

European Commission



**Draft Assessment Report prepared according to the Commission
Regulation (EU) N° 1107/2009**

BAS 750F (Mefentrifluconazole)

Volume 3 – B.5 (PPP) – BAS 750 01 F

Rapporteur Member State : United Kingdom
Co-Rapporteur Member State : France & Austria

Version History

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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

Report:	CP 5.1.1/1 Harsch M., 2014a Determination of the active ingredient Reg.No. 5834378 in EC-Formulation 2014/1097912
Guidelines:	SANCO/3030/99
GLP:	yes (certified by Landesamt fuer Umwelt, Wasserwirtschaft und Gewerbeaufsicht, Mainz, Germany)
Report:	CP 5.1.1/2 Harsch M., 2014b Validation of the Analytical Method AFL0909/01: Determination of the active ingredient Reg.No. 5834378 in EC-Formulation 2014/1097921
Guidelines:	OECD Principles of Good Laboratory Practice, GLP Principles of the German Chemikaliengesetz (Chemicals Act), 2004/10/EC, EC 1107/2009 of the European Parliament, CIPAC Guidelines on method validation, SANCO/3030/99, EPA 830.1000, EPA 830.1800
GLP:	yes (certified by Landesamt fuer Umwelt, Wasserwirtschaft und Gewerbeaufsicht, Mainz, Germany)
Study supported:	<i>KCP 2.1/1 Kroehl T., 2014 a Physical and chemical properties of BAS 750 01 F including low temperature stability (7 days at 0°C) and accelerated storage stability (14 days at 54°C) 2014/1219235</i> <i>KCP 2.7/1 Kroehl T., 2015 a Chemical and physical stability of formula BAS 750 01 F when stored for up to 3 years in PA/PE-coextruded packs - 52 week report 2015/1112122</i>

100 mg BAS 750 01 F was added to a 100 mL volumetric flask and made to volume with acetonitrile.

Analysis was performed by HPLC-UV DAD using an Acquity BEH C18 column (50 x 2.1 mm, 1.7µm) at 40 °C with UV detection at 232 nm and external standardisation. An isocratic elution is used (mobile phase A: 0.1% formic acid in water; mobile phase B: 0.1% formic acid in acetonitrile) followed by a rinsing step for detection and quantification. No CIPAC methods are available for the determination of the active substance in the preparation.

Table 5-1: Validation data

Analyte	Recovery fortification level (mg/L = % w/w in formulation]	Recoveries % range (mean, n)	Repeatability % RSD (n)	Linearity	Specificity
BAS 750 F (Reg.No. 5834378)	68.3 74.3 91.3 102.4 121.3 135.7	99.38 (n=1) 100.02 (n=1) 100.39 (n=1) 101.00 (n=1) 101.26 (n=1) 99.08 (n=1) (100.19, 6) All recoveries within the range 98 – 102 %	0.461 (5) @ 9.889 % w/w Modified Horwitz % RSDr = 1.90	57 – 165 mg/L [Approx. 57 – 164 % of nominal concentration] $y = 4579693x + 685876$ 5 standards, $r = 1.0000$	Acceptable chromatograms provided for reference item, test item, blank formulation and solvent blank. No interference > 3 % Identity confirmed by comparison of retention time, UV spectra and MS fragmentation (m/z 398.2, 400.2) of the test item and the reference item.

HPLC-UV DAD is a highly specific method and additional confirmation was not necessary. Chromatograms of the reference item, test item, blank formulation and solvent blank were provided showing no interference > 3 % at the retention time of interest. Accuracy was assessed at 6 fortification levels and in all cases the mean recovery was within the acceptable range of 98 - 102 %. To assess method precision, 5 determinations were made at each fortification level and RSD was lower than the acceptable Modified Horwitz RSDr of 1.90 %. The linear range is appropriate for the nominal test concentrations. The method is satisfactorily validated in accordance with SANCO/3030/99 rev.4.

