, 2000b
Drinking water (ADMP, ASDM and AUSN metabolites)*	Primary	0.05 µg/L	HPLC-UV	Wais and Ullrich-Mitzel, 1997
	ILV	-	-	According to SANCO/825/00 rev. 8.1, ILV is not required.
	Confirmatory	-	-	-
Surface water (Nicosulfuron)	Primary	0.05 µg/L	HPLC-MS/MS	Wolf, 2007 (This method is specific, validated on two mass transitions, so confirmatory is not required)
	Confirmatory	0.05 µg/L	HPLC-DAD	Wais, 2000b

*ADMP, ASDM and AUSN are not component of residue definition.

Study Comments: IIIA 5.3.3.5	Adequate method exists to monitor Nicosulfuron residues in ~~drinking and~~ surface water. The analytical methods presented by the applicant are active substances data, which were reviewed in the Assessment Report for Nicosulfuron and were considered adequate. Additional methods for the purpose of the evaluation are not required. With regard to the drinking water: ILV for drinking water is required according to Regulation (EU) 284/2013, legally binding in contrast to guideline. Data gap for ILV for drinking water is stated.
Agreed end-point: IIIA 5.3.3.5	Residues of Nicosulfuron in surface water: HPLC-UV LOQ = 0.05 µg/L LC-DAD LOQ = 0.05 µg/L Residues of Nicosulfuron in drinking water: Data gap on ILV for drinking water.

5.3.3.6 Description of methods for the analysis of air (KCP 5.2)

An overview on the acceptable methods and possible data gaps for analysis of Nicosulfuron in air is given in the following tables (please refer to the DAR of Nicosulfuron, June 2006 and Addendum to the DAR, July 2007).

Table 5.3-12: Validated methods for air (if appropriate)

Component of residue definition: Nicosulfuron			
Method type	**Method LOQ**	**Principle of method (i.e. GC-MS or HPLC-UV)**	**Author(s), year / missing**
Primary	1.2 µg/m^3 air 1.2 µg/m^3 air	HPLC-UV HPLC-UV	Schulz and Ullrich-Mitzel, 1995b Wais, 2000c
Confirmatory	-	-	Sufficient confirmatory methods are available for the determination in soil or water therefore confirmatory methods for the determination of residues in air are not required.

Study Comments: IIIA 5.3.3.6	Adequate method exists to monitor Nicosulfuron residues in air. The analytical methods presented by the applicant are active substances data, which were reviewed in the Assessment Report for Nicosulfuron and were considered adequate. Additional methods for the purpose of the evaluation are not required.
Agreed end-point: IIIA 5.3.3.6	Residues of Nicosulfuron in air: HPLC-UV LOQ = 1.2 µg/m^3

5.3.3.7 Description of methods for the analysis of body fluids and tissues (KCP 5.2)

As Nicosulfuron is classified as neither toxic nor highly toxic, methods for therapeutic analysis are not required (*DAR, 2005*).

5.3.3.8 Other studies/ information

No new or additional studies have been submitted.

Appendix 1 Lists of data considered in support of the evaluation

Tables considered not relevant can be deleted as appropriate.

MS to blacken authors of vertebrate studies in the version made available to third parties/public.

List of data submitted by the applicant and relied on

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
KCP 5.1.1	Małgorzata Wołoszynowska MSc	2017	Rimsulfuron 15% + Nicosulfuron 30% WG: Method development and validation for the determination of active substances content in the formulation Institute of Industrial Organic Chemistry Analytical department, Warsaw, Poland, report no. BA-37/16 GLP; unpublished	N	Sharda Cropchem Limited
KCP 5.2.1	Anna Markowicz	2019	Validation of the method for determination of Rimsulfuron in maize by liquid chromatograpchy Food Safety Labolatory Research Institute of Horticulture, Report No. 19/FSL/15/1A GLP Unpublished	N	Sharda Cropchem Limited
KCP 5.2.1.1	Manuel Rubino	2019	Validation of the analytical procedure for the determination of residues of Rimsulfuron (CAS: 122931-48-0) in maize by LC-MS CHELAB, Report No. 19.500341.0001 GLP Unpublished	N	Sharda Cropchem Limited
KCP 5.2.2	Manuel Rubino	2019	Validation of the analytical procedure for the determination of residues of Rimsulfuron (CAS: 122931-48-0) in drinking water by LC-MS CHELAB, Report No. 19.500341.0007 GLP Unpublished	N	Sharda Cropchem Limited
KCP	Magdalena Zarębska	2020	Independent Labolatory Validation of the analytical procedure for the determination of residues of	N	Sharda

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
5.2.2.1			Rimsulfuron (CAS: 122931-48-0) in drinking water by liquid chromatography Institute of Heavy Organic Synthesis “Blachownia”, Report No. 30/2020 GLP Unpublished		Cropchem Limited

List of data submitted or referred to by the applicant and relied on, but already evaluated at EU peer review

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
-	-	-	-	-	-

The following tables are to be completed by MS

List of data submitted by the applicant and not relied on

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
-	-	-	-	-	-

List of data relied on not submitted by the applicant but necessary for evaluation

Data point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Owner
-	-	-	-	-	-

Appendix 2 Detailed evaluation of submitted analytical methods

A 2.1 Analytical methods for Rimsulfuron

A 2.1.1 Methods used for the generation of pre-authorization data (KCP 5.1)

No new or additional studies have been submitted

A 2.1.2 Methods for post-authorization control and monitoring purposes (KCP 5.2)

A 2.1.2.1 Description of analytical methods for the determination of residues in plant matrices (KCP 5.2)

~~No new or additional studies have been submitted~~

A 2.1.2.1.1 Analytical method 1

A 2.1.2.1.1.1 Method validation

Comments of zRMS:	The analytical method meets the criteria of specificity, linearity, repeatability and accuracy. The method is acceptable and is suitable for determination of rimsulfuron in maize.

Reference:	KCP 5.2.1
Report	Validation of the method for determination of Rimsulfuron in maize by liquid chromatograpchy, Anna Markowicz, 2019, Report No. 19/FSL/15/1A
Guideline(s):	SANCO/3029/99 Rev. 4 SANCO/825/00 Rev. 8.1
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods

Untreated maize samples were storage in the Test Facility in a freezer (≤ -20 °C) in the dark.

