

Renewal Assessment Report

Dimethenamid-P

BAS 656 12 H

Volume 3 – B.5 Methods of analysis

Rev. 0 - 10 August 2016

Rapporteur Member State: Germany
Co-Rapporteur Member State: Bulgaria

Version history

When	What
10 August 2016	First version submitted to EFSA

Table of contents

B Summary, evaluation and assessment of the data and information

B.5	Methods of analysis.....	4
B.5.1	Methods used for the generation of pre-authorisation data	4
B.5.1.1	Analysis of the plant protection product.....	4
B.5.1.1.1	Methods for the determination of the active substance in the plant protection product	4
B.5.1.1.2	Methods for the determination of relevant impurities and formulants in the plant protection product	5
B.5.1.2	Methods for the determination of residues	6
B.5.1.2.1	Physical and chemical properties tests.....	6
B.5.1.2.2	Analytical methods in support of efficacy, toxicological, residue, fate and ecotoxicological studies	6
B.5.2	Methods for post-authorisation control and monitoring purposes	6
B.5.3	References relied on.....	7

B.5 Methods of analysis

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

B.5.1.1 Analysis of the plant protection product

B.5.1.1.1 Methods for the determination of the active substance in the plant protection product

Reference:

Ziegler (1998), Determination of the content of active ingredient dimethenamid-P in BAS 656 07 H using HPLC, BASF 98/11526, BASF (BVL no 2630568)

Ziegler (1998), Validation of the analytical method CF-A 563. Determination of dimethenamid-P in emulsifiable concentrates (BAS 656 07 H), BASF 98/11376, BASF (BVL no. 2630569)

Weatherhead (2013), Additional validation of analytical method CF-A 563 for the determination of active ingredients in BAS 656 12 H, BASF 2013/1342923, MX/13/030/1, BASF (BVL no. 2630571)

Principle of the method CF-A 563:

After homogenisation, the product is dissolved in dichloromethane and *n*-heptane. Dimethenamid-P is determined by HPLC-UV using external standard calibration.

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

Findings:

Table B.5.1-1: Validation data for the determination of dimethenamid-P in the plant protection product

	Specificity/ interferences	Linearity (R ²) (n = 5) (conc. range)	Accuracy (n = 6)		Repeatability (% RSD) (n = 6)
			fortification level (%)	mean recovery (%)	
Dimethenamid-P	demonstrated; no interferences	1.0000 0.43 - 2.55 mg/mL	50	99.7	0.36

In the study by Ziegler (1998), the validation of the method was performed with the formulation BAS 656 07 H instead of BAS 656 12 H (for comparison of the compositions see Volume 4). In the study by Weatherhead (2013), additional information on the peak identity and the absence of interferences demonstrate the applicability of analytical method CF-A 563 for BAS 656 12 H. The specificity of the method was demonstrated by retention time match and by comparison of the UV spectra with reference standard of the active substance.

Conclusion:

The method is considered acceptably validated and allows the determination of dimethenamid-P in the formulation BAS 656 12 H.

CIPAC method:

No existing CIPAC method was found to be applicable for analysis of the active substance in EC formulations.

B.5.1.1.2 Methods for the determination of relevant impurities and formulators in the plant protection product

Reference:

Walker, A. (2013), Validation of Analytical Method AFL0892/01 for the determination of Reg. No. 409284 in BAS 656 12 H, BASF 2013/1337740, BASF (BVL no 2931187)

Principle of the method AFL0892/01 for the determination of TCE:

The formulation is diluted with dichloroethane and o-xylene is added as internal standard. Quantification is made by GC-FID with external standard calibration using the ratio of TCE and the internal standard.

Column: Restek RTx-5MS 30 m x 0.32 mm, 1 µm or
Phenomenex ZB-5MS 30 m x 0.32 mm, 1 µm
Detector: FID

Findings:

Table B.5.1-2: Validation data for the determination of TCE in the plant protection product

	Specificity/ interferences	Linearity (R ²) (n = 5) (conc. range)	Accuracy		Repeatability (% RSD) (n = 6)
			fortification level (%)	mean recovery (%)	
TCE (1,1,1,2-tetrachloro- ethane)	demonstrated; no interferences	1.0000 4 - 119 µg/mL	0.02 0.18 0.36	95.8 (n = 6) 99.6 (n = 2) 99.4 (n = 2)	0.50 (at 0.12 %) 2.63 (at 0.015 %)

LOQ: 0.02 %

The identity of TCE was confirmed by retention time match and the comparison of mass spectra of sample and reference standard.

