Renewal Assessment Report

Dimethenamid-P

BAS 830 01 H

Volume 3 – B.5 Methods of analysis

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Rapporteur Member State: Germany

Co-Rapporteur Member State: Bulgaria

Version history

When	What
10 August 2016	First version submitted to EFSA

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B Summary, evaluation and assessment of the data and information

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:
Stickland (2013), Validation of analytical method AFL0879/01 for the determination of active ingredients in BAS 830 01 H, BASF 2013/1158737, MX/13/003/1, BASF (BVL no 2630756)

Stickland (2013), Quantitative determination of the active ingredients in BAS 830 01 H, BASF 2013/1158739, BASF (BVL no 2630758)

Principle of the method:

Chiral Determination of Dimethenamid-P Correction Factor
After homogenisation, the product is dissolved in THF 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 (gradient):	A: *n*-heptane / 2-propanol / THF 97:2:1 (v/v)
	B: *n*-heptane / 2-propanol / THF 80:10:3 (v/v/v)
Detector wavelength:	252 nm

The correction factor (P-enantiomeric part of the dimethenamid) in the sample is then applied to the assay of dimethenamid determined by the HPLC-UV method for the analysis of dimethenamid and quinmerac.

Determination of Dimethenamid and Quinmerac
After homogenisation, the product is dispersed in water. THF and a small amount of formic acid are added to dissolve. Dimethenamid and quinmerac are determined by HPLC-UV using external standard calibration.

Column:	Zorbax Eclipse XDB C18, 50 mm x 4.6 mm, 3.5 µm
Mobile phase (gradient):	A: H_2O / 0.5 M H_2SO_4 100:0.5 (v/v)
	B: CH_3CN / 0.5 M H_2SO_4 100:0.5 (v/v)
Detector wavelength:	220 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 = 1)		Repeatability (% RSD) (n = 6)
			fortification level (%)	mean recovery (%)	
Dimethenamid-P	demonstrated; no interferences	0.9998 0.03 – 0.10 mg/mL	18 24 31 30 32 41	99.4 98.0 98.0 98.9 98.8 99.1	0.93

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 (and quinmerac) in the formulation BAS 830 01 H.

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

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

Analytical methods for the determination of the relevant impurities in the plant protection product are missing. The applicant states that analytical methods and their validations for the determination of keto-enol and TCE in BAS 830 01 H can probably be submitted by the end of June 2016.

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	Date	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	Previously submitted Y/N If yes, old data point
KCP 5.1.1/1	Stickland L.J.	2013	21.05.2013	Validation of analytical method AFL0879/01 for the determination of active ingredients in BAS 830 01 H 2013/1158737 Battelle UK Ltd., Havant Hampshire PO9 1SA, United Kingdom GLP, unpublished BVL no 2630756	N	Y	New data for AIR3 renewal	BASF	N III A 5.1
KCP 5.1.1/2	Stickland L.J.	2013	21.05.2013	Quantitative determination of the active ingredients in BAS 830 01 H 2013/1158739 Battelle UK Ltd., Havant Hampshire PO9 1SA, United Kingdom Not GLP, unpublished BVL no 2630758	N	Y	New data for AIR3 renewal	BASF	N III A 5.1