Reference substance:

- Rimsulfuron, CAS 122931-48-0, Batch G753032 purchased from Dr. Ehrenstorfer, Purity 96.56%

Reagents:

- Triphenyl phosphate TPP, CAS 115-86-6, Batch 00020650-0138 purchased from ChromaDex, Purity 99.9%;
- Acetonitrile LC/MS grade, CAS 75-05-8, No. 1.00029.2500 from Merck;

- Ammonium formate LC/MS grade, CAS 540-69-2, No. 14266-25G from Fluka;
- ESI-L Low Concentration Tuning Mix, No. G1969-85000 from Agilent;
- Formic acid LC/MS grade, CAS 64-18-6, No. 56302-50ML-F from Fluka;
- Magnesium sulfate anhydrous, CAS 7487-88-9, No. 208094 from Sigma-Aldrich;
- Sodium chloride, CAS 7647-14-5, No. S9888 from Sigma-Aldrich;
- Sodium hydrogen citrate sesquihydrate, CAS 6132-05-4, No. 359084 from Sigma-Aldrich;
- Sodium citrate tribasic dihydrate, CAS 6132-04-3, No. S4641 from Sigma-Aldrich;
- Water LC/MS grade from Merck (Direct-Q).

Materials and apparatus:

- Analytical balance, Sartorius CPA225D-0EC, d=0.01 mg (100 g);
- Analytical balance, Sartorius CPA224S-0CE, d=0.1 mg (220 g);
- Centrifuge Tubes, Thermo Scientific, 50 mL Teflon® FEP;
- Common laboratory glassware;
- Eppendorf® centrifuge tubes, FL Medical, Round-bottom polypropylene Safe-lock, micro-centrifuge tubes (volume 2 ml);
- Freezer, Whirlpool AFG 651-B;
- Freezer, MPM MPM-270-SK-03;
- Freezer, Gorenje F6181AX;
- HPLC Autosampler vials, Anchem Amber glass, 1.5 mL short thread vial;
- Laboratory centrifuge, MPW MPW-350;
- Laboratory centrifuge, Eppendorf Mini Spin;
- Laboratory balance, Radwag PS 1200/C/2, d=0.01g (1200 g);
- Laboratory balance, Radwag WPT 5C;
- Laboratory mill, Robot Coupe R5 Plus;
- Mobile Phase Filtration Apparatus, Sartorius Stedim, Glass filtration system;
- Pipettors, Brand, Set of adjustable pipettors, checked for accuracy and precision, and capable of delivering volumes ranging from 10-1000 µL (10-100 µL, 20-200 µL, 100-1000 µL);
- PTFE Syringe Filter, Alfatec Hydrophilic PTFE, 0.22 µm, 17 mm;
- PTFE Membrane Filter, Membrane Solutions Hydrophilic PTFE, 0.22 µm, 47 mm;
- Pipettes, Glass, Class A, volumetric, various sizes (0.5 ml, 1 mL, 2.5 ml, 5 mL, 10 mL;
- QuEChERS Hand Motion Shaker, Eberbach EL680.Q;
- Refrigerator, Whirlpool, ARZ-845/H;
- Syringe, B. Braun Medical, All plastic barrel, plunger and tip (2 mL);
- Vacuum Pump, KNF Laboport N820.3FT.18;
- Volumetric flasks, Glass, Class A, various sizes (10 mL, 50 mL, 100 mL);
- Vortex Mixer, IKA MS 3 Digital.

Chromatographic conditions:

- HPLC system: Series 1260 Infinity II (Agilent Technologies), Binary Pump (G7111B), Multisampler (G7167A), Thermostatted Column Compartment MCT(G7116A);
- Pre-Column: Agilent 1290 Infinity In-Line Filter (PN: 5067-4368) with 0.3µm frit ring installed (PN: 5023-0271);
- Column: Agilent Poroshell 120 (PN: 699975-302) EC-C18 2.7µm 3.0 x 50 mm
- Column oven temperature: 40 °C;
- Injection volume: 10 µL;
- Autosampler temperature: 10 °C;
- Flow: 0.4 mL/min;
- Mobile phase A: 5 mM Ammonium Formate with 0.01% (v/v) Formic Acid in Water;
- Mobile Phase B: 5 mM Ammonium Formate with 0.01% (v/v) Formic Acid in ACN:H2O 95:5 (v/v);
- Gradient:

Time (min)	Mobile phase A%	Mobile phase B%
0.00	90	10
1.00	90	10

10.00	10	90
14.00	10	90

- Post time: 4 min;
- Total analysis time: 18 min;
- Retention time (approx.): 6.5 min for Rimsulfuron;
- MS system: Agilent Technologies 6470 Triple Quad LC/MS
- Ionization type: Electrospray (ESI, Agilent Jet Stream, G1958-65138)
- Polarity: Positive ion mode;
- Drying gas temperature: 225 °C;
- Drying gas flow: 8 (l/min);
- Sheath gas heater: 350 °C;
- Sheath gas flow: 11 (l/min);
- Nebulizer pressure: 40 (psi);
- Capillary voltage: 5000 (V);
- Nozzle voltage: 500 (V);
- Scan type: MS/MS, Multiple Reaction Monitoring (MRM);
- Time segments:

Index	Start Time (min)	Divert Valve	Polarity	Delta EMV
1	0	To Waste	Negative	0
2	5	To MS	Positive	200
3	11	To Waste	Positive	0

- Scan resolution (FWHM):

MS1- Unit (0.7 amu)	MS2- Unit (0.7 amu)

- Ion mass transition:

Ion mass transition monitored m/z			m/z	Collision energy (V)
Rimsulfuron	432.1	Trans 1:	182.0	21
		Trans 2:	325.0	13
TPP	327.1	Trans 1:	77.0	49
		Trans 2:	152.0	45

Solutions preparation:

- Mobile Phase A: 5 mM ammonium formate 0.01% Formic acid:

500 mL volumetric flask was half filled with water, 0.157 g of ammonium formate (NH4HCO2) and 50 µl of formic acid (HCOOH) was added than solution was agitated gently until all ammonium formate was completely dissolved. Volumetric flask was filled up to the mark with water, closed tightly and mixed by inverting several times. Subsequently proceeded to solvent filtration apparatus equipped with a 0.22 µm Teflon filter. After filtration solvent was transferred to amber HPLC solvent reservoir.