Conclusion

The analytical method for the determination of the impurity TCE in the formulation BAS 656 12 H is considered as acceptably validated.

Reference:

Walker, A. (2013), Validation of analytical method AFL0893/01 for the determination of Reg.No. 364801 in BAS 656 12 H, BASF 2013/1337742, BASF (BVL no 2931184)

Principle of the method AFL0893/01 for the determination of keto-enol:

The samples are prepared in isopropanol and determination of keto-enol is done with HPLC-DAD using a standard addition calibration/calculation.

Column: Zorbax Eclipse XDB-C8 250 x 4.6 mm, 6 µm
Elution solvent: 0.1 % CH₃COOH in a gradient of 90/10 to 10/90 H₂O/AcCN

Detector wavelength: 260 nm

Findings:

Table B.5.1-3: Validation data for the determination of keto-enol in the plant protection product

	Specificity/ interferences	Linearity (R ²) (n = 5) (conc. range)	Accuracy		Repeatability (% RSD) (n = 6)
			fortification level (%)	mean recovery (%)	
Keto-enol (2,4-dimethyl- thiophene-3-ol)	demonstrated; a slight interference was seen at 16.89 min. which is deemed not relevant	1.0000 0.025 - 0.5 mg/mL (equivalent to 0.05 - 1.0 %)	0.052 0.21 0.52	107 (n = 6) 105 (n = 2) 107 (n = 2)	2.37 (at 0.05 %)

LOQ: 0.0551 % w/w keto-enol relative to dimethenamid-P.

The identity of keto-enol was confirmed by retention time match and the comparison of mass spectra of sample and reference standard.

Conclusion

The analytical method for the determination of the keto-enol impurity in the formulation BAS 656 12 H is considered as acceptably validated.

B.5.1.2 Methods for the determination of residues

B.5.1.2.1 Physical and chemical properties tests

Please refer to B.5.1.1.1.

B.5.1.2.2 Analytical methods in support of efficacy, toxicological, residue, fate and ecotoxicological studies

There are no additional methods for the determination of residues in any matrix. All methods have been included in the active substance dossier.

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

Concerning analytical methods for the determination of the active substance in the formulation, reference is made to B.5.1.1.

B.5.3 References relied on

Data Point EU as of 2014	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Verte- brate study Y/N	Data Protection Claimed Y/N	Justification if data protection is claimed	Owner	Previously submitted Y/N If yes, old data point
KCP 5.1.1/1	Ziegler H.	1998	Determination of the content of active ingredient dimethenamid-P in BAS 656 07 H using HPLC 1998/11526 BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed.Rep. Not GLP, unpublished BVL no 2630568	N	N	New data for AIR3 renewal	BASF	N III A 5.1
KCP 5.1.1/2	Ziegler H.	1998	Validation of the analytical method CF-A 563. Determination of dimethenamid-P in emulsifiable concentrates (BAS 656 07 H) 1998/11376 BASF AG Agrarzentrum Limburgerhof, Limburgerhof, Germany Fed.Rep. GLP, unpublished BVL no. 2630569	N	Y	New data for AIR3 renewal	BASF	N III A 5.1
KCP 5.1.1/3	Weatherhead P.	2013	Additional validation of analytical method CF-A 563 for the determination of active ingredients in BAS 656 12 H 2013/1342923 Battelle UK Ltd., Havant Hampshire PO9 1SA, United Kingdom GLP, unpublished BVL no. 2630571	N	Y	New data for AIR3 renewal	BASF	N III A 5.1
KCP 5.1.1/3	Walker A.	2013	Validation of analytical method AFL0892/01 for the determination of Reg.No. 409284 in BAS 656 12 H 2013/1337740 Battelle UK Ltd., Havant Hampshire PO9 1SA, United Kingdom GLP, unpublished BVL no. 2931187	N	Y	New data for AIR3 renewal	BASF	N
KCP 5.1.1/4	Walker A.	2013	Validation of analytical method AFL0893/01 for the determination of Reg.No. 364801 in BAS 656 12 H 2013/1337742 Battelle UK Ltd., Havant Hampshire PO9 1SA, United Kingdom GLP, unpublished BVL no. 2931184	N	Y	New data for AIR3 renewal	BASF	N