Renewal Assessment Report

Dimethenamid-P

Volume 3 – B.5 Methods of analysis

Rev. 0 - 10 August 2016

Rapporteur Member State: Germany

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Version history

When	What
10 August 2016	First version submitted to EFSA

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B Summary of the data and information

B.5 Methods of analysis

B.5.1 Methods used for the generation of pre-approval data

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

B.5.1.1.1 Methods for the determination of pure active substance in the active substance as manufactured

Reference:
Nemitz and Genari (2013), Determination of active ingredient S-dimethenamid and its isomer R-dimethenamid in dimethenamid-P technical grade active ingredient (TGAI) by means of HPLC, BASF 2013/1066432; APL0665/01, BASF (BVL no 2630042)

Sonnenschein (2013), Validation of the analytical method APL0665/01: Determination of S-dimethenamid and its isomer R-dimethenamid in dimethenamid-P technical grade active ingredient (BAS 656 H) by means of high performance liquid chromatography (HPLC), BASF 2013/1066433; VP 063/2013, BASF (BVL no 2630044)

Principle of the method:
After homogenisation, the technical material is dissolved in *n*-heptane/2-propanol (97:3, v/v). Dimethenamid-P is determined by HPLC-UV using external standard calibration. It should be noted that dimethenamid-P produces a double peak in the chromatogram due to the restricted rotation around the thiophene ring-nitrogen bond. For quantification the peak area of both peaks has to be summed up.

Column: Chiral HPLC column, Regis (S,S) WHELK-01, 250 mm x 4.6 mm, 5 µm
Mobile phase: *n*-heptane / 2-propanol / THF, 97:2:1 (v/v/v)
Detector wavelength: 237 nm

Findings:

Table B.5.1-1: Validation data for the determination of dimethenamid-P in the technical material

	Linearity (linear between), Corr. Coeff. (n = 12)	**Precision - repeatability (%RSD)**	**Interference**
Dimethenamid-P	1562 – 2459 mg/L 0.9988	0.98	None

The specificity of the method was demonstrated by retention time match and by comparison of the UV spectra with reference standard.

Conclusion:
The method is acceptably validated and allows the determination of dimethenamid-P in the technical material.

CIPAC method:
No existing CIPAC method was found to be applicable for analysis of the active substance in the technical material.

B.5.1.1.2 Methods for the determination of relevant impurities in the active substance as manufactured

See Volume 4.

B.5.1.2 Methods for risk assessment

B.5.1.2.1 Analytical methods for soil, water, sediment, air and any additional matrices used in support of environmental fate studies

Air

The method below was not explicitly submitted for this point, but is considered appropriate here.

Method 1

Data point:	KCA 4.1.2/6
Author/year:	Zangmeister, W., 2010
Title/report number:	Validation of analytical method L0167/01: Determination of Dimethenamid-P in air, Doc. ID 2010/1126085, Study Code: 391445, ASB2013-9757

Conclusion

Although submitted by the applicant as a method for risk assessment, the method is considered a monitoring method as well (for a detailed description see B.5.2.5.1). Formally, the method is considered acceptable in accordance with SANCO/3029/99 rev 4. The validated limit of detection is 1.5 µg/m^3. However, it remains unclear for which particular endpoints and/or studies the method was used for.

Methods which do not fulfil the requirements

Table B.5.1-2: List of methods, which do not fulfil requirements

Author(s) and year	Report No	Reason
Kettner & Karapally (1994)	KCA 4.1.2 94/10638 MET9700263	The number of fortified samples per level (n=3) is not sufficient according to SANCO/3029/99 rev 4.

B.5.1.2.2 Analytical methods for soil, water, and any additional matrices used in support of efficacy studies

Particular methods for the determination of dimethenamid-P in efficacy studies were neither provided by the applicant, nor identified by the RMS.

B.5.1.2.3 Analytical methods for feed, body fluids and tissues, air and any additional matrices used in support of toxicological studies

Particular methods for the determination of dimethenamid-P and/or its metabolites in toxicological studies, including feed analysis, were not provided. Moreover, toxicological endpoints used for the derivation of ADI, ARfD and AOEL values were not determined by analytical techniques requiring assessment.

B.5.1.2.4 Analytical methods for body fluids, air and any additional matrices used in support of operator, worker, bystander and resident exposure studies

Particular methods for the determination of dimethenamid-P and/or its metabolites in operator or worker exposure studies were neither provided by the applicant, nor identified by the RMS.

B.5.1.2.5 Analytical methods for plants, plant products, processed food commodities, food of plant and animal origin, feed and any additional matrices used in support of residue studies

The residue definition for risk assessment in plant matrices used in this RAR does not comply with the previous residue definition used in the original DAR (2000, ASB2010-10566), which was prepared by Germany. This was considered in the re-evaluation of methods for risk assessment.
Analytical methods used in animal feeding studies were not provided and not considered necessary, because metabolism studies have shown that significant residues do not occur when crops with incurred residues resulting from the intended uses are fed to animals.

Method 1, BASF method L0179/02 (method used in field trials)

Data point:	KCA 4.1.7 and KCA 4.2/1
Author/year:	Lehmann, A., 2012
Title/report number:	Validation of BASF method L0179/02: Method for the determination of dimethenamid-P (BAS 656 H) and its metabolites M23, M26, M27 and M30 in plant matrices, Doc ID 2011/1182078, ASB2014-3702

Conclusion

The applicant submitted the method as both, a method for risk assessment and monitoring (for a detailed description see B.5.2.1.1). Formally, the method is considered acceptable in accordance with SANCO/3029/99 rev 4. The validated limit of detection is 0.01 mg/kg per analyte. The method was applied for all field trials considered acceptable (see B.7.3).

Methods which do not fulfil the requirements

Table B.5.1-3: List of methods, which do not fulfil requirements

Author(s) and year	Report No	Reason
Fegert & Mackenroth (1999)	KCA 4.1.2 99/10004 MET1999-753	GC-MSD was not used in the field trials considered in the risk assessment.
Smith & Bade (1991)	KCA 4.1.2 91/11820 MET1999-745	The study is not validated according to SANCO/3029/99 rev. 4. Also, GC-MSD was not used in the field trials considered in the risk assessment.
Bourry & Hertl (1991)	KCA 4.1.2 91/11840 MET1999-750	The study is not validated according to SANCO/3029/99 rev. 4. Also, GC-NPD was not used in the field trials considered in the risk assessment.
Bourry & Hertl (1991)	91/11824 MET1999-751	Insufficient LOQ (0.02 mg/kg). GC-NPD and GC-MSD were not used in the field trials considered in the risk assessment.
Bade (1991)	KCA 4.1.2 91/11839 MET1999-752	The study is not validated according to SANCO/3029/99 rev. 4. Also, GC-NPD was not used in the field trials considered in the risk assessment.
Raunft & Benz (1999)	KCA 4.1.2 99/100005 MET1999-754	Field residue trial with insufficient validation. The number of fortified samples per level (n = 1-2) is not sufficient according to SANCO/3029/99 rev. 4. Also, GC-MSD was not used in the field trials considered in the risk assessment.
Meumann & Benz (1999)	KCA 4.1.2 99/10006 MET1999-755	Field residue trial with insufficient validation. The number of fortified samples per level (n = 1-2) is not sufficient according to SANCO/3029/99 rev. 4. Also, GC-MSD was not used in the field trials considered in the risk assessment.
Schulz (1999)	KCA 4.1.2 99/10007 MET1999-756	Field residue trial with insufficient validation. The number of fortified samples per level (n = 1-2) is not sufficient according to SANCO/3029/99 rev. 4. Also, GC-MSD was not used in the field trials considered in the risk assessment.
Weeren & Schmidt (1995)	KCA 4.1.2 95/10127 MET9700261	The number of fortified samples per level (n = 2) is not sufficient according to SANCO/3029/99 rev. 4. Also, GC-MSD was not used in the field trials considered in the risk assessment.
Anonymous (1999)	KCA 4.1.2 98/11530 MET2005-637	Compilation of active substances determinable by DFG S19. Does not contain validation data.
Bourry & Hertl (1993)	KCA 4.1.2 93/11481 MET9700262	The number of fortified samples per level (n = 1-2) is not sufficient according to SANCO/3029/99 rev. 4.

B.5.1.2.6 Analytical methods for soil, water, sediment, feed and any additional matrices used in support of ecotoxicological studies

Soil

The methods below were not explicitly submitted for this point, but are considered appropriate here.

Method 1, BASF Method L0109/01

Data point: KCA 4.1.2/2

Author/year: Obermann, M., 2008

Title/report number: Validation of Analytical Method L0109/01: Determination of dimethenamid-P and its Metabolites Reg.No. 360714 and Reg.No. 360715 in Soil using HPLC/MS-MS, Doc No. 2008/1042152, study code 148916, ASB2010-4519

Method 2, BASF Method L0109/02

Data point: KCA 4.1.2/1

Author/year: Tilting, N., 2014

Title/report number: Validation of analytical method L0109/02: Determination of dimethenamid-P (BAS 656 H) and its metabolites Reg No. 360715 (M23), Reg No. 360714 (M27) and Reg No. 360712 (M31) in soil and sediment by HPLC/MS-MS, Doc ID 2013/1110235, Study code 380201 method L0109/02, ASB2014-8294

Conclusion

Although submitted by the applicant as methods for risk assessment, these methods are considered monitoring methods as well (for a detailed description see B.5.2.3.1). Formally, the methods are considered acceptable in accordance with SANCO/3029/99 rev 4. However, it remains unclear for which particular endpoints and/or studies the methods were used for.

Water

The methods below were not explicitly submitted for this point, but are considered appropriate here.

Method 1

Data point: KCA 4.1.2/3

Author/year: Jooß, S., 2012

Title/report number: Determination of dimethenamid-P and its metabolites M23, M27 and M31 in water, Doc. ID 2012/1278546, Study code: 2711 G, 353459, ASB2014-8295

Method 2, BASF Method 519/0

Data point: KCA 4.1.2/5

Author/year: Schulz, H.; Meyer, M., 2007

Title/report number: Determination of Dimethenamid-P and its Metabolites M23 and M27 in Tap and Surface Water - Validation of the Method 519/0, Doc ID 2007/1054384, Study No.148913, ASB2010-4520

Method 3

Data point: KCA 4.1.2/4

Author/year: Mewis, A., 2013

Title/report number: Validation of an analytical method for determination of metabolites of dimethenamid-P in water, Doc. ID 2013/1349800, Study code: S13-03461, Study ID 703063, ASB2014-8296

Conclusion

Although submitted by the applicant as methods for risk assessment, these methods are considered monitoring methods as well (for a detailed description see B.5.2.4.1 and B.5.2.4.3). Formally, the methods are considered acceptable in accordance with SANCO/3029/99 rev 4. However, it remains unclear for which particular endpoints and/or studies the methods were used for.

Methods which do not fulfil the requirements

Table B.5.1-4: List of methods, which do not fulfil requirements

Author(s) and year	Report No	Reason
Bourry & Hertl (1995)	KCA 4.1.2 95/10124 MET9700259	No GLP stated. The number of fortified samples at LOQ level is not sufficient according to SANCO/3029/99 rev 4.
Bourry & Hertl (1991)	KCA 4.1.2 91/11840 MET1999-750	The number of levels and the fortified samples per level is not sufficient according to SANCO/3029/99 rev 4.
Bourry & Hertl (1991)	KCA 4.1.2 91/11824 MET1999-751	The number of fortified samples per level is not sufficient according to SANCO/3029/99 rev 4.
Bade (1991)	KCA 4.1.2 91/11839 MET1999-752	The number of fortified samples per level is not sufficient according to SANCO/3029/99 rev 4.
Gasser (1998)	KCA 4.1.2 98/10385 MET1999-785	The number of fortified samples per level is not sufficient according to SANCO/3029/99 rev 4.
MacGregor & Markley (1996)	97/5256 ASB2014-5111	The number of fortified samples per level is not sufficient according to SANCO/3029/99 rev 4.
Mayer (1994)	94/11279 MET1999-783	The number of fortified samples per level is not sufficient according to SANCO/3029/99 rev 4.
Smith & Bade (1991)	91/11837 MET9700260	No GLP stated. Test matrix used is deionised water, which is not considered acceptable due to the lack of ions and dissolved organic carbon

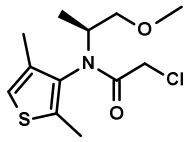
B.5.1.2.7 Analytical methods for water, buffer solutions, organic solvents and any additional matrices used in the physical and chemical properties test

Particular methods for the determination of dimethenamid-P and/or its metabolites used in the physical and chemical properties test were not provided.