- Mobile Phase B: 5mM Ammonium formate 0.01% formic acid in ACN: H2O, 95:5 (v/v):

500 mL mixing cylinder was filled with 475 mL of acetonitrile. Then, 25 mL volumetric flask was half filled with water, 0.157 g of ammonium formate (NH4HCO2) and 50 µl of formic acid (HCOOH) was added than solution was agitated gently until all ammonium formate was completely dissolved. Volumetric flask was filled up to the mark with water, closed tightly and mixed by inverting several times. Content of volumetric flask was transferred to mixing cylinder. Cylinder was closed and mixed by inverting several times. Subsequently proceeded to solvent filtration apparatus equipped with 0.22 µm Teflon filter. After filtration solvent was transferred to HPLC solvent reservoir.

- Extraction mixture: Acetonitrile (+1 Vol% formic acid):

500 mL volumetric flask was half filled with acetonitrile, 5 mL of formic acid (HCOOH) was added and the solution was agitated gently. Volumetric flask was filled up to the mark with acetonitrile, closed tightly and mixed by.

- Working and stability testing standard solutions:

Using balance reading to five decimals places the appropriate amount of reference item neat standard (corrected for purity) was weighted into appropriate volumetric flask such that when diluted in acetonitrile it yield standard solution of reference item containing 1 mg/mL. Then such prepared stock was used to prepare the standards solutions by diluting with appropriate volume of acetonitrile to obtain the indicated concentrations.

- Solvent and matrix-matched calibration solutions for rimsulfuron:

The respective final sample extracts of control (untreated) samples are fortified with working solution of Rimsulfuron. Exemplary pipetting scheme for the preparation of solvent and matrix matched calibrations at 0.001 µg/mL and 0.01 µg/mL is presented below.

Additions		Solvent based		Matrix-matched	
Calibration levels (µg/mL)		0.001	0.01	0.001	0.01
Volume of sample extract (µL)		-	-	200	200
Volume of Solvent A (µL)		700	700	700	700
Volume of Acetonitrile (µL)		200	200	-	-
Volume of TPP (µL)		50	50	50	50
Pesticide working solutions	0.02 (µg/mL)	50	-	50	-
	0.2 (µg/mL)	-	50	-	50
Total volume (µL)		1000	1000	1000	1000

Sample preparation:

- Sample extraction

5.00 g ± 0.05 g of homogenized matrix was weighed into a 50 mL Teflon centrifuge tube. Sample weight was recorded. If necessary, fortification of the concurrent recovery sample(s) by aliquoting the fortification standard onto the matrix was carried out at this step. The tube was shaken in a vortex mixer for 1 min and allowed to stand for about 5 min. Using glass volumetric pipette 10 mL of water was added. Using glass volumetric pipette 10 mL of acidified acetonitrile (1% formic acid v/v) was added. The Teflon centrifuge tube was closed tightly and shaken thoroughly for 2 min.

- Liquid-Liquid Partition

A salt mixture (4 g ± 0.2 g of magnesium sulfate anhydrous and 1 g ± 0.05 g of sodium chloride) was added and the centrifuge tube was closed and shaken for 1 min. The extract was centrifuged at 8100 rpm for 5 min.

- Sample Dilution

An aliquot of 0.2 mL of was transferred to new Eppendorf safe-lock tube and subsequently diluted with 0.7 mL of solvent A, 0.05 mL of acetonitrile and 0.05 mL of TPP (0.5 µg/mL). Content was mix gently and filtered through the 0.22 µm Teflon filter attached to a syringe direct into amber HPLC vial. Vial was labelled so that it may be identified.

Results and discussions

Specificity:

The method is specific for the determination of Rimsulfuron by virtue of the chromatographic separation and selective detection system used. Reagents blank and control specimens were extracted and analyzed according to the method to investigate the presence of residue and/or background interference at the retention time of the analyte. For both ion mass transition 1 and 2, the specimen showed no significant interference (≤ 30% LOQ) at the retention time of the analyte.

Linearity:

The linearity of the detector response for Rimsulfuron was demonstrated by single determination of matrix-matched calibration standards at nine concentration levels ranging from 0.0002 µg/mL to 0.1 µg/mL for maize whole plant and grain. This range correspond from 0.002 mg/kg to 1 mg/kg thus covering the range from no more than 30 % of the LOQ and at least + 20 % of the highest analyte concentration level detected in samples.

Accuracy and Precision:

Five recovery determinations were performed at the LOQ (0.01 mg/kg) and at the 10 x LOQ (0.1 mg/kg) for maize grain and at the LOQ (0.01 mg/kg) and at the 50 x LOQ (0.5 mg/kg) for maize whole plant respectively. Analysis was performed by extraction and single injection.

The mean recovery values at the fortification levels of 0.01 mg/kg, 0.1 mg/kg and 0.5 mg/kg for both ion mass transitions were all in the range 70 – 110 % and thus comply with the standard acceptance criteria. All precision values at the fortification levels of 0.01 mg/kg, 0.1 mg/kg and 0.5 mg/kg for both ion mass

transitions were < 20%.

Limit of Quantification (LOQ):
The LOQ of the method was defined as the lowest analyte concentration at which the methodology had been successfully validated. Thus, an LOQ of 0.01 mg/kg was confirmed for Rimsulfuron in maize matrices.