Analytical Method AFL0928/01: Determination of [REDACTED] in BAS 750 01 F by GC

Report: CP 5.1.1/3
Harsch M., Rilinger D., 2016 b
Analytical method AFL0928/02 - Determination of [REDACTED] in BAS 750 01 F by GC
2016/1008398

Guidelines: <none>

GLP: no

Report: CP 5.1.1/4
Harsch M., 2015 c
Validation of the analytical method AFL0928/01: Determination of [REDACTED] in BAS 750 01 F by GC
2015/1180116

Guidelines: ABNT NBR 14029, EPA 830.1800, EPA 830.1000, SANCO/3030/99, CIPAC Guidelines on method validation, EC 1107/2009 (14 June 2011), 2004/10/EC, GLP Principles of the German Chemikaliengesetz (Chemicals Act), OECD Principles of Good Laboratory Practice

GLP: yes
(certified by Landesamt fuer Umwelt, Wasserwirtschaft und Gewerbeaufsicht, Mainz, Germany)

Report: CP 5.1.1/5
Rilinger D., 2016 b
Additional validation to analytical method AFL0928/01
2016/1008397

Guidelines: EC 1107/2009 of the European Parliament, 2004/10/EC, CIPAC Guidelines on method validation, SANCO/3030/99, EPA 830.1000, EPA 830.1800, ABNT NBR 14029

GLP: yes
(certified by Landesamt fuer Umwelt, Wasserwirtschaft und Gewerbeaufsicht, Mainz, Germany)

Principle of the method

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

[REDACTED]

Stability in calibration and sample solutions

[REDACTED]

Table 5-2: Stability of [REDACTED] in calibration and sample solutions

[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]

[REDACTED]

Validation

Table 5-3: Validation data

Table S.3: Validation data							

[illegible]

B.5.1.1.1. Methods in soil, water, sediment, air and any additional matrices used in support of environmental fate studies

Report:	CA 4.1.2/21 Becker M., Kamp H., 2015h BAS 750 01 F - Validation of an analytical method for the analysis of BAS 750 01 F in a mixture of acetone and olive oil using HPLC-UV 2015/1188600
Guidelines:	SANCO/3029/99 rev. 4 (11 July 2000), EEC 91/414 Annex II (Part A Section 4), EEC 91/414 Annex III (Part A Section 5)
GLP:	yes (certified by Landesamt fuer Umwelt, Wasserwirtschaft und Gewerbeaufsicht, Mainz, Germany)
Study supported:	KCP 7.1.6/2 Becker M. Kamp H., 2015 a Analytical report - BAS 750 01 F - Concentration control analyses in a mixture of acetone and olive oil 4+1 (V/V) 2014/1278739

The samples (mixture of acetone and olive oil (4+1, v/v) were diluted completely with isopropanol using appropriate volumetric flasks to obtain sample solution with test substance concentrations that match the calibration range. If the amount of test substance in the sample solution was greater than the calibration range, dilution was required with matrix solution to bring the concentration within the linear range.

Analysis was performed by HPLC-UV DAD using a Kinetix C18 column (100 x 4.6 mm, 5 µm) at ambient temperature with detection at 230 nm and external calibration. A gradient elution was used (mobile phase A: 0.1% formic acid in acetonitrile; mobile phase B: 0.1% formic acid in water).

Table 5-4: Validation data

Analyte	LOQ (mg/g)	Recovery fortification level (mg/g)	Recoveries % range (mean, n)	Repeatability % RSD (n)	Linearity	Specificity
BAS 750 01 F	5	5	94.8 – 99.1 (97.5, 5)	1.8 (5)	10.46 – 104.6 mg/L	Acceptable chromatograms presented for blank samples with and without internal standards added, calibration samples and a QC sample. No interference >30% of LOQ
		20	97.0 – 99.4 (98.2, 5)	1.0 (5)	$r^2 = 0.9999$, 7 standards, $y = 0.131779x + 0.0035$	
		60	98.9 – 99.8 (99.4, 5)	0.3 (5) Overall: 1.4 (15)		

HPLC-UV DAD is a highly specific method and additional confirmation was not necessary. Chromatograms of blank samples with and without internal standards added, calibration samples and a QC sample were provided showing no interference >30% LOQ at the retention time of interest. Accuracy was assessed at 3 fortification levels including the LOQ and in all cases the mean recovery was within the acceptable range of 70 – 110 %. To assess method precision, 5 determinations were made at each fortification level and RSDs were within the acceptable limit of 20 %. The overall RSD was 1.4 %. The linear range is appropriate for the nominal test concentrations. The LOQ of the method is 5 mg/g. The method is satisfactorily validated in accordance with SANCO/3029/99 rev.4.