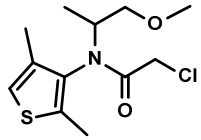
B.5.2 Methods for post-approval control and monitoring purposes

Information on the active substance and further analytes. As dimethenamid-P is the *S*-isomer of the racemic mixture dimethenamid, methods for the latter are applicable for dimethenamid-P as well.

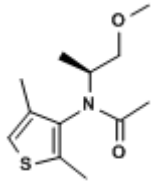
Name, code	Dimethenamid-P, BAS 656 H, Reg. No. 363851, CAS No 163515-14-8
IUPAC	S-2-chloro-N-(2,4-dimethyl-3-thienyl)-N-(2-methoxy-1-methylethyl)-acetamide
Formula	$C_{12}H_{18}ClNO_2S$
Molecular Weight	275.79 g mol^{-1}



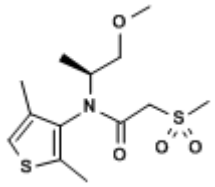
Name, code	Dimethenamid, SAN-582H, CAS No 87674-68-8
IUPAC	(RS)-2-chloro-N-(2,4-dimethyl-3-thienyl)-N-(2-methoxy-1-methylethyl)-acetamide
Formula	$C_{12}H_{18}ClNO_2S$
Molecular Weight	275.79 g mol^{-1}



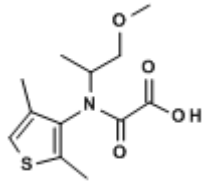
Name, code	M3
IUPAC	N-(2,4-dimethylthiophen-3-yl)-N-[(2S)-1-methoxypropan-2-yl]acetamide
Formula	$C_{12}H_{19}NO_2S$
Molecular Weight	241.35 g mol^{-1}



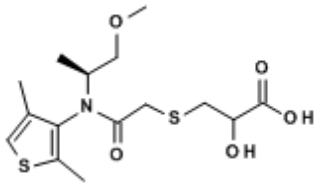
Name, code	M10
IUPAC	N-(2,4-dimethyl-3-thienyl)-N-[(1S)-2-methoxy-1-methyl-ethyl]-2-methylsulfonyl-acetamide
Formula	$C_{13}H_{21}NO_4S_2$
Molecular Weight	319.44 g mol^{-1}



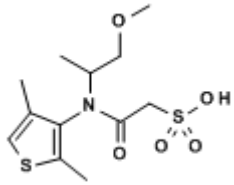
Name, code	M23, Reg. No. 360715
IUPAC	N-(2,4-dimethyl-3-thienyl)-N-(2-methoxy-1-methylethyl)-oxalamic acid (Oxalamid)
Formula	$C_{12}H_{17}NO_4S$
Molecular Weight	271.33 g mol^{-1}



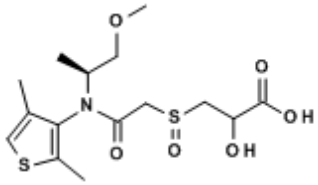
Name, code	M26, Reg. No. 5886781
IUPAC	3-{[(2,4-dimethyl-3-thienyl)-N-(2-methoxy-1-methylethyl)-carbamoyl]-methylsulfanyl}-2hydroxy-propionic acid
Formula	$C_{15}H_{23}NO_5S_2$
Molecular Weight	361.48 g mol^{-1}



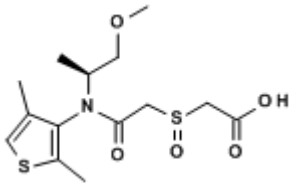
Name, code	M27, Reg. No. 360714
IUPAC	Sodium [(2,4-dimethyl-thiophen-3-yl)-(2-methoxy-1-methylethyl)-carbamoyl] methanesulfonate
Formula	$C_{12}H_{18}NNaO_5S_2$
Molecular Weight	343.40 g mol^{-1}



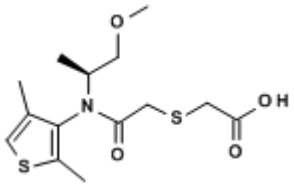
Name, code	M30, Reg. No. 5296352
IUPAC	3-[(2-{(2,4-dimethyl-3-thienyl)[(1S)-2-methoxy-1-methylethyl]-amino}-2-oxoethyl)-lsulfinyl}-2hydroxy-propanonic acid
Formula	$C_{15}H_{23}NO_6S_2$
Molecular Weight	377.48 g mol^{-1}



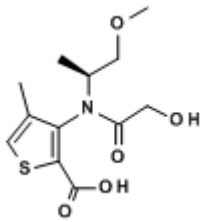
Name, code	M31, Reg. No. 5886777
IUPAC	[[(2,4-dimethyl-thiophen-3-yl)-(2-methoxy-1-methyl-ethyl)-carbamoyl]-methanesulfinyl]-acetic acid
Formula	$C_{14}H_{21}NO_5S_2$
Molecular Weight	347.44 g mol^{-1}



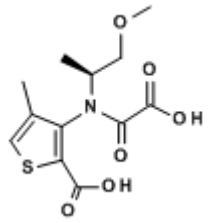
Name, code	M32, Reg. No. 5886785
IUPAC	2-[2-[(2,4-dimethyl-3-thienyl)-[(1S)-2-methoxy-1-methyl-ethyl]amino]-2-oxo-ethyl]sulfanylacetic acid
Formula	$C_{14}H_{21}NO_4S_2$
Molecular Weight	331.45 g mol^{-1}



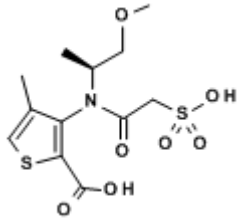
Name, code	M43, Reg. No. 5917262
IUPAC	3-{(hydroxyacetyl)[(2S)-1-methoxypropan-2-yl]amino}-4-methylthiophene-2-carboxylic acid
Formula	$C_{12}H_{17}NO_5S$
Molecular Weight	287.33 g mol^{-1}



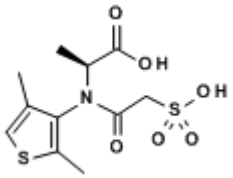
Name, code	M45, Reg. No. 5917261
IUPAC	3-[((1S)-2-methoxy-1-methyl-ethyl)-oxalo-amino]-4-methyl-thiophene-2-carboxylic acid
Formula	$C_{12}H_{15}NO_6S$
Molecular Weight	301.32 g mol^{-1}



Name, code	M47, Reg. No. 5917260
IUPAC	3-{[(2S)-1-methoxypropan-2-yl](sulfoacetyl)amino}-4-methylthiophene-2-carboxylic acid
Formula	$C_{12}H_{17}NO_7S_2$
Molecular Weight	351.40 g mol^{-1}



Name, code	M54
IUPAC	(2S)-2-[(2,4-dimethyl-3-thienyl)-(2-sulfoacetyl)amino]propanoic acid
Formula	$C_{11}H_{15}NO_6S_2$
Molecular Weight	321.37 g mol^{-1}



New residue analytical methods for monitoring are provided. This is justified on the grounds that these methods are required for crop groups (fatty, i.e. sunflower, soybean) previously not considered as representative uses to allow compliance with existing or proposed MRLs.
Furthermore, several methods assessed in the previous DAR do not fulfill the requirements laid down in the most recent guidance document on pesticide residue methods (SANCO/825/00 rev.8.1).
The methods of analysis summarised below are intended to fully replace the methods assessed in the original review.

B.5.2.1 Analytical methods for the determination of residues in or on food and feed of plant origin

B.5.2.1.1 Acceptable methods/reports

Study 1

Data point:	KCA 4.1.7 and KCA 4.2/1
Report:	Validation of BASF method L0179/02: Method for the determination of dimethenamid-P (BAS 656 H) and its metabolites M23, M26, M27 and M30 in plant matrices, Lehmann, A., 2012, Doc ID 2011/1182078, ASB2014-3702
Guideline(s):	Yes (SANCO/825/00 rev. 6, SANCO/3029/99 rev.4, OPPTS 860.1340, Commission Directive 96/46/EC)
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid-P, metabolites M23, M26, M27, M30
Analyte(s) determined as:
Dimethenamid-P, metabolites M23, M26, M27, M30

Principle of the method:
Samples are homogenised in methanol, followed by centrifugation. An aliquot of the supernatant is taken and diluted with water to obtain a methanol/water ratio of 1+1. Sample concentration in final extracts is 0.025 g/mL. The concentrations of all analytes in the final extract are quantified by LC-MS/MS using a Zorbax Eclipse XDB-C18 column. Positive electrospray ionisation is chosen for parent dimethenamid-P and two MRM transitions (m/z 276→244, 276→168) are monitored. For the metabolites M23, M26, M27 and M30 negative electrospray ionisation is chosen and two MRM transitions per analyte were monitored: m/z 320→121, 320→80 (M27), m/z 360→272, 360→142 (M26), m/z 270→198, 270→166 (M23), m/z 376→136, 376→270 (M30).

Results:

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.
Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-1.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-1.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-1 is considered as limit of quantification.
Matrix effects:
Matrix effects were not investigated
Calibration (linearity):
Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points): yes
Accepted calibration range in concentration units: 0.05 - 5 ng/mL

Accepted calibration range in mass fraction units:	0.002 - 0.2 mg/kg
Calibration conducted with matrix matched standards:	no
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid (see Table B.5.2-1).

Conclusion

The analytical method by Lehmann (2012) is suitable as enforcement method for dimethenamid-P and metabolites M23, M26, M27 and M30 in dry crops (starch and protein containing), commodities with high water content, commodities with high acid content and commodities with high oil content. A confirmatory method for dry crops (starch and protein containing), commodities with high water content, commodities with high acid content and commodities with high oil content is provided by full validation of a second MS/MS transition. Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ in all matrices tested.

Table B.5.2-1: Validation of the method by Lehmann (2012) for residues of dimethenamid-P and metabolites M23, M26, M27, M30 in food of plant origin, ASB2014-3702)

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
Dimethenamid-p						
Lehmann (2012) (ASB2014-3702)	maize whole plant	LC-MS/MS, XDB C18, ESI+, m/z 276→244	0.01 0.1	103.9 104.1	1.9 1.2	5 5
	maize seed		0.01 0.1	115.6 107.6	3.0 2.6	5 5
	sugar beet leaves		0.01 0.1	105.3 106.6	1.4 1.3	5 5
	sugar beet roots		0.01 0.1	103.7 105.4	0.5 1.1	5 5
	rape seed		0.01 0.1	105.8 105.9	0.9 1.3	5 5
	strawberries		0.01 0.1	102.1 105.3	1.2 0.8	5 5
	onions		0.01 0.1	104.6 105.4	1.0 1.0	5 5
	dried beans		0.01 0.1	106.6 101.0	2.6 1.0	5 5
	maize whole plant	LC-MS/MS, XDB C18, ESI+, m/z 276→168	0.01 0.1	103.1 103.3	1.9 0.7	5 5
	maize seed		0.01 0.1	115.0 107.0	3.0 3.0	5 5
	sugar beet leaves		0.01 0.1	105.4 107.0	0.8 1.3	5 5
	sugar beet roots		0.01 0.1	104.3 105.4	1.6 0.9	5 5
	rape seed		0.01 0.1	106.4 106.2	1.3 1.7	5 5
	strawberries		0.01 0.1	101.4 105.5	0.2 1.0	5 5
	onions		0.01 0.1	104.1 105.4	1.0 0.9	5 5
	dried beans		0.01 0.1	105.9 100.8	1.4 1.2	5 5
Metabolite M23						
Lehmann (2012) (ASB2014-3702)	maize whole plant	LC-MS/MS, XDB C18, ESI-, m/z 270→198	0.01 0.1	98.3 105.2	5.1 1.5	5 5
	maize seed		0.01 0.1	90.2 88.4	3.6 3.1	5 5
	sugar beet leaves		0.01 0.1	104.2 106.7	1.9 1.6	5 5
	sugar beet roots		0.01 0.1	103.0 103.8	2.3 1.2	5 5