Table A 1: Recovery results from method validation of Rimsulfuron using the analytical method

Matrix	Analyte	Fortification level (mg/kg) (*n* = 5)	Mean recovery (%)	RSD (%)	Comments
Maize whole plant	Rimsulfuron	0.01	Trans 1: 88 Trans 2: 88	Trans 1: 3.5 Trans 2: 3.5	-
Maize whole plant	Rimsulfuron	0.5	Trans 1: 92 Trans 2: 93	Trans 1: 4.3 Trans 2: 4.1	-
Maize grain	Rimsulfuron	0.01	Trans 1: 75 Trans 2: 76	Trans 1: 4.0 Trans 2: 6.2	-
Maize grain	Rimsulfuron	0.1	Trans 1: 83 Trans 2: 83	Trans 1: 3.6 Trans 2: 3.5	-

Table A 2: Characteristics for the analytical method used for validation of Rimsulfuron residues in maize

	Rimsulfuron
Specificity	The method is specific. Reagents blank and control specimens were extracted and analyzed according to the method to investigate the presence of residue and/or background interference at the retention time of the analyte. For both ion mass transitions, the specimen showed no significant interference (above 30 % of the LOQ) at the retention time of the analyte.
Calibration (type, number of data points)	It was evalueated 9 different levels of concentration covering the range from no more than 30 % of the LOQ and at least + 20 % of the highest analyte concentration level detected in samples. Regresion plot of Transition 1 for maize whole plant: $y=0.576358x + 0.0002272284$, $R^2=0.99972503$ Regresion plot of Transition 2 for maize whole plant: $y=0.244676x + 0.0001974397$, $R^2=0.99968640$ Regresion plot of Transition 1 for maize grain: $y=0.531089x + 0.0007327460$, $R^2=0.99875093$ Regresion plot of Transition 2 for maize grain: $y=0.225681x + 0.0004165412$, $R^2=0.99832024$
Calibration range	Accepted calibration range in concentration units from 0.002 mg/kg to 1 mg/kg. Corresponding calibration range in mass ratio units for the sample was 0.01 mg/kg.
Assessment of matrix effects is presented	Yes
Limit of determination/quantification	Limit of determination was 0.002 mg/kg; Limit of quantification was 0.01 mg/kg.

Conclusion

According to SANTE/2020/12830 Rev.1 the method was sufficiently validated and it is suitable for de-

termination of Rimsulfuron in maize.

A 2.1.2.1.1.2 Independent laboratory validation

Comments of zRMS:	The analytical method meets the criteria of specificity, linearity, repeatability and accuracy. The method is acceptable and is suitable for determination of rimsulfuron in maize.

Reference:	KCP 5.2.1.1
Report	Validation of the analytical procedure for the determination of residues of Rimsulfuron (CAS: 122931-48-0) in maize by LC-MS, Manuel Rubino, 2019, Report No. 19.500341.0001
Guideline(s):	SANCO/3029/99 Rev. 4 SANCO/825/00 Rev. 8.1 OECD-204/2014
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods

Maize specimens were supplied by the Test Facility and the absence of Rimsulfuron was ensured before using the test item. The test substance was grinded and stored in a freezer before use (about -20°C).

Reference substance:
- Rimsulfuron, CAS 122931-48-0, Batch 774850 purchased from HPC Standards GmbH, Purity 99.4 %

Reagents:
- MilliQ water, SRA 787;
- Acetonitrile, TI-0038770 purchased from VWR;
- Ammonium formate, TI-0037141 purchased from Sigma Aldrich;
- Bakerbond Octadecyl (C18) 40 µm, TI0017316 purchased from J.T.Baker;
- Formic acid (99-100%), TI-0028375 purchased from VWR;
- Magnesium sulfate anhydrous, TI-0023069 purchased from Sigma Aldrich.

Materials and apparatus:
- Common analytical glassware;
- Fridge, SRA 7;
- Analytical balance (±0,1 mg), SRA 602;
- Analytical balance (±0,01 mg), SRA 768;
- Vortex;
- Ultrasonic bath, SRA 469;
- Centrifuge, SRA 55;
- Thermostatic bath equipped with N_2 flow, SRA 66;
- Syringe filter 0.45 µm RC-membrane;
- MS XEVO TQS (Waters-Micromass), SRA 470;
- Acquity UPLC BEH C18, 50 mm x 2.1 mm x 1.7 µm (ID: LC 23).

Instrumental conditions:

- Column: Acquity UPLC BEH C18, 50 mm x 2.1 mm x 1.7 µm (LC 23);
- Mobile Phase A: 10 mM ammonium formate buffer pH 4;
- Mobile Phase B: Acetonitrile;
- Flow: 0.2 mL/min;
- Retention time: ~5.10 minutes;
- Injection Volume: 10 µL;
- Detector: MS XEVO TQS (Waters-Micromass), SRA 470;
- Source: ESI+;
- Source temp.: 150 °C;
- Nebulizer: 6 bar;
- Cone gas: 150 L/h;
- Desolvation gas: 800 L/h;
- Run time: 13 minutes;
- Run mode: MRM (see table below):

	Precursor ion m/z		m/z	Collision energy
Rimsulfuron	432	Quantifier ion (trans 1):	182	20
		Qualifier ion (trans 2):	325	15

- Elution: Gradient

Time (min)	Mobile phase A%	Mobile phase B%
0.00	100	0
0.50	100	0
8.50	0	100
11.50	0	100
11.60	100	0
13.00	100	0

Solutions preparation:

- Mobile phase A (10 mM ammonium formate buffer pH 4):

About 0.62 g of ammonium formate were into a 1000 ml volumetric flask and dissolved with about 500 ml of milliQ water. 0.22 ml of formic acid were added and then the solution was diluted to volume with milliQ water. pH was 4.

- Mobile phase B:

Acetonitrile.

- Blank solution:

10 mM ammonium formate buffer pH 4: acetonitrile, 50:50 (v:v).

- Extraction phase (1% acid formic in acetonitrile:

In a 1000 ml volumetric flask containing about 50 ml of acetonitrile, about 10 ml of formic acid were introduced and then diluted to volume with acetonitrile.

- Stock Reference Standard Solution (SRSS):

10.52 mg (± 0.01 mg) of rimsulfuron were accurately weighed into a 25 m| volumetric flask and diluted to volume with acetonitrile. The final concentration was 418.28 mg/L.

- Intermediate Reference Solution A (IRS-A):

0.15 ml of SRSS were introduced into a 20 ml volumetric flask and diluted to volume with acetonitrile. The final concentration was 3.14 mg/L.