B.5.1.2. Methods for the determination of residues

B.5.1.2.1. Methods in soil, water, sediment, feed and any additional matrices used in support of ecotoxicology studies

Report:	KCP 10.2.1/1 [REDACTED] 2014 a BAS 750 01 F - Rainbow trout, acute toxicity test 2014/1117112
Guidelines:	SANCO/3029/99 rev.4
GLP:	yes
Report:	KCP 10.2.1/3 Turek T., 2015 a BAS 750 01 F - Daphnia magna acute immobilization test 2014/1117111
Guidelines:	SANCO/3029/99 rev.4
GLP:	yes
Report:	KCP 10.2.1/5 Turek T., 2015 b BAS 750 01 F - Pseudokirchneriella subcapitata SAG 61.81 - Growth inhibition test 2014/1117110
Guidelines:	SANCO/3029/99 rev.4
GLP:	yes
Studies supported:	<i>KCP 10.2.1/1</i> [REDACTED] 2014 a <i>BAS 750 01 F - Rainbow trout, acute toxicity test</i> 2014/1117112 <i>KCP 10.2.1/3</i> <i>Turek T., 2015 a</i> <i>BAS 750 01 F - Daphnia magna acute immobilization test</i> 2014/1117111 <i>KCP 10.2.1/5</i> <i>Turek T., 2015 b</i> <i>BAS 750 01 F - Pseudokirchneriella subcapitata SAG 61.81 - Growth inhibition test</i> 2014/1117110

The sample was prepared by application of the water sample to an ENVI-18 (3mL, 500 mg) column previously washed twice with 5mL methanol and twice with 5 mL water. The column was dried for 5 minutes and eluted with 5 mL methanol. The eluate was dried and redissolved in acetonitrile.

Analysis was performed by HPLC-DAD using a Pursuit XRs 3 C8 column (150 x 4.6 mm) with UV detection at 230 nm and external calibration. The mobile phase was acetonitrile: 0.05% solution of orthophosphoric acid (90:10 v/v for rainbow trout and pseudokirchneriella subcapitata; 55:45 v/v for daphnia magna).

Table 5-5: Validation data

Matrix	Analyte	LOQ (mg/L)	Recovery fortification level (mg/L)	Recoveries % range (mean, n)	Repeatability % RSD (n)	Linearity	Specificity
Water	BAS 750 F	0.02	0.02 1.87	95 – 110 (100, 5) 95 - 98 (93, 5)	5.0 (5) 3.6 (5) Overall: 6.1 (10)	1 – 100 mg/L $r = 0.9997$, 7 standards, $y = 0.05308x + 0.06023$	Acceptable chromatograms presented for control and test item. No interference >30% of LOQ

HPLC-DAD is a highly specific method and additional confirmation was not necessary. Chromatograms of the control and test item were provided showing no interference >30% LOQ at the retention time of interest. Accuracy was assessed at 2 fortification levels and in all cases the mean recovery was within the acceptable range of 70 – 110 %. To assess method precision, 5 determinations were made at each fortification level and RSDs were within the acceptable limit of 20 %. The overall RSD was 6.1 %. Acceptable linearity has been demonstrated in the range 1 – 100 µg/mL. The LOQ of the method is 0.002 mg/L. The method is satisfactorily validated in accordance with SANCO/3029/99 rev.4.