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
	rape seed		0.01 0.1	107.1 106.6	3.9 1.8	5 5
	strawberries		0.01 0.1	99.0 106.2	2.7 1.8	5 5
	onions		0.01 0.1	106.1 105.7	4.8 1.8	5 5
	dried beans		0.01 0.1	99.3 93.0	3.5 1.6	5 5
	maize whole plant	LC-MS/MS, XDB C18, ESI-, m/z 270→166	0.01 0.1	104.4 105.9	3.0 2.0	5 5
	maize seed		0.01 0.1	87.9 87.3	4.6 3.0	5 5
	sugar beet leaves		0.01 0.1	105.5 105.0	4.9 1.7	5 5
	sugar beet roots		0.01 0.1	91.3 100.9	4.3 2.2	5 5
	rape seed		0.01 0.1	107.0 105.4	7.2 1.8	5 5
	strawberries		0.01 0.1	99.8 106.6	5.7 1.7	5 5
	onions		0.01 0.1	105.4 107.6	5.5 1.2	5 5
	dried beans		0.01 0.1	99.4 94.8	5.7 2.3	5 5
Metabolite M26						
Lehmann (2012) (ASB2014-3702)	maize whole plant	LC-MS/MS, XDB C18, ESI-, m/z 360→272	0.01 0.1	96.7 107.0	3.4 1.5	5 5
	maize seed		0.01 0.1	87.2 94.9	6.1 1.6	5 5
	sugar beet leaves		0.01 0.1	104.8 107.5	1.7 2.4	5 5
	sugar beet roots		0.01 0.1	101.0 108.9	3.2 2.6	5 5
	rape seed		0.01 0.1	102.4 106.0	2.0 2.0	5 5
	strawberries		0.01 0.1	104.9 106.0	2.1 1.3	5 5
	onions		0.01 0.1	97.2 113.7	6.7 4.1	5 5
	dried beans		0.01 0.1	98.6 107.6	2.0 3.2	5 5
	maize whole plant	LC-MS/MS, XDB C18, ESI-, m/z 360→142	0.01 0.1	95.1 105.2	7.8 3.2	5 5
	maize seed		0.01 0.1	86.3 95.2	3.6 2.6	5 5
	sugar beet		0.01	103.2	4.1	5

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
	leaves		0.1	107.5	1.6	5
	sugar beet roots		0.01 0.1	102.2 109.8	4.0 0.9	5 5
	rape seed		0.01 0.1	106.8 106.3	2.5 2.8	5 5
	strawberries		0.01 0.1	101.3 106.7	3.4 3.0	5 5
	onions		0.01 0.1	89.5 109.8	6.0 3.1	5 5
	dried beans		0.01 0.1	105.2 106.6	3.4 3.5	5 5
Metabolite M27						
Lehmann (2012) (ASB2014-3702)	maize whole plant	LC-MS/MS, XDB C18, ESI-, m/z 320→121	0.01 0.1	104.8 104.5	3.8 1.1	5 5
	maize seed		0.01 0.1	83.4 82.6	1.7 2.0	5 5
	sugar beet leaves		0.01 0.1	106.2 105.7	1.9 1.5	5 5
	sugar beet roots		0.01 0.1	101.8 104.4	1.1 1.1	5 5
	rape seed		0.01 0.1	103.0 101.7	2.7 1.9	5 5
	strawberries		0.01 0.1	102.9 105.2	2.0 1.3	5 5
	onions		0.01 0.1	103.3 104.1	2.3 0.2	5 5
	dried beans		0.01 0.1	99.2 97.3	2.5 1.7	5 5
	maize whole plant	LC-MS/MS, XDB C18, ESI-, m/z 320→80	0.01 0.1	105.2 105.6	3.4 0.7	5 5
	maize seed		0.01 0.1	84.0 82.2	2.8 2.2	5 5
	sugar beet leaves		0.01 0.1	104.6 106.7	2.4 1.3	5 5
	sugar beet roots		0.01 0.1	105.0 105.2	1.8 1.2	5 5
	rape seed		0.01 0.1	103.1 101.5	0.9 1.8	5 5
	strawberries		0.01 0.1	101.7 105.0	1.8 1.2	5 5
	onions		0.01 0.1	104.0 105.0	2.0 1.5	5 5
	dried beans		0.01 0.1	98.9 97.2	2.6 1.5	5 5

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
Metabolite M30						
Lehmann (2012) (ASB2014-3702)	maize whole plant	LC-MS/MS, XDB C18, ESI-, m/z 376→136	0.01 0.1	108.2 102.4	8.5 2.6	5 5
	maize seed		0.01 0.1	83.5 86.4	5.4 1.3	5 5
	sugar beet leaves		0.01 0.1	103.4 103.3	7.8 0.9	5 5
	sugar beet roots		0.01 0.1	105.8 104.6	4.9 1.8	5 5
	rape seed		0.01 0.1	96.5 101.8	10.6 1.9	5 5
	strawberries		0.01 0.1	93.6 105.6	6.8 2.5	5 5
	onions		0.01 0.1	108.5 104.0	2.3 1.9	5 5
	dried beans		0.01 0.1	94.0 93.6	6.9 2.2	5 5
	maize whole plant	LC-MS/MS, XDB C18, ESI-, m/z 376→270	0.01 0.1	98.5 102.8	7.4 2.6	5 5
	maize seed		0.01 0.1	89.0 86.5	7.2 1.2	5 5
	sugar beet leaves		0.01 0.1	92.7 104.6	6.9 1.8	5 5
	sugar beet roots		0.01 0.1	101.8 104.3	4.0 2.0	5 5
	rape seed		0.01 0.1	100.6 99.7	11.1 3.4	5 5
	strawberries		0.01 0.1	99.4 105.6	5.7 1.8	5 5
	onions		0.01 0.1	119.1 102.4	3.5 2.1	5 5
	dried beans		0.01 0.1	92.5 91.6	4.0 2.4	5 5

Study 2

Independent laboratory validation (ILV) of the method by Lehmann (2012, ASB2014-3702)

Data point:	KCA 4.1.2/8 and KCA 4.2/2
Report:	Rogers, P.; Fiorito, B.; Shi, Y., 2014, Independent laboratory validation of BASF analytical method L0179/02: "Method for the determination of dimethenamid-P (BAS 656 H, Reg. No. 363851), M23 (Reg. No. 360715), M26 (Reg. No. 360716), M27 (Reg. No. 360714) and M30 (Reg. No. 5296352) in plant matrices", Doc ID 2013/7002656, Study No. 390388, ASB2014-8333
Guideline(s):	Yes (SANCO/825/00 rev. 8.1, ENV/JMMONO(2007)17, OPPTS 860.1340)
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid-P, metabolites M23, M26, M27, M30
Analyte(s) determined as:
Dimethenamid-P, metabolites M23, M26, M27, M30

Principle of the method:
The study refers to a method by Dolich (2012), which was not submitted by the applicant. Based on the summary is seems to be the same method as in the study of Lehmann (2012, ASB2014-3702). Except from the use of matrix matched standards, no details and deviations are reported.

Results:

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.
Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-2.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-2.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-2 is considered as limit of quantification.
Matrix effects:
Matrix effects were not investigated
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	yes
Accepted calibration range in concentration units:	0.05-10 ng/mL
Accepted calibration range in mass fraction units:	0.002-0.4 mg/kg
Calibration conducted with matrix matched standards:	yes
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid (see Table B.5.2-2).

Conclusion

The study refers to a method by Dolich (2012), which was not submitted by the applicant. Nevertheless, the study is considered as an independent laboratory validation of the method described by Lehmann (2012, ASB2014-3702) for dimethenamid-P and the metabolites M23, M26, M27 and M30 in dry crops (starch and protein containing), commodities with high water content, commodities with high acid content and commodities with high oil content. However, the ILV lacks a detailed method description including instrumental parameters and possible deviations. Also no explanation was given regarding the occurrence of signal enhancement of up to 300 %, requiring matrix matched calibration.

Table B.5.2-2: Validation of the independent laboratory validation by Rogers (2014) for residues of dimethenamid-P and metabolites M23, M26, M27, M30 in food of plant origin, ASB2014-8333)

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
Dimethenamid-P						
Rogers (2014) (ASB2014-8333)	strawberries	LC-MS/MS, C18 column, ESI+, m/z 276→244	0.01 0.1	95 96	2.9 2.2	5 5
	dried beans		0.01 0.1	85 87	2.3 3.5	5 5
	rape seed		0.01 0.1	105 103	13.1 12.0	5 5
	maize forage		0.01 0.1	108 102	2.3 2.0	5 5
	maize seed		0.01 0.1	113 104	4.2 1.5	5 5
	strawberries	LC-MS/MS, C18 column, ESI+, m/z 276→168	0.01 0.1	98 97	3.4 2.8	5 5
	dried beans		0.01 0.1	87 87	3.3 2.4	5 5
	rape seed		0.01 0.1	107 103	13.1 11.7	5 5
	maize forage		0.01 0.1	106 101	2.0 1.6	5 5
	maize seed		0.01 0.1	109 105	3.2 1.6	5 5
Metabolite M23						
Rogers (2014) (ASB2014-8333)	strawberries	LC-MS/MS, C18 column, ESI-, m/z 270→198	0.01 0.1	102 102	2.1 1.5	5 5
	dried beans		0.01 0.1	72 74	4.1 4.3	5 5
	rape seed		0.01 0.1	104 104	4.6 5.5	5 5
	maize forage		0.01 0.1	109 107	5.1 1.7	5 5
	maize seed		0.01 0.1	101 99	3.2 1.8	5 5
	strawberries	LC-MS/MS, C18 column, ESI-, m/z 270→166	0.01 0.1	101 101	7.1 1.5	5 5
	dried beans		0.01 0.1	73 75	10.2 6.0	5 5
	rape seed		0.01 0.1	109 99	2.4 3.9	5 5
	maize forage		0.01 0.1	113 105	5.6 3.8	5 5
	maize seed		0.01 0.1	105 97	3.4 1.2	5 5

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
Metabolite M26						
Rogers (2014) (ASB2014-8333)	strawberries	LC-MS/MS, C18 column, ESI+, m/z 360→272	0.01 0.1	107 101	2.1 1.0	5 5
	dried beans		0.01 0.1	71 74	5.9 8.0	5 5
	rape seed		0.01 0.1	109 107	7.1 9.0	5 5
	maize forage		0.01 0.1	103 104	5.6 1.5	5 5
	maize seed		0.01 0.1	103 99	2.8 2.5	5 5
	strawberries	LC-MS/MS, C18 column, ESI+, m/z 360→142	0.01 0.1	107 99	5.1 1.4	5 5
	dried beans		0.01 0.1	68 71	8.8 7.3	5 5
	rape seed		0.01 0.1	108 106	10.1 9.4	5 5
	maize forage		0.01 0.1	103 100	4.9 1.5	5 5
	maize seed		0.01 0.1	104 99	4.3 1.9	5 5
Metabolite M27						
Rogers (2014) (ASB2014-8333)	strawberries	LC-MS/MS, C18 column, ESI+, m/z 320→121	0.01 0.1	102 100	2.0 1.9	5 5
	dried beans		0.01 0.1	71 74	4.3 6.4	5 5
	rape seed		0.01 0.1	99 106	1.9 1.3	5 5
	maize forage		0.01 0.1	109 111	2.4 0.8	5 5
	maize seed		0.01 0.1	102 100	1.5 1.8	5 5
	strawberries	LC-MS/MS, C18 column, ESI+, m/z 320→80	0.01 0.1	103 102	1.7 1.5	5 5
	dried beans		0.01 0.1	74 74	4.6 7.1	5 5
	rape seed		0.01 0.1	102 106	2.9 3.2	5 5
	maize forage		0.01 0.1	109 112	3.8 0.6	5 5
	maize seed		0.01 0.1	102 99	3.2 1.0	5 5
Metabolite M30						
Rogers (2014) (ASB2014-	strawberries	LC-MS/MS, C18 column,	0.01 0.1	104 101	8.9 2.4	5 5