- Intermediate Reference Solution B (IRS-B):

1 ml of IRS-A was introduced into a 10 ml volumetric flask and diluted to volume with acetonitrile. The final concentration was 0.31 mg/L.

- Intermediate Reference Solution C (IRS-C):

1 ml of IRS-B was introduced into a 10 ml volumetric flask and diluted to volume with acetonitrile. The final concentration was 0.03 mg/L.

- Linearity solutions:

Linearity solutions were prepared in order to cover the range from about 30% LOQ (0.003 mg/kg) to about 30xLOQ (0.38 mg/kg) on the sample. LOQ corresponds to 0.01 mg/kg. Solutions were filled up to volume with blank solution. Solution corresponding to about LOQ in the sample, was injected in triplicate

for system suitability evaluation; the other solutions were individually injected.

Sample extraction:
About 1 g (± 0.1 mg) of maize was weighed into a 50 ml falcon and 10 ml of extraction phase were added. After vortexing for about 1 min, the sample was introduced into the ultrasonic bath for 5 minutes. The tube was centrifuged at 4700 rpm for 5 min and then the purification step was carried out.
6 ml of supernatant were transferred into a 10 ml plastic tube, containing about 900 mg of magnesium sulphate anhydrous and 150 mg of C18 resin. Vortexed for about1 min and centrifuged at 4000 rpm for 5 min. 1 ml of supernatant was transferred into a 10 ml tube and dried by N_2 flux. The dried sample was then resuspended into 1 ml of blank solution. Vortexed for about 1 min, filtered, transferred into an HPLC vial and injected. The sample was prepared in duplicate.

Reference solution in matrix (for matrix effect calculation):
0.5 ml of supernatant of purified sample were transferred into a 10 ml tube and dried by N_2 flux. The dried sample was then resuspended into 0.5 ml of solution (reference standard at 10xLOQ), vortexed for about 1 min, filtered, transferred into an HPLC vial and injected.

Spiked Sample at LOQ level:
About 1 g (± 0.1 mg) of maize was weighed into a 50 ml falcon, added 0.30 ml of IRS-C solution and added 10 ml of extraction phase. After vortexing for about 1 min, the sample was introduced into the ultrasonic bath for 5 minutes. The tube was centrifuged at 4700 rpm for 5 min and then the purification step was carried out.
6 ml of supernatant were transferred into a 10 ml plastic tube, containing about 900 mg of magnesium sulphate anhydrous and 150 mg of C18 resin. Vortexed for about 1 min and centrifuged at 4000 rpm for 5 min. 1 ml of supernatant was transferred into a 10 ml tube and dried by N_2 flux. The dried sample was then resuspended into 1 ml of blank solution. Vortexed for about 1 min, filtered, transferred into an HPLC vial and inject The sample was prepared in quintuplicate.

Spiked Sample at 10xLOQ level:
About 1 g (± 0.1 mg) of maize was weighed into a 50 ml falcon, added 0.30 ml of IRS-B solution and added 10 ml of extraction phase. After vortexing for about 1 min, the sample was introduced into the ultrasonic bath for 5 minutes. The tube was centrifuged at 4700 rpm for 5 min and then the purification step was carried out.
6 ml of supernatant were transferred into a 10 ml plastic tube, containing about 900 mg of magnesium sulphate anhydrous and 150 mg of C18 resin. Vortexed for about 1 min and centrifuged at 4000 rpm for 5 min. 1 ml of supernatant was transferred into a 10 ml tube and dried by N_2 flux. The dried sample was then resuspended into 1 ml of blank solution. Vortexed for about 1 min, filtered, transferred into an HPLC vial and inject. The sample was prepared in quintuplicate.

Results and discussions

Specificity:
The method is capable to determine the analyte in the presence of the sample matrix. No significant peaks (≤ 30% LOQ) are detected at RT of the target analyte in the blank and test solution with respect to the spiked test solution for both transition 1 and 2.

Linearity:
The method linearity was evaluated at 5 different levels of concentration, ranging from at least 30% LOQ (0.003 mg/kg) to about 30xLOQ (0.38 mg/kg) of analyte on the sample.

Repeatability precision:
Repeatability evaluation was performed on aliquots of sample spiked with Rimsulfuron at LOQ (about 0.01 mg/kg), and 10xLOQ (about 0.10 mg/kg). 5 replicate analyses were performed for each spiking level.

Accuracy:
Recovery is included between 70% and 110% in all cases, in accordance with acceptance criteria.

Limit of Quantification (LOQ):
LOQ is the lowest concentration level where an acceptable degree of linearity, accuracy and precision is established. In this case LOQ corresponds to 0.01 mg/kg.

Table A 3: Recovery results from independent laboratory validation of Rimsulfuron using the analytical method

Matrix	Analyte	Fortification level (mg/kg) (n = 5)	Mean recovery (%)	RSD (%)	Comments
Maize	Rimsulfuron	0.01	Trans 1: 106 Trans 2: 84	Trans 1: 3 Trans 2: 1	-
Maize	Rimsulfuron	0.10	Trans 1: 92 Trans 2: 83	Trans 1: 1 Trans 2: 2	-

Table A 4: Characteristics for the analytical method used for independent laboratory validation of Rimsulfuron residues in maize

	Rimsulfuron
Specificity	The method is capable to determine the analyte in the presence of the sample matrix. No significant peaks (≤30% LOQ) are detected at RT of the target analyte in the blank and test solution with respect to the spiked test solution for both Transition 1 and 2.
Calibration (type, number of data points)	It was evalueated 5 different levels of concentration starting from 30% of the LOQ to at least 120% of the expected highest concentration. Regresion plot of Transition 1: $y=2128335x$, $R^2=0.9960$ Regresion plot of Transition 2: $y=781595x$, $R^2=0.9948$
Calibration range	Accepted calibration range in concentration units from 0.003 mg/kg to 0.38 mg/kg. Corresponding calibration range in mass ratio units for the sample was 0.01 mg/kg.
Assessment of matrix effects is presented	Yes.
Limit of determination/quantification	Limit of quantification was 0.01 mg/kg.

Conclusion

According to SANTE/2020/12830 Rev.1 the method was sufficiently validated and it is acceptable as ILV for the primary method for determination of Rimsulfuron in maize.