Report: KCP 10.4.1.2/1
Hamberger A., 2015 a
BAS 752 AM F - A field study to investigate effects on earthworm fauna in Southern Germany
2015/1000261

Guidelines: SANCO/3029/99 rev.4

GLP: yes

Study supported: KCP 10.4.1.2/1
Hamberger A., 2015 a
BAS 752 AM F - A field study to investigate effects on earthworm fauna in Southern Germany
2015/1000261

Principle of the method

In soil: A 5 g soil sample was extracted with 40 mL of a mixture of acetonitrile/water (70/30, v/v). After centrifugation an aliquot of 10 mL was taken (extract 1). The same extraction procedure was repeated and after centrifugation a second aliquot of 10 mL (extract 2) was combined with extract 1 and thoroughly mixed. Analysis was performed by LC-MS/MS on a Phenomenex Luna 5 μ C18 column (150 x 3 mm, 5 μ m) at 40 °C with external standardisation and ESI⁺ detection monitoring two mass transitions (m/z 398 \rightarrow 162 (quantification) and 398 \rightarrow 182 (confirmation)).

In calcium chloride extracts: 10 g soil sample was added to a 250 mL flask and 100 mL of a 10 mM calcium chloride solution was added and the tube shaken and centrifuged. Analysis was performed by HPLC-MS/MS using the same conditions as for soil.

Soil characteristics

Table 5-6: Soil characteristics

Cation exchange capacity (meq/100g)	8.1
Soil class (USDA)	Silt loam
Total Organic Carbon (%)	1.4
Total Carbon (%)	1.4
pH (CaCl₂)	5.23
Water holding capacity	32.0

Matrix effects

Blank extracts from each matrix were spiked with defined concentrations of BAS 750 F. Matrix effects were calculated as the mean ratio of calculated concentrations of standards in matrix against standards in solvent for soil extracts and against CaCl₂ for standards in CaCl₂ soil extracts. Measurements were taken across all concentrations (0.05 – 10 ng/mL) and for both mass transitions.

Table 5-7: Matrix effects

Analyte	Matrix	Mass transition (m/z)	Matrix effect (%)
BAS 750 F	Soil	398 \rightarrow 182	-30
		398 \rightarrow 162	-27
	Calcium chloride extracts	398 \rightarrow 182	0
		398 \rightarrow 162	+1

Significant matrix effects were observed for BAS 750 F in soil samples. Therefore matrix-matched standards were used for the analysis of BAS 750 F in soil samples. No significant matrix effects were observed for BAS 750 F in CaCl₂ nevertheless matrix calibration was used for analysis of BAS 750 F in CaCl₂ extracts.

Stability of extracts

As samples were analysed within 24 hours of extraction, the storage stability in the final extract was not required to be determined.

Validation data

Table 5-8: Validation data

Matrix	Analyte	LOQ (mg/kg)	Mass transition (m/z)	Recovery fortification level (mg/kg)	Recoveries % range (mean, n)	Repeatability % RSD (n)	Linearity	Specificity
Soil	BAS 750 F	0.002	398 → 182	0.002	90 – 99 (95, 5)	4 (5)	0.025 – 10 ng/mL [Approx. 0.0004 – 0.16 mg/kg] 8 standard s, y = 6.98×10^3 + 9.06, r = 0.9999	Acceptable chromatograms presented for standard in blank matrix, untreated soil sample, fortified sample and treated soil sample No interference >30% of LOQ Identity confirmed by additional mass transition
			398 → 162	0.002	95 – 104 (100, 5)	4 (5)		
				0.6	102 – 105 (104, 5)	1 (5) Overall: 6 (10)		
				0.6	101 – 111 (107, 5)	3 (5) Overall: 5 (10)		
Calcium chloride extracts	BAS 750 F	0.002	398 → 182	0.002	78 – 85 (80, 3)	5 (3)	0.025 – 100 ng/mL [Approx. 0.00025 – 1 mg/kg] 9 standard s, y = 2.4×10^5 + 2.31×10^4 , r = 0.9976	Acceptable chromatograms presented for standard in blank matrix, untreated soil sample, fortified sample and treated soil sample No interference >30% of LOQ Identity confirmed by additional mass transition
			398 → 162	0.002	98 – 101 (100, 3)	2 (3)		
				0.02	106 – 110 (108, 3)	2 (3)		
				0.1	88 – 98 (92, 3)	6 (3) Overall: 13 (9)		
				0.02	110 – 115 (113, 3)	2 (3)		
				0.1	91 – 96 (94, 3)	3 (3) Overall: 8 (9)		