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
8333)	dried beans	ESI+, m/z 376→136	0.01 0.1	66 68	9.8 9.0	5 5
	rape seed		0.01 0.1	103 108	7.8 3.9	5 5
	maize forage		0.01 0.1	111 100	4.9 1.2	5 5
	maize seed		0.01 0.1	95 94	5.9 4.3	5 5
	strawberries	LC-MS/MS, C18 column, ESI+, m/z 376→270	0.01 0.1	105 106	11.9 0.5	5 5
	dried beans		0.01 0.1	73 68	7.5 7.2	5 5
	rape seed		0.01 0.1	94 104	5.8 5.0	5 5
	maize forage		0.01 0.1	115 101	8.5 2.3	5 5
	maize seed		0.01 0.1	98 93	9.9 2.9	5 5

Study 3

Data point: KCA 4.2/3

Report: Diamaduros, B., 2014, Validation of BASF analytical method R0038/01: "Analytical method (Modified QuEChERS) for the determination of the residues of dimethenamid-P (Reg. No. 363851) and metabolites M26 (Reg. No. 360716) and M30 (Reg. No. 5296352) in plant matrices at a LOQ of 0.01 mg/kg using LC-MS/MS", Doc ID 2013/7002627, Study No. 718508, ASB2014-8334

Guideline(s): Yes (SANCO/825/00 rev. 8.1, ENV/JMMONO(2007)17, OPPTS 860.1340)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid-P, metabolites M26, M30
Analyte(s) determined as:
Dimethenamid-P, metabolites M26, M30

Principle of the method:
Samples are determined according to EN 15662:2008 (QuEChERS method): The sample material is extracted by shaking with acetonitrile and water (added water depends on the water amount in sample). Separation of the water and acetonitrile phases is obtained by addition of sodium citrate, sodium hydrogencitrate sesquihydrate, magnesium sulfate and sodium chloride. Samples are centrifuged (optional freezing out of an aliquot of the acetonitrile phase) and an aliquot of the supernatant is diluted with methanol water (50 + 50, v/v). Sample concentration in final extracts is

0.001 or 0.01 g/mL. The concentrations of all analytes in the final extracts are quantified by LC-MS/MS using an Atlantis T3 C18 UPLC column. Positive electrospray ionisation is set for parent dimethenamid-P and two MRM transitions (m/z 276→244, 276→168) are monitored. For the metabolites M26 and M30 negative electrospray ionisation is set and two MRM transitions per analyte are monitored: m/z 360→272, 360→142 (M26), m/z 376→91, 376→136 (M30).

Results:

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected except for metabolite M30 in soybean (54 %)
Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-3. For the metabolites M26 and M30 recoveries were not acceptable in fatty and dry matrices.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-3.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-3 is considered as limit of quantification.
Matrix effects:
Significant matrix effects (>20 %) were not observed.
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	yes
Accepted calibration range in concentration units:	0.01 - 0.5 ng/mL
Accepted calibration range in mass fraction units:	0.001 - 0.5 mg/kg
Calibration conducted with matrix matched standards:	no
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid except for the metabolites M26 and M30 where recoveries were not acceptable in fatty and dry matrices (see Table B.5.2-3).

Conclusion

The analytical method by Diamaduros (2014, ASB2014-8334) is suitable as enforcement method for dimethenamid-P in dry crops (starch and protein containing), commodities with high water content, commodities with high acid content and commodities with high oil content. For the metabolites M26 and M30 recoveries were not acceptable in dry bean and soybean seed for both, the primary and confirmatory transition. A confirmatory method for dry crops (starch and protein containing), commodities with high water content, commodities with high acid content and commodities with high oil content is provided by full validation of a second MS/MS transition. Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ, with the exception of metabolite M30 in soybean (54 %).
As the study by Diamaduros (2014, ASB2014-8334) was not considered as an enforcement method by the applicant, no independent laboratory validation was performed.

Table B.5.2-3: Validation of the method by Diamaduros (2014, ASB2014-8334) for residues of dimethenamid-P and metabolites M26 and M30 in food of plant origin)

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
Dimethenamid-p						
Diamaduros (2014) (ASB2014-8334)	grape	LC-MS/MS, ESI+, Atlantis T3 C18 column, m/z 276→244	0.01 0.1	91 93	3 3	5 5
	dry bean		0.01 0.1	93 97	1 2	5 5
	lettuce		0.01 0.1	91 95	5 1	5 5
	soybean seed		0.01 0.1	81 87	4 4	5 5
	barley grain		0.01 0.1	89 93	3 4	5 5
	grape	LC-MS/MS, ESI+, Atlantis T3 C18 column, m/z 276→168	0.01 0.1	93 92	3 5	5 5
	dry bean		0.01 0.1	92 91	4 3	5 5
	lettuce		0.01 0.1	90 93	2 5	5 5
	soybean seed		0.01 0.1	78 87	3 5	5 5
	barley grain		0.01 0.1	95 93	2 3	5 5
Metabolite M26						
Diamaduros (2014) (ASB2014-8334)	grape	LC-MS/MS, ESI+, Atlantis T3 C18 column, m/z 360→272	0.01 0.1	86 92	4 5	5 5
	dry bean		0.01 0.1	68 68	4 10	5 5
	lettuce		0.01 0.1	88 95	4 7	5 5
	soybean seed		0.01 0.1	55 64	11 3	5 5
	barley grain		0.01 0.1	92 88	9 8	5 5
	grape	LC-MS/MS, ESI+, Atlantis T3 C18 column, m/z 360→142	0.01 0.1	88 92	3 5	5 5
	dry bean		0.01 0.1	66 67	3 10	5 5
	lettuce		0.01 0.1	85 92	4 8	5 5
	soybean seed		0.01 0.1	58 65	10 3	5 5
	barley grain		0.01 0.1	86 84	8 7	5 5

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
Metabolite M30						
Diamaduros (2014) (ASB2014-8334)	grape	LC-MS/MS, ESI+, Atlantis T3 C18 column, m/z 376→91	0.01 0.1	69 78	3 4	5 5
	dry bean		0.01 0.1	39 31	4 13	5 5
	lettuce		0.01 0.1	73 78	6 6	5 5
	soybean seed		0.01 0.1	74 32	11 12	5 5
	barley grain		0.01 0.1	68 69	14 8	5 5
	grape	LC-MS/MS, ESI+, Atlantis T3 C18 column, m/z 376→136	0.01 0.1	67 78	6 4	5 5
	dry bean		0.01 0.1	35 29	6 13	5 5
	lettuce		0.01 0.1	73 81	6 6	5 5
	soybean seed		0.01 0.1	20 23	16 9	5 5
	barley grain		0.01 0.1	66 68	14 10	5 5

B.5.2.1.2 Extraction efficiency of analytical methods used for samples of plant origin

As the mean residue of dimethenamid-P and metabolite M30 in maize grain, soybean, sunflower seed sugar beet root and oilseed rape seed samples from supervised field trials were determined at <0.01 mg/kg, the evidence of sufficient extraction efficiency is not needed.

B.5.2.2 Analytical methods for the determination of residues in or on food and feed of animal origin

B.5.2.2.1 Acceptable methods/reports

Study 1

Data point: KCA 4.2/4

Report: Validation of BASF analytical method R0037/01: "Analytical method for the determination of the residues of dimethenamid-P (Reg. No. 363851) and metabolites M26 (Reg. No. 360716) and M30 (Reg. No. 5296352) in animal matrices at a LOQ of 0.01 mg/kg using LC-MS/MS", Gordon, B., 2014, Doc No. 2013/7002631, Study No. 718604, method R0037/01, ASB2014-8335

Guideline(s): Yes (SANCO/825/00 rev. 8.1, ENV/JMMONO(2007)17, OPPTS

	860.1340)
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid-P, metabolites M26, M30
Analyte(s) determined as:
Dimethenamid-P, metabolites M26, M30

Principle of the method:
Samples are homogenised (milk is shaken) with acetonitrile/water (1 + 1, v/v), followed by centrifugation. An aliquot of the supernatant is taken and diluted with methanol/water (1 + 1, v/v). Sample concentration in final extracts is 0.0005 or 0.002 g/mL. The concentrations of all analytes in the final extract are quantified by LC-MS/MS using an Atlantis T3 C18 UPLC column. Positive electrospray ionisation is chosen for parent dimethenamid-P and two MRM transitions (m/z 276→244, 276→168) are monitored. For the metabolites M26 and M30 negative electrospray ionisation is set and two MRM transitions per analyte are monitored: m/z 360→272, 360→142 (M26), m/z 376→91, 376→136 (M30).

Findings

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.

Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-4.

Repeatability (precision):
For repeatability of analytical results see Table B.5.2-4.

Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-4 is considered as limit of quantification.

Matrix effects:
Significant matrix effects (>20 %) were observed in milk for dimethenamid-P up to 37 %, M26 up to 25 % and M30 up to 24 %.

Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	yes
Accepted calibration range in concentration units:	0.004 - 0.2 ng/mL
Accepted calibration range in mass fraction units:	0.002 - 0.4 mg/kg
Calibration conducted with matrix matched standards:	no
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid (see Table B.5.2-4).

Conclusion

The analytical method by Gordon (2014, ASB2014-8335) would be suitable as enforcement method for dimethenamid-P and metabolites M26 and M30 in milk, meat, fat, liver, kidney and eggs.
A confirmatory method for milk, meat, fat, liver, kidney and eggs is provided by full validation of a second MS/MS transition.
Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ in all matrices tested.

Table B.5.2-4: Validation of the method by Gordon (2014) for residues in food of animal origin, ASB2014-8335

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
Dimethenamid-p						
Gordon (2014) (ASB2014-8335)	muscle	LC-MS/MS, ESI+, Atlantis T3 C18 column, m/z 276→244	0.01 0.1	91 91	11 8	5 5
	kidney		0.01 0.1	96 103	5 7	5 5
	liver		0.01 0.1	88 95	8 2	5 5
	fat		0.01 0.1	100 99	2 4	5 5
	milk		0.01 0.1	83 101	3 4	5 5
	egg		0.01 0.1	98 99	3 4	5 5
	muscle	LC-MS/MS, ESI+, Atlantis T3 C18 column, m/z 276→168	0.01 0.1	89 91	8 9	5 5
	kidney		0.01 0.1	87 100	15 7	5 5
	liver		0.01 0.1	94 96	8 4	5 5
	fat		0.01 0.1	98 99	3 5	5 5
	milk		0.01 0.1	81 92	7 6	5 5
	egg		0.01 0.1	103 96	6 5	5 5
M26						
Gordon (2014) (ASB2014-8335)	muscle	LC-MS/MS, ESI-, Atlantis T3 C18 column, m/z 360→272	0.01 0.1	103 100	9 8	5 5
	kidney		0.01 0.1	102 102	10 4	5 5
	liver		0.01 0.1	103 102	8 5	5 5
	fat		0.01 0.1	100 105	3 3	5 5
	milk		0.01 0.1	97 108	3 2	5 5
	egg		0.01 0.1	102 102	2 4	5 5
	muscle	LC-MS/MS, ESI-, Atlantis T3 C18 column, m/z 360→142	0.01 0.1	107 97	10 8	5 5
	kidney		0.01 0.1	99 100	16 5	5 5
	liver		0.01	101	9	5

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
			0.1	101	5	5
	fat		0.01 0.1	97 104	6 5	5 5
	milk		0.01 0.1	94 107	3 5	5 5
	egg		0.01 0.1	102 103	3 5	5 5
M30						
Gordon (2014) (ASB2014-8335)	muscle	LC-MS/MS, ESI-, Atlantis T3 C18 column, m/z 376→91	0.01 0.1	104 99	13 5	5 5
	kidney		0.01 0.1	108 103	9 5	5 5
	liver		0.01 0.1	98 99	8 5	5 5
	fat		0.01 0.1	99 104	3 7	5 5
	milk		0.01 0.1	107 104	5 4	5 5
	egg		0.01 0.1	111 99	4 3	5 5
	muscle	LC-MS/MS, ESI-, Atlantis T3 C18 column, m/z 376→136	0.01 0.1	105 96	13 8	5 5
	kidney		0.01 0.1	104 98	12 4	5 5
	liver		0.01 0.1	97 97	9 5	5 5
	fat		0.01 0.1	106 106	5 4	5 5
	milk		0.01 0.1	116 102	3 6	5 5
	egg		0.01 0.1	106 98	2 3	5 5

B.5.2.2.2 Extraction Efficiency of analytical methods used for samples of animal origin

As no residues are expected in animal matrices at this point, the evaluation of extraction efficiency is not required.