A 2.1.2.1.1.3 Confirmatory method (if required)

No confirmatory method is required

A 2.1.2.2 Description of analytical methods for the determination of residues in animal matrices (KCP 5.2)

No new or additional studies have been submitted

A 2.1.2.3 Description of Methods for the Analysis of Soil (KCP 5.2)

No new or additional studies have been submitted

A 2.1.2.4 Description of Methods for the Analysis of Water (KCP 5.2)

~~No new or additional studies have been submitted~~

A 2.1.2.4.1 Analytical method 1

A 2.1.2.4.1.1 Method validation

Comments of zRMS:	The analytical method meets the criteria of specificity, linearity, repeatability and accuracy. The method is acceptable and is suitable for determination of rimsulfuron in drinking water.

Reference:	KCP 5.2.2
Report	Validation of the analytical procedure for the determination of residues of Rimsulfuron (CAS: 122931-48-0) in drinking water by LC-MS, Manuel Rubino, 2019, Report No. 19.500341.0007
Guideline(s):	SANCO/3029/99 Rev. 4 SANCO/825/00 Rev. 8.1 OECD-204/2014
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods

Drinking water specimens were supplied by the Test Facility and the absence of Rimsulfuron was ensured before using the test item. The test substance was stored in fridge at 4°C.

Reference substance:
- Rimsulfuron, CAS 122931-48-0, Batch 774850 purchased from HPC Standards GmbH, Purity 99.4 %

Reagents:
- MilliQ water, SRA 787;
- Methanol, TI-0038421 purchased from VWR;
- Acetonitrile, TI-0038183 purchased from Merck;
- Ammonium formate, TI-0037141 purchased from Sigma Aldrich;
- Formic acid, TI-0028375 purchased from VWR;
- Isolute column C18 500 mg/3 ml (C18), ID: TI-0Q28490, purchased from Biotage.

Materials and apparatus:

- Common analytical glassware;
- Fridge, SRA 7;
- Analytical balance (±0,01 mg), SRA 768;
- Vortex;
- Centrifuge, SRA 55;
- Thermostatic bath equipped with N_2 flow, SRA 66;
- MS XEVO TQS (Waters-Micromass), SRA 470;
- Acquity UPLC BEH C18, 50 mm x 2.1 mm x 1.7 µm (ID: LC 23).

Instrumental conditions:

- Column: Acquity UPLC BEH C18, 50 mm x 2.1 mm x 1.7 µm (LC 23);
- Mobile Phase A: 10 mM ammonium formate buffer pH 4;
- Mobile Phase B: methanol;
- Flow: 0.2 mL/min;
- Retention time: 6.40 minutes;
- Injection Volume: 5 µL;
- Detector: MS XEVO TQS (Waters-Micromass), SRA 470;
- Source: ESI+;
- Source temp.: 150 °C;
- Nebulizer: 6 bar;
- Cone gas: 150 L/h;
- Desolvation gas: 800 L/h;
- Run time: 13 minutes;
- Run mode: MRM (see table below):

	Precursor ion m/z		m/z	Collision energy
Rimsulfuron	432	Quantifier ion (trans 1):	182	20
		Qualifier ion (trans 2):	325	15

- Elution: Gradient

Time (min)	Mobile phase A%	Mobile phase B%
0.00	100	0
0.30	100	0
8.50	0	100
11.50	0	100
11.60	100	0
13.00	100	0

Solutions preparation:

- Mobile phase A (10 mM ammonium formate buffer pH 4):

About 0.62 g of ammonium formate were accurately weighed (±0.01 g) into a 1000 ml volumetric flask and dissolved with about 500 ml of milliQ water. 0.22 ml of formic acid were added and then the solution was diluted to volume with milliQ water. pH was 4.

- Mobile phase B:

Methanol.

- Blank solution:

10 mM ammonium formate buffer pH 4: acetonitrile, 50:50 (v:v).

- Stock Reference Standard Solution (SRSS):

10.27 mg (± 0.10 mg) of rimsulfuron were accurately weighed into a 25 m| volumetric flask and diluted to volume with acetonitrile. The final concentration was 408.34 mg/L.

- Intermediate Reference Solution A (IRS-A):

0.15 ml of SRSS were introduced into a 20 ml volumetric flask and diluted to volume with acetonitrile. Then, 1 ml of this solution was introduced into a 10 ml of volumetric flask and diluted to volume with blank solution. The final concentration was 306.25 µg/L.

- Intermediate Reference Solution B (IRS-B):

1 ml of IRS-A was introduced into a 10 ml volumetric flask and diluted to volume with blank solution. The final concentration was 30.63 mg/L.

- Linearity solutions:

Linearity solutions were prepared in order to cover the range from about 30% LOQ (0.013 µg/l) to about 30xLOQ (1.53 µg/L) on the sample. LOQ corresponds to 0.05 µg/L. Solutions were filled up to volume with blank solution. Solution corresponding to about LOQ in the sample, was injected in triplicate for system suitability evaluation; the other solutions were individually injected.

Sample extraction:
80.0 mL (±0.1 mL) of drinking water were introduced into a becker and added 0.5 ml of formic acid. Then, the water was percolated using a regular vacuum through a C18 cartridge (previously conditioned with 3 ml of methanol and 3 ml of milliQ water) with a flow rate of 2 drop/sec. Then, the cartridge was centrifuged (2000 rpm, 2 min) to eliminate all the water. The analyte was eluted with 4 ml of blank solution and recovered. Transferred into an HPLC vial and inject. The sample was prepared in duplicate.

Reference solution in matrix (for matrix effect calculation):
80.0 ml (±0.1 ml) of drinking water were introduced into a becker and added 0.5 ml of formic acid. Then, the water was percolated using a regular vacuum through a C18 cartridge (previously conditioned with 3 ml of methanol and 3 ml of milliQ water) with a flow rate of 2 drop/sec. Then, the cartridge was centrifuged (2000 rpm, 2 min) to eliminate all the water. The analyte was eluted with 4 ml of blank solution and recovered. 0.5 ml of etuate were transferred in 10 ml of tube and dried by N_2 flux. The dried sample was resuspended with 0.5 ml of solution corresponded at 10xLOQ on the sample. Vortexed and transferred into an HPLC vial and injected.