HPLC-MS/MS is a highly specific technique and two mass transitions (3 ions) are monitored for BAS 750 F as required by the guidance. Chromatograms of standard in blank matrix, untreated soil sample, fortified sample and treated soil sample have been presented showing no interferences >30% LOQ at the retention time of

interest. Accuracy was assessed at 2 fortification levels for each matrix of interest corresponding to LOQ and 10xLOQ. In all cases the mean recovery was within the acceptable range of 70 – 110 %. To assess method precision, between 3 and 5 determinations were made at each fortification level and the RSDs were within the acceptable limit of 20%. The overall RSDs were between 5 – 13 %. The linear range is appropriate for the nominal test concentrations in calcium chloride extracts but does not cover fortification at 0.6 mg/kg in soil. Matrix-matched standards were used. The LOQ of the method is 0.002 mg/kg. The method is satisfactorily validated in accordance with SANCO/3029/99 rev.4 for soil. Although only 3 determinations were made at each fortification for calcium chloride extracts, the RMS regards the method as sufficiently validated for the purposes of the regulatory process.

Report: KCP 10.4.1.2/2
Schulz L., 2015 a
Effects of BAS 750 01 F on earthworms under field conditions
2015/1000163

Guidelines: SANCO/3029/99 rev.4

GLP: yes

Study supported: KCP 10.4.1.2/2
Schulz L., 2015 a
Effects of BAS 750 01 F on earthworms under field conditions
2015/1000163

Principle of the method: Soil analysis

A 5 g soil sample was extracted with 40 mL of a mixture of acetonitrile/water (70/30, v/v) and centrifuged (extract 1). The same extraction procedure was repeated (extract 2). Both the supernatants were combined in a 100 mL volumetric flask which was subsequently filled to the mark with extraction solution.

Principle of the method: Soil desorption experiments

5 g soil was added to 50 mL of 0.01 M CaCl₂, shaken and centrifuged. The supernatant was decanted and diluted 1:1 with acetonitrile.

Analysis was performed by LC-MS/MS using an Aquasil C18 – Thermo column (150 x 3 mm, 3 µm) at 25 °C with MRM monitoring two mass transitions (m/z 398 → 133 (quantification) and 398 → 182 (confirmation)) and external calibration. A gradient elution was used (mobile phase A: 0.1 % formic acid in water; mobile phase B: 1 % formic acid in acetonitrile).

Matrix effects

The soil specimens were checked for matrix effects calculated as the ratio of calculated concentrations of a solvent calibration standard with a matrix-matched calibration standard at LOQ level ($c = 0.52$ ng/mL).

Table 5-9: Matrix effects

Analyte	Matrix	Mass transition (m/z)	Matrix effect (%)
BAS 750 F	Soil	398 → 182	102
		398 → 133	97

No significant matrix effects (< 3 %) were observed. Matrix matched calibration standards were not needed.

Stability of working solutions

The stability of BAS 750 F in working solutions was tested by determination of the quotient (peak area/concentration) at a concentration of 1 ng/mL against freshly prepared standards.

Table 5-10: Stability of working solutions

Matrix	Analyte	Mass transition	Days of storage	Quotient (peak area/concentration)
Acetonitrile/water (70/30, v/v)	BAS 750 F	398 → 182	27	152184
			0	174868

The working solutions of BAS 750 F are considered to be stable in acetonitrile/H₂O; 70/30 (v/v) on storage for at least 27 days.