B.5.2.2.3 Methods which do not fulfil the requirements

Table B.5.2-5: List of methods, which do not fulfil requirements

Author(s) and year	Report No	Reason
Perez & Patel (2014)	KCA 4.2/5 2013/7002632 ASB2014-8336	The study is submitted as an ILV of the study by Gordon (2014, ASB2014-8335). However, the ion transitions used differed from the one used in the primary method. Here, the $^{13}C1$ peak was used which results in a significantly reduced sensitivity. This is probably the reason for the disappearance of the lower calibration points and the fortifications at 0.01 mg/kg in the background noise. Consequently the ILV is considered not acceptable.

B.5.2.3 Analytical methods for the determination of residues in soil

B.5.2.3.1 Acceptable methods/reports

Study 1

Data point: KCA 4.1.2/2

Report: Validation of Analytical Method L0109/01: Determination of Dimethenamid-P and its Metabolites Reg.No. 360714 and Reg.No. 360715 in Soil using HPLC/MS-MS, Obermann, M., 2008, Doc No. 2008/1042152, study code 148916, ASB2010-4519

Guideline(s): Yes (SANCO/825/00 rev. 7, SANCO/3029/99 rev.4)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid-P, metabolites M23, M27
Analyte(s) determined as:
Dimethenamid-P, metabolites M23, M27

Principle of the method:
Samples are shaken with methanol/water (6+4, v/v) followed by centrifugation. The supernatant is diluted with water. The sample concentration in final extracts is 0.005 or 0.05 g/mL. The concentrations of all analytes in the final extract are quantified by LC-MS/MS using a Zorbax Eclipse XDB C18 column. Positive electrospray ionisation is chosen for parent dimethenamid-P and two

MRM transitions (m/z 276→168, 276→244) are monitored. For the metabolites M23 and M27 negative electrospray ionisation is set and two MRM transitions per analyte are monitored: m/z 270→166, 270→198 (M23), m/z 320→80, 320→121 (M27).

Findings

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.
Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-6.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-6.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-6 is considered as limit of quantification.
Matrix effects:
Matrix effects were not investigated.
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	no
Accepted calibration range in concentration units:	0.1-1 ng/mL
Accepted calibration range in mass fraction units:	0.002-0.2 mg/kg
Calibration conducted with matrix matched standards:	yes
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid (see Table B.5.2-6).

Conclusion

The analytical method by Obermann (2008, ASB2010-4519) is suitable as enforcement method for dimethenamid-P and metabolites M23 and M27 at a level of 0.005 mg/kg in soil. A confirmatory method for soil is provided by full validation of a second MS/MS transition. Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ in all matrices tested.

Table B.5.2-6: Validation of the method by Obermann (2008) for residues in soil, ASB2010-4519

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
Dimethenamid-P						
Obermann (2008) (ASB2010-4519)	Lufa 5M	LC-MS/MS, C18, ESI+, m/z 276→168	0.005 0.05	85.8 78.3	1.5 14.1	5 7
	Lufa 2.2		0.005 0.05	96.4 94.2	2.0 4.2	5 5
	Lufa 5M	LC-MS/MS, C18, ESI+, m/z 276→244	0.005 0.05	85.8 78.5	1.5 13.6	5 7
	Lufa 2.2		0.005 0.05	96.5 94.6	1.8 3.3	5 5
M23						
Obermann (2008) (ASB2010-4519)	Lufa 5M	LC-MS/MS, C18, ESI+, m/z 270→166	0.005 0.05	91.3 98.1	11.9 7.5	5 7
	Lufa 2.2		0.005 0.05	102.8 97.7	11.8 15.7	5 5
	Lufa 5M	LC-MS/MS, C18, ESI+, m/z 270→198	0.005 0.05	94.5 94.1	3.5 7.0	5 7
	Lufa 2.2		0.005 0.05	99.0 101.1	3.9 3.7	5 5
M27						
Obermann (2008) (ASB2010-4519)	Lufa 5M	LC-MS/MS, C18, ESI+, m/z 320→80	0.005 0.05	88.8 94.1	3.5 6.3	5 6
	Lufa 2.2		0.005 0.05	91.9 95.3	10.7 2.7	5 5
	Lufa 5M	LC-MS/MS, C18, ESI+, m/z 320→121	0.005 0.05	91.4 95.4	2.2 4.4	5 6
	Lufa 2.2		0.005 0.05	101.5 102.2	6.3 4.4	5 5

Study 2

Data point: KCA 4.1.2/1

Report: Validation of analytical method L0109/02: Determination of dimethenamid-P (BAS 656 H) and its metabolites Reg No. 360715 (M23), Reg No. 360714 (M27) and Reg No. 360712 (M31) in soil and sediment by HPLC/MS-MS, Tilting, N., 2014, Doc ID 2013/1110235, Study code 380201 method L0109/02, ASB2014-8294

Guideline(s): Yes (SANCO/825/00 rev. 8.1, SANCO/3029/99 rev.4)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid-P, metabolites M23, M27, M31
Analyte(s) determined as:
Dimethenamid-P, metabolites M23, M27, M31

Principle of the method:
Samples are shaken with methanol/water (6 + 4, v/v) followed by centrifugation. The supernatant is diluted with water. The sample concentration in final extracts is 0.005 or 0.05 g/mL. The concentrations of all analytes in the final extract are quantified by LC-MS/MS using a Zorbax Eclipse XDB C18 column. Positive electrospray ionisation is chosen for parent dimethenamid-P and two MRM transitions (m/z 276→244, 276→168) are monitored. For the metabolites M23 M27 and M31 negative electrospray ionisation is set and two MRM transitions per analyte are monitored: m/z 270→198, 270→166 (M23), m/z 320→121, 320→80 (M27) and m/z 346→240, 346→198 (M31).

Findings

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.
Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-7.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-7.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-7 is considered as limit of quantification.
Matrix effects:
Matrix effects were not investigated.
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	yes
Accepted calibration range in concentration units:	0.05-1 ng/mL
Accepted calibration range in mass fraction units:	0.001-0.20 mg/kg
Calibration conducted with matrix matched standards:	yes
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid (see Table B.5.2-7).

Conclusion

The analytical method by Tilting (2014, ASB2014-8294) is suitable as enforcement method for dimethenamid-P and metabolites M23, M27 and M31 in soil. A confirmatory method is provided by full validation of a second MS/MS transition/two additional fragment ions. Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ in all matrices tested.

Table B.5.2-7: Validation of the method by Tilting (2014) for residues in soil, ASB2014-8294

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
Dimethenamid-P						
Tilting (2014) (ASB2014-8294)	Lufa 2.2	LC-MS/MS, C18, ESI+, m/z 276→244	0.005 0.05	98.2 99.4	1.4 1.6	5 5
	Lufa 5M		0.005 0.05	99.9 106.2	1.8 1.0	5 5
	Sediment		0.005 0.05	99.8 102.6	1.2 2.8	5 5
	Lufa 2.2	LC-MS/MS, C18, ESI+, m/z 276→168	0.005 0.05	98.7 97.3	1.4 1.1	5 5
	Lufa 5M		0.005 0.05	99.8 106.4	2.1 1.4	5 5
	Sediment		0.005 0.05	100.5 101.0	1.3 3.3	5 5
M23						
Tilting (2014) (ASB2014-8294)	Lufa 2.2	LC-MS/MS, C18, ESI+, m/z 270→198	0.005 0.05	96.6 97.4	2.7 3.1	5 5
	Lufa 5M		0.005 0.05	100.7 104.2	2.7 3.4	5 5
	Sediment		0.005 0.05	99.0 104.1	4.7 3.4	5 5
	Lufa 2.2	LC-MS/MS, C18, ESI+, m/z 270→166	0.005 0.05	101.5 91.4	3.4 4.4	5 5
	Lufa 5M		0.005 0.05	96.4 100.1	9.7 9.5	5 5
	Sediment		0.005 0.05	99.0 102.6	4.5 3.8	5 5
M27						
Tilting (2014) (ASB2014-8294)	Lufa 2.2	LC-MS/MS, C18, ESI+, m/z 320→121	0.005 0.05	95.9 96.5	3.1 3.1	5 5
	Lufa 5M		0.005 0.05	101.0 101.9	3.9 2.6	5 5
	Sediment		0.005 0.05	101.5 102.4	2.9 4.2	5 5
	Lufa 2.2	LC-MS/MS, C18, ESI+, m/z 320→80	0.005 0.05	97.1 96.2	2.1 2.4	5 5
	Lufa 5M		0.005 0.05	99.4 98.2	1.9 2.7	5 5
	Sediment		0.005 0.05	99.0 100.3	2.1 2.6	5 5

Reference	Matrix	Detection method	Fortification level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses
M31						
Tilting (2014) (ASB2014-8294)	Lufa 2.2	LC-MS/MS, C18, ESI+, m/z 346→240	0.005 0.05	97.9 99.5	6.1 2.7	5 5
	Lufa 5M		0.005 0.05	99.0 95.3	4.8 7.7	5 5
	Sediment		0.005 0.05	97.1 95.4	1.8 6.5	5 5
	Lufa 2.2	LC-MS/MS, C18, ESI+, m/z 346→198	0.005 0.05	96.2 98.1	5.1 5.0	5 5
	Lufa 5M		0.005 0.05	97.0 96.8	5.1 5.4	5 5
	Sediment		0.005 0.05	100.2 100.3	3.5 6.8	5 5

B.5.2.4 Analytical methods for the determination of residues in drinking/surface water

B.5.2.4.1 Acceptable methods/reports

Study 1

Data point: KCA 4.1.2/3

Report: Determination of dimethenamid-P and its metabolites M23, M27 and M31 in water, Jooß, S., 2012, Doc. ID 2012/1278546, Study code: 2711 G, 353459, ASB2014-8295

Guideline(s): Yes (SANCO/825/00 rev. 8.1, SANCO/3029/99 rev.4)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid-P, metabolites M23, M27, M31
Analyte(s) determined as:
Dimethenamid-P, metabolites M23, M27, M31

Principle of the method:
Samples of drinking and surface water containing 0.1 % formic acid are analysed by direct injection into the chromatographic system. The concentrations of all analytes in the final extract are quantified by standards in HPLC water using LC-MS/MS using a Zorbax Eclipse XDB C18 column. Positive electrospray ionisation is set for parent dimethenamid-P and two MRM transitions (m/z 276→244, 276→168) are monitored. For the metabolites M23 M27 and M31 negative electrospray ionisation is set and two MRM transitions per analyte are monitored: m/z 270→198, 270→166 (M23), m/z 320→121, 320→80 (M27) and m/z 346→240, 346→198 (M31).

Findings

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.
Recovery (accuracy):
The recovery obtained with standards in water for HPLC is considered as recovery in this study. Alternatively, this "recovery" can be interpreted as matrix effect. For accuracy of analytical results see Table B.5.2-8.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-8.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-8 is considered as limit of quantification.
Matrix effects:
Significant matrix effects (i.e. deviations of recovery from 100 %) were not observed.
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	yes
Accepted calibration range in concentration units:	0.009 - 0.4 ng/mL
Accepted calibration range in mass fraction units:	0.009 - 0.4 µg/L
Calibration conducted with matrix matched standards:	no
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid (see Table B.5.2-8).

Conclusion

The analytical method by Jooß (2012, ASB2014-8295) is suitable as enforcement method for dimethenamid-P and metabolites M23, M27 and M31 in drinking and surface water. A confirmatory method for drinking/surface water is provided by full validation of a second MS/MS transition. Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ in all matrices tested.