Spiked Sample at LOQ level:
80.0 ml (±0.1 ml) of drinking water were introduced into a becker, added 0.15 ml of IRS-B and added 0.5 ml of formic acid. Then, the water was percolated using a regular vacuum through a C18 cartridge (previously conditioned with 3 ml of methanol and 3 ml of milliQ water) with a flow rate of 2 drop/sec. Then, the cartridge was centrifuged (2000 rpm, 2 min) to eliminate all the water. The analyte was eluted with 4 ml of blank solution and recovered. Transferred into an HPLC vial and injected. The sample was prepared in quintuplicate.

Spiked Sample at 10xLOQ level:
80.0 ml (±0.1 ml) of drinking water were introduced into a becker, added 0.15 ml of IRS-A and added 0.5 ml of formic acid. Then, the water was percolated using a regular vacuum through a C18 cartridge (previously conditioned with 3 ml of methanol and 3 ml of milliQ water) with a flow rate of 2 drop/sec. Then, the cartridge was centrifuged (2000 rpm, 2 min) to eliminate all the water. The analyte was eluted with 4 ml of blank solution and recovered. Transferred into an HPLC vial and injected, The sample was prepared in quintuplicate.

Results and discussions

Specificity:
The method is capable to determine the analyte in the presence of the sample matrix. No significant peaks (≤ 30% LOQ) are detected at RT of the target analyte in the blank and test solution with respect to the spiked test solution for both transition 1 and 2.

Linearity:
The method linearity was evaluated at 5 different levels of concentration, ranging from at least 30% LOQ (0.015 µg/L) to about 30xLOQ (1.53 µg/L) of analyte on the sample.

Repeatability precision:
Repeatability evaluation was performed on aliquots of sample spiked with Rimsulfuron at LOQ (about 0.05 µg/L), and 10xLOQ (about 0.50 µg/L). 5 replicate analyses were performed for each spiking level.

Accuracy:
Recovery is included between 70% and 110% in all cases, in accordance with acceptance criteria.

Limit of Quantification (LOQ):
LOQ is the lowest concentration level where an acceptable degree of linearity, accuracy and precision is established. In this case LOQ corresponds to 0.05 µg/L.

Table A 5: Recovery results from method validation of Rimsulfuron using the analytical method

Matrix	Analyte	Fortification level (µg/L) (n = 5)	Mean recovery (%)	RSD (%)	Comments
Drinking water	Rimsulfuron	0.05	Trans 1: 104 Trans 2: 105	Trans 1: 0.5 Trans 2: 3.8	-
Drinking water	Rimsulfuron	0.5	Trans 1: 105 Trans 2: 107	Trans 1: 1.1 Trans 2: 1.6	-

Table A 6: Characteristics for the analytical method used for validation of Rimsulfuron residues in drinking water

	Rimsulfuron
Specificity	The method is capable to determine the analyte in the presence of the sample matrix. No significant peaks (≤30% LOQ) are detected at RT of the target analyte in the blank and test solution with respect to the spiked test solution for both Transition 1 and 2.
Calibration (type, number of data points)	It was evalueated 5 different levels of concentration starting from 30% of the LOQ to at least 120% of the expected highest concentration. Regresion plot of Transition 1: $y=7904x$, $R^2=0.9994$ Regresion plot of Transition 2: $y=2157x$, $R^2=0.9953$
Calibration range	Accepted calibration range in concentration units from 0.015 µg/L to 1.53 µg/L. Corresponding calibration range in mass ratio units for the sample was 0.05 µg/L.
Assessment of matrix effects is presented	Yes
Limit of determination/quantification	Limit of determination was 0.015 µg/L. Limit of quantification was 0.05 µg/L.

Conclusion

According to SANTE/2020/12830 Rev.1 the method was sufficiently validated and it is suitable for determination of Rimsulfuron in drinking water.

A 2.1.2.4.1.2 Independent laboratory validation

Comments of zRMS:	The analytical method meets the criteria of specificity, linearity, repeatability and accuracy. The method is acceptable and is suitable for determination of rimsulfuron in drinking water.

Reference:	KCP 5.2.2.1
Report	Independent Labolatory Validation of the analytical procedure for the determination of residues of Rimsulfuron (CAS: 122931-48-0) in drinking water by liquid chromatography, Magdalena Zarębska, 2020, Report No. 30/2020
Guideline(s):	SANCO/3029/99 Rev. 4 SANCO/825/00 Rev. 8.1
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods

The objective of this study was to perform Independent Laboratory Validation Study of the analytical method for the determination of Rimsulfuron residues in drinking water matrix. The analysis was performed on drinking water according the procedure delivered by the Sponsor "Validation of the analytical procedure for the determination of Rimsulfuron (CAS: 122931-48-0), in drinking water by LC/MS", M. Rubino, 19.500341.0007.

Reference substance:
- Rimsulfuron, CAS 122931-48-0, Batch BCBZ5003 purchased from Sigma Aldrich, Purity 98.3 %

Reagents:
- Water Direct Q3 UV remote, Merck Millipore, CAS 7732-18-5;
- Acetonitrile LC/MS grade, CAS 75-05-8;
- Methanol LC/MS grade, CAS 67-56-1;
- Ammonium formate, LC/MS grade; CAS 540-69-2;
- Formic acid LC/MS, CAS 64-18-6.

Materials and apparatus:
- liquid chromatograph Dionex UltiMate 3000 RS with computer program „CHROMELEON version 6.80";
- mass spectrometer Sciex Q TRAP 4000 with computer program "ANALYST version 1.5.1";
- chromatographic column Synergi Fusion RP, 4µm, 50x2mm;
- analytical balance type AG285, Mettler Toledo;
- automatic pipette Brand Transferpette S 10-100 µl;
- automatic pipette Brand Transferpette S 100-1000 µl;
- Chromabond C18 columns;
- A class laboratory glassware.