Validation data

Table 5-11: Validation data

Matrix	Analyte	LOQ (mg/kg)	Mass transition (m/z)	Recovery fortification level (mg/kg)	Recoveries % range (mean, n)	Repeatability % RSD (n)	Linearity	Specificity
Spray targets	BAS 750 F	1.115	398 → 182	1.115	93.09 – 126.82 (110.67, 4)	14.3 (4)	0.1039 to 5.195 ng/mL	Acceptable chromatograms provided for standard solutions, untreated specimen, fortified specimen and treated specimen. No interference >30% LOQ
				2.230	89.28 – 175.16 (118.28, 4)	33 (4)	[Approx. 0.002 – 0.1 mg/kg]	
				4.46	107.29 – 113.65 (110.63, 4)	2.5 (4) Overall: 19.7 (12)	8 standards, y = 11834x + 440.1, r = 0.99983	
Soil cores	BAS 750 F	0.200	398 → 182	0.200	135.90 – 184.40 (151.08, 4)	14.9 (4)	0.1039 to 5.195 ng/mL	Acceptable chromatograms provided for standard solutions, untreated specimen, fortified specimen and treated specimen. No interference >30% LOQ
				0.400	119.33 – 137.50 (126.75, 4)	6.1 (4)	[Approx. 0.002 – 0.1 mg/kg]	
				0.800	102.51 – 146.25 (123.61, 5)	16.5 (4) Overall: 15.5 (12)	8 standards, y = 11834x + 440.1, r = 0.99983	
Desorption	BAS 750 F	0.200	398 → 182	0.200	135.90 – 184.40 (151.08, 4)	14.9 (4)	0.01039 to 2.078 ng/mL	Acceptable chromatograms provided for standard solutions, untreated specimen, fortified specimen and treated specimen. No interference >30% LOQ
				0.400	119.33 – 137.50 (126.75, 4)	6.1 (4)	[Approx. 0.0002 – 0.04 mg/kg]	
				0.800	102.51 – 146.25 (123.61, 4)	16.5 (4) Overall: 15.5 (4)	8 standards, y = 7078x + 517.6, r = 0.99992	

Additional recovery data

The preparation of “laboratory procedural recovery specimens” was performed by fortification of untreated soil samples with BAS 750 F. Single determinations of recovery were taken at each fortification level as shown in Table 5-12.

Table 5-12: additional recovery data

Matrix	Mass transition (<i>m/z</i>)	LOQ	Recovery fortification level	Recoveries % (n)	Repeatability % RSD (n)
Spray targets	398 → 182	0.01 mg kg	0.01 0.6 7.1	90.2 (1) 95.3 (1) 99.0 (1)	Overall: 4.6 (3)
Soil cores	398 → 182	0.01 mg/kg	0.01 mg/kg 0.1 mg/kg 1.4 mg/kg	92.2 (1) 88.5 (1) 84.4 (1)	Overall: 4.4 (3)
Desorption	398 → 182	0.1 µg/L	0.1 µg/L 1 µg/L 10 µg/L 26 µg/L	93.5 93.3 86.0 87.3	Overall: 4.8 (3)

Validation summary

HPLC-MS/MS is a highly specific technique and two mass transitions (3 ions) are monitored for BAS 750 F as required by the guidance. Chromatograms of standard solutions, untreated specimen, fortified specimen and treated specimen have been presented showing no interferences >30% LOQ at the retention time of interest. Accuracy was assessed at 3 fortification levels for each matrix of interest. In all cases the mean recovery was outside the acceptable range of 70 – 110 % in every case however there were only minor deviation in all cases except for fortification at 0.200 mg/kg in soil cores. To assess method precision, 4 determinations were made at each fortification level, less than the 5 in accordance with the guidance and the RSDs were within the acceptable limit of 20% except for the 2.230 mg/kg fortification in spray targets which was 33 %, however this is due to an outlier at 3.9 mg/kg. The overall RSDs were between 15.5 and 19.7 %. The linear range does not cover the fortification concentrations. Matrix-matched standards were used. The LOQ of the method is 0.2 mg/kg for soil cores and desorption experiments and 1.115 mg/kg for spray targets. The additional recovery data generated was within the range of 70 – 110 %. Therefore, although the method is not fully validated in accordance with SANCO/3029/99 rev.4, the RMS regards the method as sufficiently validated for the purposes of the regulatory process.