Table B.5.2-8: Validation of the method by Jooß (2012) for residues in drinking/surface water, ASB2014-8295)

Reference	Matrix	Detection method	Fortification level [µg/L]	Average recovery [%]	RSD [%]	No. of analyses
Dimethenamid-P						
Jooß (2012) (ASB2014-8295)	Drinking water	LC-MS/MS, C18, ESI+, m/z 276→244	0.03 0.3	109 110	5 4	5 5
	Surface water		0.03 0.3	110 109	7 4	5 5
	Drinking water	LC-MS/MS, C18, ESI+, m/z 276→168	0.03 0.3	108 110	6 5	5 5
	Surface water		0.03 0.3	110 110	5 5	5 5
M23						
Jooß (2012) (ASB2014-8295)	Drinking water	LC-MS/MS, C18, ESI+, m/z 270→198	0.03 0.3	107 98	3 6	5 5
	Surface water		0.03 0.3	108 104	7 6	5 5
	Drinking water	LC-MS/MS, C18, ESI+, m/z 270→166	0.03 0.3	103 98	8 6	5 5
	Surface water		0.03 0.3	102 103	8 6	5 5
M27						
Jooß (2012) (ASB2014-8295)	Drinking water	LC-MS/MS, C18, ESI+, m/z 320→121	0.03 0.3	106 96	7 6	5 5
	Surface water		0.03 0.3	110 105	3 5	5 5
	Drinking water	LC-MS/MS, C18, ESI+, m/z 320→80	0.03 0.3	110 97	6 7	5 5
	Surface water		0.03 0.3	110 106	7 6	5 5
M31						
Jooß (2012) (ASB2014-8295)	Drinking water	LC-MS/MS, C18, ESI+, m/z 346→240	0.03 0.3	107 97	6 5	5 5
	Surface water		0.03 0.3	110 103	8 6	5 5
	Drinking water	LC-MS/MS, C18, ESI+, m/z 346→198	0.03 0.3	97 91	12 6	5 5
	Surface water		0.03 0.3	99 103	7 5	5 5

Study 2

Independent laboratory validation (ILV) for drinking water of the method by Jooß (2012) (ASB2014-8295)

Data point:	KCA 4.2/6
Report:	Independent laboratory validation of BASF analytical method: "Determination of dimethenamid-P and its metabolites M23, M27 and M31 in water" for dimethenamid-P only, Liu, W.; Shi, Y., 2014, Doc ID 2014/7000491, Study No. 732240, Alliance Pharma Study Number 140232, ASB2014-8337
Guideline(s):	Yes (SANCO/825/00 rev. 8.1, OCSPP 850.6100)
Deviations:	No
GLP:	Yes
Acceptability:	Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid-P (In contrast to the title of the study, this ILV does not validate the method for metabolites.)
Analyte(s) determined as:
Dimethenamid-P

Principle of the method:
Similar to the method by Jooß (2012, ASB2014-8295). Calibration was done with external standards in methanol.

Findings

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.
Recovery (accuracy):
The recovery obtained with standards in methanol is considered as recovery in this study. Alternatively, this “recovery” can be interpreted as matrix effect. For accuracy of analytical results see Table B.5.2-9.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-9.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-9 is considered as limit of quantification.
Matrix effects:
Matrix effects are not evaluated.
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	yes
Accepted calibration range in concentration units:	0.009-0.4 ng/mL
Accepted calibration range in mass fraction units:	0.009-0.4 µg/L
Calibration conducted with matrix matched standards:	no
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were

found to be valid (see Table B.5.2-9).

Conclusion

The study is considered as an independent laboratory validation for drinking water of the method described by Jooß (2012, ASB2014-8295).

Table B.5.2-9: Validation of the independent laboratory validation by Liu & Shi (2014) for residues in drinking water, ASB2014-8295)

Reference	Matrix	Detection method	Fortification level [µg/L]	Average recovery [%]	RSD [%]	No. of analyses
Dimethenamid-p						
Liu & Shi (2014) (ASB2014-8337)	Drinking water	LC-MS/MS, C18, ESI+, m/z 276→244	0.03 0.3	97 99	6.1 2.8	5 5
	Drinking water	LC-MS/MS, C18, ESI+, m/z 276→168	0.03 0.3	97 100	2.9 1.9	5 5

Study 3

Data point: KCA 4.1.2/5

Report: Determination of Dimethenamid-P and its Metabolites M23 and M27 in Tap and Surface Water - Validation of the Method 519/0, Schulz, H.; Meyer, M., 2007, Doc ID 2007/1054384, Study No.148913, ASB2010-4520

Guideline(s): Yes (SANCO/825/00 rev. 7)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid-P, metabolites M23, M27
Analyte(s) determined as:
Dimethenamid-P, metabolites M23, M27

Principle of the method:
The method description for extraction is taken from Grote (2003, MET2005-206), Validation of analytical method No. 519/0: LC/MS determination of BAS 479 H (Metazachlor, 114252) and its metabolites BH 479-4 (211193) and BH 479-8 (291634) in tap, surface and leachate water, Study No. 2003/1005471, MET2005-206.
Samples of drinking and surface water are adjusted with 6 M HCl to pH 2, followed by SPE on a Bond Elut ENV (styrene divinylbenzene polymer) cartridge. Analytes are eluted with methanol. The extracts are evaporated to dryness and reconstituted in water/methanol (8 + 2, v/v). Sample concentration in final extracts is 5 g/mL. The concentrations of all analytes in the final extract are quantified by LC-MS/MS using a Zorbax Eclipse XDB C18 column. Positive electrospray ionisation is set for parent dimethenamid-P and two MRM transitions (m/z 276→244, 276→168) are monitored. For the metabolites M23 and M27 negative electrospray ionisation is set and two MRM transitions per analyte are monitored: m/z 270→198, 270→166 (M23) and m/z 320→121, 320→80 (M27).

Findings

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.
Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-10.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-10.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-10 is considered as limit of quantification.
Matrix effects:
Matrix effects are not evaluated.
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	yes
Accepted calibration range in concentration units:	0.1 - 5 ng/mL
Accepted calibration range in mass fraction units:	0.02 - 1 µg/L
Calibration conducted with matrix matched standards:	no
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid (see Table B.5.2-10).

Conclusion

The analytical method by Schulz & Meyer (2007, ASB2010-4520) is suitable as an enforcement method for dimethenamid-P and metabolites M23 and M27 in drinking and surface water. A confirmatory method for drinking/surface water is provided by a second MS/MS transition. For the second transition no calibration and chromatograms are shown. However, it is still accepted as a confirmatory method since recoveries und RSDs are fully reported. Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ in all matrices tested.
The study was not validated by an independent laboratory.

Table B.5.2-10: Validation of the method by Schulz & Meyer (2007) for residues in drinking/surface water, ASB2010-4520)

Reference	Matrix	Detection method	Fortification level [µg/L]	Average recovery [%]	RSD [%]	No. of analyses
Dimethenamid-P						
Schulz & Meyer (2007) (ASB2010-4520)	Drinking water	LC-MS/MS, C18, ESI+, m/z 276→244	0.05 0.5	74.1 76.8	1.2 3.8	5 5
	Surface water		0.05 0.5	87.6 87.0	1.8 2.3	5 5
	Drinking water	LC-MS/MS, C18, ESI+, m/z 276→168	0.05 0.5	73.7 77.3	2.1 3.7	5 5
	Surface water		0.05 0.5	86.5 87.9	2.8 3.1	5 5
M23						
Schulz & Meyer (2007) (ASB2010-4520)	Drinking water	LC-MS/MS, C18, ESI+, m/z 270→198	0.05 0.5	91.8 89.3	1.0 3.7	5 5
	Surface water		0.05 0.5	106.6 104.0	0.8 1.3	5 5
	Drinking water	LC-MS/MS, C18, ESI+, m/z 270→166	0.05 0.5	89.3 89.5	3.8 2.6	5 5
	Surface water		0.05 0.5	106.0 103.9	1.8 1.2	5 5
M27						
Schulz & Meyer (2007) (ASB2010-4520)	Drinking water	LC-MS/MS, C18, ESI+, m/z 320→121	0.05 0.5	91.6 89.2	3.8 3.5	5 5
	Surface water		0.05 0.5	103.5 106.5	4.5 1.6	5 5
	Drinking water	LC-MS/MS, C18, ESI+, m/z 320→80	0.05 0.5	92.2 89.9	2.7 2.9	5 5
	Surface water		0.05 0.5	109.0 106.0	1.5 1.6	5 5

Study 4

Data point: KCA 4.2

Report: Laboratory Method Trial for the Residue Method AM-0853-0491-0 Determination of SAN-582H in Water, Colin, T., 1998, Reg. Doc. Number 98/5177, Study Number 98083, MET1999-540

Guideline(s): Yes (European Commission Draft Working Document on Residue Analytical Methods (8064/VI/97-rev 1))

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid

Analyte(s) determined as:
Dimethenamid

Principle of the method:
Samples of drinking and surface water are extracted by means of SPE on a C18 cartridges and dimethenamid is eluted with methanol/water (85 + 15, v/v). The extracts are evaporated to the aqueous remainder followed by liquid-liquid partitioning with toluene and drying of the toluene phase over sodium sulfate. The sample concentration in final extracts is 500 mL/mL. The concentration of dimethenamid in the final extract is quantified by GC-MSD using a Zorbax HP-5MS column and monitoring the fragment ions m/z 154, 203, 230. A confirmatory analysis is also performed using GC-NPD using a SPB-5 column.

Findings

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.
Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-11.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-11.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-11 is considered as limit of quantification.
Matrix effects:
Matrix effects are not evaluated.
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	no (4 points)
Accepted calibration range in concentration units:	25 - 500 ng/mL
Accepted calibration range in mass fraction units:	0.05 - 1 µg/L
Calibration conducted with matrix matched standards:	no
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from three different fragment ions were used for quantification. All results were found to be valid (see Table B.5.2-11). Additionally samples are analysed by GC-NPD.

Conclusion

The analytical method by Colin (1998, MET1999-540) was submitted as method for risk assessment (KCA 4.1.2), but is considered here as a suitable enforcement method for dimethenamid and in drinking and surface water. A confirmatory method for drinking and surface water is provided by full validation of two additional fragment ions. Recoveries at 0.1 µg/L in drinking water with GC-MSD detection were >120 %. The highest fortification level of 10 µg/L is outside of the calibrated range. Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ in all matrices tested.

Table B.5.2-11: Validation of the method by Colin (1998) for residues in drinking/surface water, MET1999-540

Reference	Matrix	Detection method	Fortification level [µg/L]	Average recovery [%]	RSD [%]	No. of analyses
Dimethenamid-P						
Colin (1998) (MET1999-540)	Drinking water	GC-MSD, HP-5MS, m/z 154	0.05 0.1 1 10	106.7 126.2 84.9 75.0	11.0 3.8 4.0 9.0	5 5 5 5
	Surface water		0.05 0.1 1 10	111.9 102.9 85.7 86.2	4.5 15.0 5.1 7.7	5 5 5 5
	Drinking water	GC-MSD, HP-5MS, m/z 203	0.05 0.1 1 10	109.0 133.4 86.0 75.4	8.2 5.1 2.8 9.0	5 5 5 5
	Surface water		0.05 0.1 1 10	109.0 104.6 84.5 84.9	8.3 13.3 5.1 7.4	5 5 5 5
	Drinking water	GC-MSD, HP-5MS, m/z 230	0.05 0.1 1 10	110.7 134.6 85.4 74.3	7.4 9.3 2.8 10.0	5 5 5 5
	Surface water		0.05 0.1 1 10	116.3 104.7 83.7 84.2	5.0 15.2 4.3 7.0	5 5 5 5
	Drinking water	GC-NPD, SPB-5	0.05 0.1 1 10	131.0 113.0 102.9 90.2	12.4 6.9 3.8 6.7	5 5 5 5
	Surface water		0.05 0.1 1 10	98.8 101.8 81.4 80.6	8.5 8.0 3.9 4.3	5 5 5 5

B.5.2.4.2 Additional studies/reports

Study 1

Data point: KCA 4.1.2/4

Report: Validation of an analytical method for determination of metabolites of dimethenamid-P in water, Mewis, A., 2013, Doc. ID 2013/1349800, Study code: S13-03461, Study ID 703063, ASB2014-8296

Guideline(s): Yes (SANCO/825/00 rev. 8.1, SANCO/3029/99 rev.4, OPPTS 850.7100)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods:

Fortified analyte(s):
Metabolites M3, M10, M23, M27, M31, M32, M43, M45, M47 and M54
Analyte(s) determined as:
Metabolites M3, M10, M23, M27, M31, M32, M43, M45, M47 and M54

Principle of the method:
Characterised surface water samples are acidified with HCl and extracted by SPE on Oasis HLB cartridges. Analytes are eluted with acetonitrile/methanol/28 % aqueous ammonia solution (50 + 50 + 2, v/v/v), followed by dilution of an aliquot with 10 % acetic acid. The sample concentration in final extracts is 10 mL/mL. The concentrations of all analytes in the final extract are quantified by LC-MS/MS using a Zorbax Eclipse XDB C18 column. Positive electrospray ionisation is set for M3 and M656PH010 and the MRM transitions m/z 242→210, 242→168 and m/z 320→288, 320→166 are monitored, respectively. For all other metabolites negative electrospray ionisation is set and the following two MRM transitions per analyte are monitored: m/z 270→198, 270→166 (M23), m/z 320→121, 320→80 (M27), m/z 346→240, 346→198 (M31), m/z 330→240, 330→198 (M656H032), m/z 286→242 286→210 (M656PH043), m/z 300→184, 300→228 (M656PH045), m/z 350→306, 350→121 (M656PH047), m/z 320→198, 320→121 (M656PH054).