Chromatographic conditions:
- Column: Acquity UPLC BEH C18, 50 mmx2.1 mm, 1.7µm (LC 120) changed to Synergi Fusion RP, 4µm, 50x2mm BA-AB MS 07 - No impact;
- Detector: MS/MS;
- Solvent A: 10mM ammonium formate buffer pH 4;
- Solvent B: Methanol;
- Eluent flow: 0.2 mL/min changed to 0.6 mL/min - No impact;
- Column oven temperature: 25°C
- Retention time: ~6.4 minutes changed to ~3.8 minutes due to different column length - No im-

pact;
- Injection volume: 5µl;
- Elution mode: Gradient

Time (min)	Mobile phase A%	Mobile phase B%
0.00	100	0
0.30	100	0
8.50	0	100
11.50	0	100
11.60	100	0
13.00	100	0
Changed to due to different column length – No impact		
0.00	100	0
0.10	100	0
3.00	0	100
4.00	0	100
4.10	100	0
5.00	100	0

- MS System: Sciex Q TRAP 4000

Precursor ion m/z			m/z
Rimsulfuron	432	Quantification (trans 1):	182
		Confirmation (trans 2):	325

- Ionisation type: Electrospray;
- Polarity: Positive ion mode;
- Temperature: 600°C;
- Nebulizing gas, Gas1: 60 psig;
- Drying gas, Gas2: 50 psig;
- Curtain Gas: 35 psig;
- Ion Spray Voltage: 5200 V;
- Scan type: MS/MS, Multiple Reaction Monitoring (MRM);
- Scan resolution: MS1/MS2 –UNIT (0.7 amu);
- Dwell time: 150 msec.

Results and discussions

Specificity:
For both ion mass transitions of Test Solution (unfortified sample) and Blank Solution, the value obtained was lower than 30% of value obtained by injection of standard solution 1.0µg/l (concentration of the standard solution corresponding to 0.05µg/l, LOQ) thus no significant interferences or contamination by Rimsulfuron were found on matrix blank sample.

Linearity:
The linearity of the detector response for Rimsulfuron was demonstrated by single injection of calibration standards at five concentration levels ranging from about 30% LOQ (0.31µg/l what corresponds to 0.015 µg/l) to about 30xLOQ (30.61µg/l what corresponds to 1.53µg/l) of analyte on the sample.

Precision (repeatability):
Repeatability evaluation was performed on aliquots of sample spiked with Rimsulfuron at LOQ (about 0.05 µg/L), and 10xLOQ (about 0.5 µg/L). 5 replicate analyses were performed for each spiking level. % RSD at each fortified level was calculated for both transitions.

Accuracy:
The mean recovery values at the fortification levels of LOQ (0.05 µg/L) and 10xLOQ (0.5 µg/L) for both ion mass transitions of Rimsulfuron were all in the range of 70% – 110% and thus comply with the standard acceptance criteria.

Limit of Quantification (LOQ):
The LOQ of the method at 0.05 µg/L was defined as the lowest analyte concentration at which the methodology was successfully validated.

Table A 7: Recovery results from independent laboratory validation of Rimsulfuron using the analytical method

Matrix	Analyte	Fortification level (µg/L) (n = 5)	Mean recovery (%)	RSD (%)	Comments
Drinking water	Rimsulfuron	0.05	Trans 1: 94 Trans 2: 94	Trans 1: 5.9 Trans 2: 5.0	-
Drinking water	Rimsulfuron	0.5	Trans 1: 101 Trans 2: 102	Trans 1: 1.9 Trans 2: 2.5	-

Table A 8: Characteristics for the analytical method used for independent laboratory validation of Rimsulfuron residues in drinking water

	Rimsulfuron
Specificity	The method is specific. No significant interferences or contaminations (≤30% LOQ) are detected at RT of the target analyte in the blank and test solution with respect to the spiked test solution for both Transition 1 and 2.
Calibration (type, number of data points)	It was evalueated 5 different levels of concentration starting from 30% of the LOQ to 30xLOQ. Regresion plot of Transition 1: $y=3.18e+004x+4.96e+004$, $R^2=0.9980$ Regresion plot of Transition 2: $y=1.85e+004x+2.89e+004$, $R^2=1$
Calibration range	Accepted calibration range in concentration units from 0.015 µg/L to 1.53 µg/L. Corresponding calibration range in mass ratio units for the sample was 0.05 µg/L.
Assessment of matrix effects is presented	Yes
Limit of determination/quantification	Limit of determination was 0.015 µg/L; Limit of quantification was 0.05 µg/L.

Conclusion

According to SANTE/2020/12830 Rev.1 the method was sufficiently validated and it is acceptable as ILV for the primary method for determination of Rimsulfuron in drinking water.

A 2.1.2.4.1.3 Confirmatory method (if required)

No confirmatory method is required

A 2.1.2.5 Description of Methods for the Analysis of Air (KCP 5.2)

No new or additional studies have been submitted

A 2.1.2.6 Description of Methods for the Analysis of Body Fluids and Tissues (KCP 5.2)

No new or additional studies have been submitted

A 2.1.2.7 A.2.A.9 Other Studies/ Information

No new or additional studies have been submitted

A 2.2 Analytical methods for Nicosulfuron

A 2.2.1 Methods used for the generation of pre-authorization data (KCP 5.1)

No new or additional studies have been submitted

A 2.2.2 Methods for post-authorization control and monitoring purposes (KCP 5.2)

A 2.2.2.1 Description of analytical methods for the determination of residues in plant matrices (KCP 5.2)

No new or additional studies have been submitted

A 2.2.2.2 Description of analytical methods for the determination of residues in animal matrices (KCP 5.2)

No new or additional studies have been submitted

A 2.2.2.3 Description of Methods for the Analysis of Soil (KCP 5.2)

No new or additional studies have been submitted

A 2.2.2.4 Description of Methods for the Analysis of Water (KCP 5.2)

No new or additional studies have been submitted

A 2.2.2.5 Description of Methods for the Analysis of Air (KCP 5.2)

No new or additional studies have been submitted

A 2.2.2.6 Description of Methods for the Analysis of Body Fluids and Tissues (KCP 5.2)

No new or additional studies have been submitted

A 2.2.2.7 A.2.A.9 Other Studies/ Information

No new or additional studies have been submitted