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

Methods for post-approval control and monitoring purposes are reported in Volume 3 CA B-5 of this DAR.

B.5.3. REFERENCES RELIED ON

Data Point	Author(s)	Year	Title Company Report No. Source (where different from company) GLP or GEP status Published or not	Vertebrate study Y/N	Data protection claimed Y/N	Justification if data protection is claimed	Owner	Previous evaluation
KCA 4.1.2/21	Becker M. Kamp H.	2015 h	BAS 750 01 F - Validation of an analytical method for the analysis of BAS 750 01 F in a mixture of acetone and olive oil using HPLC-UV 2015/1188600 BASF SE, Ludwigshafen/Rhein, Germany Fed.Rep. yes Unpublished	No	Yes	Data for first Approval	BASF	N.A
KCP 5.1.1/1	Harsch M.	2014 a	Determination of the active ingredient Reg.No. 5834378 in EC-Formulation 2014/1097912 BASF SE, Limburgerhof, Germany Fed.Rep. no Unpublished	No	No	Not applicable	BASF	N.A.
KCP 5.1.1/2	Harsch M.	2014 b	Validation of the analytical method AFL0909/01: Determination of the Active Ingredient Reg.No. 5834378 in EC-	No	Yes	Data for first Approval	BASF	N.A.

			Formulation 2014/1097921 BASF SE, Limburgerhof, Germany Fed.Rep. yes Unpublished					
KCP 5.1.1/3	Harsch M. Rilinger D.	2016 b	Analytical method AFL0928/02 - Determination of [REDACTED] [REDACTED] in BAS 750 01 F by GC 2016/1008398 BASF SE, Limburgerhof, Germany Fed.Rep. no Unpublished	No	No	Not applicable	BASF	N.A
KCP 5.1.1/4	Harsch M.	2015 c	Validation of the analytical method AFL0928/01: Determination of [REDACTED] [REDACTED] in BAS 750 01 F by GC 2015/1180116 BASF SE, Limburgerhof, Germany Fed.Rep. yes Unpublished	No	Yes	Data for first Approval	BASF	N.A
KCP 5.1.1/5	Rilinger D.	2016 b	Additional validation to analytical method AFL0928/01 2016/1008397 BASF SE, Limburgerhof, Germany Fed.Rep. yes	No	Yes	Data for first Approval	BASF	N.A

			Unpublished					
KCP 10.2.1/1	■■■■■ ■	2014 a	BAS 750 01 F - Rainbow trout, acute toxicity test 2014/1117112 ■■■■■ ■ ■■■■■ ■■■■ ■■■■■ ■■■■■■■■ yes Unpublished	Yes	Yes	Data for first Approval	BASF	N.A.
KCP 10.2.1/3	Turek T.	2015 a	BAS 750 01 F - Daphnia magna acute immobilization test 2014/1117111 Institute of Industrial Organic Chemistry, Pszczyna, Poland yes Unpublished	No	Yes	Data for first Approval	BASF	N.A.
KCP 10.2.1/5	Turek T.	2015 b	BAS 750 01 F - Pseudokirchneriella subcapitata SAG 61.81 - Growth inhibition test 2014/1117110 Institute of Industrial Organic Chemistry, Pszczyna, Poland yes Unpublished	No	Yes	Data for first Approval	BASF	N.A.
KCP 10.4.1.2/1	Hamberger A.	2015 a	BAS 752 AM F - A field study to investigate effects on earthworm fauna in Southern Germany 2015/1000261 Eurofins	No	Yes	Data for first Approval	BASF	N.A.

			Agroscience Services EcoChem GmbH, Niefern- Oeschelbronn, Germany Fed.Rep. yes Unpublished					
KCP 10.4.1.2/2	Schulz L.	2015 a	Effects of BAS 750 01 F on earthworms under field conditions 2015/1000163 BioChem agrar Labor fuer biologische und chemische Analytik GmbH, Gerichshain, Germany Fed.Rep. yes Unpublished	No	Yes	Data for first Approval	BASF	N.A.