Findings

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.
Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-12.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-12.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-12 is considered as limit of quantification.
Matrix effects:
Significant matrix effects up to 25 % were observed. Therefore matrix matched standards are used.
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	yes
Accepted calibration range in concentration units:	0.075 - 10 ng/mL
Accepted calibration range in mass fraction units:	0.0075 - 1 µg/L
Calibration conducted with matrix matched standards:	yes
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid (see Table B.5.2-12).

Conclusion

The analytical method by Mewis (2013, ASB2014-8296) is suitable as an enforcement method for the dimethenamid metabolites M3, M10, M23, M27, M31, M32, M43, M45, M47 and M54 in surface water. A confirmatory method for all metabolites is provided by full validation of a second MS/MS transition. Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ in all matrices tested.

Table B.5.2-12: Validation of the method by Mewis (2013) for residues in surface water, ASB2014-8296)

Reference	Matrix	Detection method	Fortification level [µg/L]	Average recovery [%]	RSD [%]	No. of analyses
M3						
Mewis (2013) (ASB2014-8296)	Surface water	LC-MS/MS, C18, ESI+, m/z 242→210	0.025 0.25	110 98	3.5 1.7	5 5
	Surface water	LC-MS/MS, C18, ESI+, m/z 242→168	0.025 0.25	105 98	4.7 3.5	5 5
M10						
Mewis (2013) (ASB2014-8296)	Surface water	LC-MS/MS, C18, ESI+, m/z 320→288	0.025 0.25	109 101	4.4 2.1	5 5
	Surface water	LC-MS/MS, C18, ESI+, m/z 320→166	0.025 0.25	106 104	4.7 2.1	5 5
M23						
Mewis (2013) (ASB2014-8296)	Surface water	LC-MS/MS, C18, ESI-, m/z 270→198	0.025 0.25	99 100	16.4 3.0	5 5
	Surface water	LC-MS/MS, C18, ESI-, m/z 270→166	0.025 0.25	99 91	14.1 3.0	5 5
M27						
Mewis (2013) (ASB2014-8296)	Surface water	LC-MS/MS, C18, ESI-, m/z 320→121	0.025 0.25	98 97	6.1 3.2	5 5
	Surface water	LC-MS/MS, C18, ESI-, m/z 320→80	0.025 0.25	109 102	5.3 0.4	5 5
M31						
Mewis (2013) (ASB2014-8296)	Surface water	LC-MS/MS, C18, ESI-, m/z 346→240	0.025 0.25	104 89	9.8 5.0	5 5
	Surface water	LC-MS/MS, C18, ESI-, m/z 346→198	0.025 0.25	110 102	12.6 5.5	5 5
M32						
Mewis (2013) (ASB2014-8296)	Surface water	LC-MS/MS, C18, ESI-, m/z 330→240	0.025 0.25	97 97	15.9 8.2	5 5
	Surface water	LC-MS/MS, C18, ESI-, m/z 330→198	0.025 0.25	110 105	11.3 2.9	5 5

Reference	Matrix	Detection method	Fortification level [µg/L]	Average recovery [%]	RSD [%]	No. of analyses
M43						
Mewis (2013) (ASB2014-8296)	Surface water	LC-MS/MS, C18, ESI-, m/z 286→242	0.025 0.25	110 98	10.8 5.4	5 5
	Surface water	LC-MS/MS, C18, ESI-, m/z 286→210	0.025 0.25	103 101	6.1 5.8	5 5
M45						
Mewis (2013) (ASB2014-8296)	Surface water	LC-MS/MS, C18, ESI-, m/z 300→184	0.025 0.25	110 95	9.5 1.1	5 5
	Surface water	LC-MS/MS, C18, ESI-, m/z 300→228	0.025 0.25	110 105	4.6 3.0	5 5
M47						
Mewis (2013) (ASB2014-8296)	Surface water	LC-MS/MS, C18, ESI-, m/z 350→306	0.025 0.25	89 97	15.0 4.4	5 5
	Surface water	LC-MS/MS, C18, ESI-, m/z 350→121	0.025 0.25	109 98	10.5 3.6	5 5
M54						
Mewis (2013) (ASB2014-8296)	Surface water	LC-MS/MS, C18, ESI-, m/z 320→198	0.025 0.25	100 92	19.1 5.1	5 5
	Surface water	LC-MS/MS, C18, ESI-, m/z 320→121	0.025 0.25	110 95	18.2 5.2	5 5

Study 2

Independent laboratory validation (ILV) for drinking water of the method by Mewis (2013) (ASB2014-8296)

Data point: KCA 4.2/7

Report: Independent laboratory validation of method MGeN0001/13: Method for the determination of dimethenamid-P metabolites in water by LC-MS/MS, Yang, J.; Michener, P., 2014, Doc. ID 2013/7002762, Study No: 698271, ASB2014-8338

Guideline(s): Yes (SANCO/825/00 rev. 8.1, OECD ENV/JM/MONO (2007)17, OPPTS 850.6100)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods:

Fortified analyte(s):
Metabolites M3, M10, M23, M27, M31, M32, M43, M45, M47 and M54
Analyte(s) determined as:
Metabolites M3, M10, M23, M27, M31, M32, M43, M45, M47 and M54

Principle of the method:
The study refers to a method MGeN0001/13 by Mewis (2013), which was not submitted by the applicant. However, the ILV uses the same methodology then the above study by Mewis (2013, ASB2014-8296).

Findings

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected except for the primary transition of M47 in surface water with 35 %.
Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-13.
Repeatability (precision):
For repeatability of analytical results see Table B.5.2-13.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-13 is considered as limit of quantification.
Matrix effects:
Matrix effects are not evaluated.
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	yes
Accepted calibration range in concentration units:	0.075-100 ng/mL
Accepted calibration range in mass fraction units:	0.0075-10 µg/L
Calibration conducted with matrix matched standards:	yes
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid (see Table B.5.2-13).

Conclusion

The analytical method by Yang & Michener (2014, ASB2014-8338) is suitable as enforcement method for the dimethenamid metabolites M3, M10, M23, M27, M31, M32, M43/44, M45/46, M47/48 and M54/58 in drinking and surface water. A confirmatory method for all metabolites is provided by full validation of a second MS/MS transition. Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ in all matrices tested, except for the primary transition of M47/48 in surface water with 35 %. The study is considered as an independent laboratory validation of the method described by Mewis (2013, ASB2014-8296).

Table B.5.2-13: Validation of the method by Yang & Michener (2014) for residues in drinking and surface water, ASB2014-8338)

Reference	Matrix	Detection method	Fortification level [µg/L]	Average recovery [%]	RSD [%]	No. of analyses
M3						
Yang & Michener (2014) (ASB2014-8338)	Drinking water	LC-MS/MS, C18, ESI+, m/z 242→210	0.025 2.5	97 95	5 2	5 5
	Surface water		0.025 2.5	105 87	5 3	5 5
	Drinking water	LC-MS/MS, C18, ESI+, m/z 242→168	0.025 2.5	97 95	6 2	5 5
	Surface water		0.025 2.5	105 87	4 1	5 5
M10						
Yang & Michener (2014) (ASB2014-8338)	Drinking water	LC-MS/MS, C18, ESI+, m/z 320→288	0.025 2.5	100 99	6 2	5 5
	Surface water		0.025 2.5	107 88	5 3	5 5
	Drinking water	LC-MS/MS, C18, ESI+, m/z 320→166	0.025 2.5	96 101	6 1	5 5
	Surface water		0.025 2.5	105 88	6 1	5 5
M23						
Yang & Michener (2014) (ASB2014-8338)	Drinking water	LC-MS/MS, C18, ESI-, m/z 270→198	0.025 2.5	105 98	3 4	5 5
	Surface water		0.025 2.5	107 103	5 3	5 5
	Drinking water	LC-MS/MS, C18, ESI-, m/z 270→166	0.025 2.5	102 100	6 3	5 5
	Surface water		0.025 2.5	100 103	10 4	5 5
M27						
Yang & Michener (2014) (ASB2014-8338)	Drinking water	LC-MS/MS, C18, ESI-, m/z 320→121	0.025 2.5	105 95	4 4	5 5
	Surface water		0.025 2.5	109 109	8 4	5 5
	Drinking water	LC-MS/MS, C18, ESI-, m/z 320→80	0.025 2.5	106 95	4 3	5 5
	Surface water		0.025 2.5	108 106	6 3	5 5

Reference	Matrix	Detection method	Fortification level [µg/L]	Average recovery [%]	RSD [%]	No. of analyses
M31						
Yang & Michener (2014) (ASB2014-8338)	Drinking water	LC-MS/MS, C18, ESI-, m/z 346→240	0.025 2.5	105 108	5 1	5 5
	Surface water		0.025 2.5	107 91	9 2	5 5
	Drinking water	LC-MS/MS, C18, ESI-, m/z 346→198	0.025 2.5	104 105	8 1	5 5
	Surface water		0.025 2.5	103 88	4 4	5 5
M32						
Yang & Michener (2014) (ASB2014-8338)	Drinking water	LC-MS/MS, C18, ESI-, m/z 330→240	0.025 2.5	107 102	2 4	5 5
	Surface water		0.025 2.5	103 106	8 3	5 5
	Drinking water	LC-MS/MS, C18, ESI-, m/z 330→198	0.025 2.5	106 101	5 3	5 5
	Surface water		0.025 2.5	103 105	6 2	5 5
M43						
Yang & Michener (2014) (ASB2014-8338)	Drinking water	LC-MS/MS, C18, ESI-, m/z 286→242	0.025 2.5	92 105	7 1	5 5
	Surface water		0.025 2.5	98 88	4 2	5 5
	Drinking water	LC-MS/MS, C18, ESI-, m/z 286→210	0.025 2.5	103 105	11 2	5 5
	Surface water		0.025 2.5	102 87	17 2	5 5
M45						
Yang & Michener (2014) (ASB2014-8338)	Drinking water	LC-MS/MS, C18, ESI-, m/z 300→184	0.025 2.5	93 95	5 3	5 5
	Surface water		0.025 2.5	99 86	8 2	5 5
	Drinking water	LC-MS/MS, C18, ESI-, m/z 300→228	0.025 2.5	95 95	7 3	5 5
	Surface water		0.025 2.5	100 91	13 3	5 5
M47						
Yang & Michener (2014) (ASB2014-8338)	Drinking water	LC-MS/MS, C18, ESI-, m/z 350→306	0.025 2.5	94 100	4 2	5 5
	Surface water		0.025 2.5	103 92	5 2	5 5
	Drinking water	LC-MS/MS, C18, ESI-, m/z 350→121	0.025 2.5	93 99	17 3	5 5
	Surface water		0.025 2.5	99 90	15 2	5 5

Reference	Matrix	Detection method	Fortification level [µg/L]	Average recovery [%]	RSD [%]	No. of analyses
M54						
Yang & Michener (2014) (ASB2014-8338)	Drinking water	LC-MS/MS, C18, ESI-, m/z 320→198	0.025 2.5	105 97	8 5	5 5
	Surface water		0.025 2.5	105 106	9 4	5 5
	Drinking water	LC-MS/MS, C18, ESI-, m/z 320→121	0.025 2.5	100 99	7 4	5 5
	Surface water		0.025 2.5	107 109	8 4	5 5

B.5.2.5 Analytical methods for the analysis in air

B.5.2.5.1 Acceptable methods/reports

Study 1

Data point: KCA 4.1.2/6

Report: Validation of analytical method L0167/01: Determination of dimethenamid-P in air, Zangmeister, W., 2010, Doc. ID 2010/1126085, Study Code: 391445, ASB2013-9757

Guideline(s): Yes (SANCO/825/00 rev. 7, SANCO/3029/99 rev.4)

Deviations: No

GLP: Yes

Acceptability: Yes

Materials and methods:

Fortified analyte(s):
Dimethenamid-P
Analyte(s) determined as:
Dimethenamid-P

Principle of the method:
Air is sampled on Tenax adsorption tubes at a temperature of 35 °C and relative humidity of 81 %. The total volume of air sampled for 6 hours is equal to 540 L. Analytes are extracted from the Tenax resin with acetone by means of sonication, followed by centrifugation. Extract aliquots are diluted 1:100 with methanol/water (1/1; v/v) and in case of higher residues appropriately (e.g. 1:10.000). The concentrations in the final extracts are quantified by LC-MS/MS using a Betasil C18 column. Positive electrospray ionisation is set for parent dimethenamid-P and two MRM transitions (m/z 276→168, 276→244) are monitored.

Findings

Selectivity (specificity):
Signals from interferences >30 % of LOQ in blank samples were not detected.
Recovery (accuracy):
For accuracy of analytical results see Table B.5.2-14.

Repeatability (precision):
For repeatability of analytical results see Table B.5.2-14.
Limit of quantification (LOQ):
For each matrix type the lowest successfully validated level in Table B.5.2-14 is considered as limit of quantification.
Matrix effects:
Matrix effects are not evaluated.
Calibration (linearity):

Does the calibration consist of at least 3 levels (duplicated points) or 5 levels (single points):	yes
Accepted calibration range in concentration units:	0.1 - 2.0 ng/mL
Accepted calibration range in mass fraction units:	0.185 - 370 µg/m³
Calibration conducted with matrix matched standards:	no
Sample chromatogram spiked at LOQ demonstrates sufficient sensitivity and signal-to-noise ratio:	yes

Confirmation:
Signals obtained from two different MRM transitions were used for quantification. Both results were found to be valid (see Table B.5.2-14).

Conclusion

The analytical method by Zangmeister (2010, ASB2013-9757) is suitable as enforcement method for dimethenamid-P in air. A confirmatory method for air is provided by full validation of a second MS/MS transition. Blank values in chromatograms for quantification and confirmatory transitions, are generally below 30 % of LOQ in all matrices tested. No breakthrough is observed.

Table B.5.2-14: Validation of the method by Zangmeister (2010) for residues in air, ASB2013-9757

Reference	Matrix	Detection method	Fortification level [µg/m³]	Average recovery [%]	RSD [%]	No. of analyses
Zangmeister (2010) (ASB2013-9757)	Air	LC-MS/MS, C18, ESI+, m/z 276→168	1.5 150	87.9 97.0	12.3 3.1	5 6
	Air	LC-MS/MS, C18, ESI+, m/z 276→244	1.5 150	87.9 97.0	12.1 3.5	5 6

B.5.2.5.2 Methods which do not fulfil the requirements

Table B.5.2-15: List of methods, which do not fulfil requirements

Author(s) and year	Report No	Reason
Kettner & Karapally (1994)	KCA 4.1.2 94/10638 MET9700263	The number of fortified samples per level (n = 3) is not sufficient according to SANCO/825/00 rev. 8.1.

B.5.2.6 Analytical methods for the analysis in body fluids and tissues

Tissues: See method by Gordon (2014, ASB2014-8335) under B.5.2.2.1.
Body fluids: No methods were provided. This is considered a data gap.

B.5.3 References relied on

The applicant has not provided a search of published literature. Therefore, a literature search was performed in order to identify relevant studies in the peer reviewed literature was perfomed by the RMS. The search strategy comprised of combining “dimethenamid” with words or word fragments typical in residue anatytical studies such as method*, assay*, valid*, quantit*, “gas chromatography*", "liquid chromatography*”, and calibr*. These combinations were used with the following literature databases: Pub Med, Scopus, Tox Net, Web of Science. Several studies were considered as potentially relevant, but were not further evaluated as all data requirements on analytical methods for monitoring are fulfilled with studies provided with the dossier.

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**	**Data protection claimed Y/N**	**Justification if data protection is claimed**	**Owner**
	Germany	2000	Dimethenamid-P (Monograph) GLP: N Published: Yes ASB2010-10566	N	N	-	LIT
KCA 4.1.1/1	Nemitz A., Genari G.	2013	Determination of active ingredient S-dimethenamid and its isomer R-dimethenamid in dimethenamid-P technical grade active ingredient (TGAI) by means of HPLC 2013/1066432 BASF SE, Limburgerhof, Germany Fed.Rep. GLP: no Unpublished BVL no. 2630042	N	Y	New data for AIR3 renewal	BAS
KCA 4.1.1/2	Sonnen-schein L.	2013	Validation of the analytical method APL0665/01: Determination of S-dimethenamid and its isomer R-dimethenamid in dimethenamid-P technical grade active ingredient (BAS 656 H) by means of high performance liquid chromatography (HPLC) 2013/1066433 Allessa Chemie GmbH, Frankfurt/Main, Germany Fed.Rep. GLP: yes Unpublished BVL no. 2630044	N	Y	New data for AIR3 renewal	BAS
KCA 4.2	Colin, T.	1998	Laboratory method trial for the residue method AM-0853-0491-0 - Determination of SAN-582H in water	No	No	-	BAS

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			98/5177 ! 137S14 GLP: yes Unpublished MET1999-540				
KCA 4.2	Diamadu-ros, B.	2014	Validation of BASF analytical method R0038/01: "Analytical method (Modified QuEChERS) for the determination of the residues of dimethenamid-P (Reg. No. 363851) and metabolites M26 (Reg. No. 360716) and M30 (Reg. No. 5296352) in plant matrices at a LOQ of 0.01 mg/kg using LC-MS/MS" 2013/7002627 ! 718508 ! method R0038/01 GLP: Yes Published: No BVL-2630063, ASB2014-8334	No	Yes	New data for AIR3 renewal	BAS
KCA 4.2	Gordon, B.	2014	Validation of BASF analytical method R0037/01: "Analytical method for the determination of the residues of dimethenamid-P (Reg. No. 363851) and metabolites M26 (Reg. No. 360716) and M30 (Reg. No. 5296352) in animal matrices at a LOQ of 0.01 mg/kg using LC-MS/MS" 2013/7002631 ! 718604 ! method R0037/01 GLP: Yes Published: No BVL-2630065, ASB2014-8335	No	Yes	New data for AIR3 renewal	BAS
KCA 4.2	Liu, W.; Shi, Y.	2014	Independent laboratory validation of BASF analytical method: "Determination of dimethenamid-P and its metabolites M23, M27 and M31 in water" for dimethenamid-P only 2014/7000491 ! 732240 ! 140232 GLP: Yes Published: No BVL-2630068, ASB2014-8337	No	Yes	New data for AIR3 renewal	BAS
KCA 4.2	Yang, J.;	2014	Independent laboratory	No	Yes	New data for	BAS

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	Michener, P.		validation of method MGeN0001/13: Method for the determination of dimethenamid-P metabolites in water by LC-MS/MS 2013/7002762 ! 698271 ! 053-0908 ! PASC-REP-0447 ! method MGeN0001/13 GLP: Yes Published: No BVL-2630069, ASB2014-8338			AIR3 renewal	
KCA 4.2, KCA 4.1.2	Lehmann, A.	2012	Validation of BASF method L0179/02: Method for the determination of dimethenamid-P (BAS 656 H) and its metabolites M23, M26, M27 and M30 in plant matrices 2011/1182078 ! 390386 ! method L0179/02 GLP: Yes Published: No BVL-2630056, BVL-2630058, ASB2014-3702	No	Yes	New data for AIR3 renewal	BAS
KCA 4.2, KCA 4.1.2	Rogers, P.; Fiorito, B.; Shi, Y.	2014	Independent laboratory validation of BASF analytical method L0179/02: "Method for the determination of dimethenamid-P (BAS 656 H, Reg. No. 363851), M23 (Reg. No. 360715), M26 (Reg. No. 360716), M27 (Reg. No. 360714) and M30 (Reg. No. 5296352) in plant matrices" 2013/7002656 ! 390388 ! 130913 ! method L0179/02 GLP: Yes Published: No BVL-2630057, BVL-2630060, ASB2014-8333	No	Yes	New data for AIR3 renewal	BASF
KCA 4.2	Grote, C.	2003	Validation of analytical method No. 519/0: LC/MS determination of BAS 479 H (Metazachlor, 114252) and its metabolites BH 479-4 (211193) and BH 479-8 (291634) in tap, surface and leachate water GLP: yes Unpublished 2003/1005471 ! 153307 !	N	N	-	BAS

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	Data protection claimed Y/N	Justification if data protection is claimed	Owner
			method 519/0, MET2005-206				
KCA 4.1.2	Jooß, S.	2012	Determination of dimethenamid-P and its metabolites M23, M27 and M31 in water 2012/1278546 ! P 2711 G ! 353459 GLP: Yes Published: No BVL-2630050, BVL-2630051, ASB2014-8295	No	Yes	New data for AIR3 renewal	BAS
KCA 4.1.2	Mewis, A.	2013	Validation of an analytical method for determination of metabolites of dimethenamid-P in water 2013/1349800 ! S13-03461 ! S13-03461-L1 ! 703063 GLP: Yes Published: No BVL-2630052, BVL-2630053, ASB2014-8296	No	Yes	New data for AIR3 renewal	BAS
KCA 4.1.2	Obermann, M.	2008	Validation of analytical method L0109/01: Determination of dimethenamid-P and its metabolites Reg.No.360714 and Reg.No.360715 in soil using HPLC/MS-MS 2008/1042152 ! 148916 ! method L0109/01 GLP: Yes Published: No BVL-2630049, ASB2010-4519	No	Yes	New data for AIR3 renewal	BAS
KCA 4.1.2	Schulz, H.; Meyer, M.	2007	Determination of dimethenamid-P and its Metabolites M23 and M27 in tap and surface water - Validation of the method 519/0 2007/1054384 ! 148913 ! IF-07/00871632 ! method 519/0 GLP: Yes Published: No BVL-2630054, ASB2010-4520	No	Yes	New data for AIR3 renewal	BAS
KCA 4.1.2	Tilting, N.	2014	Validation of analytical method L0109/02: Determination of dimethenamid-P (BAS 656 H) and its metabolites Reg No.	No	Yes	New data for AIR3 renewal	BAS

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	Data protection claimed Y/N	Justification if data protection is claimed	Owner
			360715 (M23), Reg No. 360714 (M27) and Reg No. 360712 (M31) in soil and sediment by HPLC/MS-MS 2013/1110235 ! 380201 ! method L0109/02 GLP: Yes Published: No BVL-2630048, ASB2014-8294				
KCA 4.1.2	Zangmeis-ter, W.	2010	Validation of analytical method L0167/01: Determination of dimethenamid-P in air 2010/1126085 ! 391445 ! method L0167/01 GLP: Yes Published: No BVL-2630055, ASB2013-9757	No	Yes	New data for AIR3 renewal	BAS