BAS 650F; EPA PC Code 119210
BASF Corporation; EPA Company Code
ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT

Data Requirement: EPA Guideline: 835.6200
OECD Data Point: IIA 4.5

Test material:

Common name: BAS 650F.
Chemical name:
IUPAC: 5-Ethyl-6-octyl[1,2,4]triazolo[1,5-α]pyrimidin-7-amine.
CAS: 5-Ethyl-6-octyl[1,2,4]triazolo[1,5-a]pyrimidin-7-amine.
CAS No: 865318-97-4.
Synonyms: Ametoctradin; M650F00; Reg. No. 4993353.
SMILES string: N1C=NN2C=1N=C(CC)C(CCCCCCCC)=C2N (EpiSuite version 4.0).

Primary Reviewer: Lynne Binari
Cambridge Environmental
Signature: Lynne Binari
Date:

Secondary Reviewer: Kathleen Ferguson
Cambridge Environmental
Signature: Kathleen P. Ferguson
Date:

QC/QA Manager: Joan Gaidos
Cambridge Environmental
Signature: Joan Gaidos
Date:

Final Reviewer:
EPA
Signature:
Date: 6/2/11

For MRIDs 47700037 and 47700038, referenced page numbers, except for page 1, are located at the bottom, right-hand corner of the page; *i.e.* "page * of 50" for MRID 47700037 and "page * of 56" for MRID 47700038. For MRID 48435701, referenced page numbers, except for page 1, are located at the top, right-hand corner of the page; *i.e.* "page * of 221".

ANALYTICAL METHOD: EPA MRID No. 47700037. Class, T., and I-C. Beck. 2008. Validation of the BASF analytical method 574/0 for the determination of residues of BAS 650 F in tap and surface water samples. Unpublished study performed by PTRL Europe GmbH, Ulm, Germany; sponsored by BASF SE, Limburgerhof, Germany; and submitted by BASF Corporation, Research Triangle Park, North Carolina; 50 pages (pp. 1-2, 5). PTRL Europe Report and Study No.: P 1519 G (pp. 1, 4). BASF Report No.: 250801_1 and Registration Document No.: 2008/1035627 (p. 1). Experimental start date June 9, 2008, and completion date June 16, 2008 (p. 5). Final report issued July 16, 2008 (p. 1).

ANALYTICAL METHOD: EPA MRID No. 47700038. Class, T., and I-C. Beck. 2008. Validation of analytical method L0113 for the determination of residues of the BAS 650 F metabolites M650F01, M650F02, M650F03 and M650F04 in water samples. Unpublished study performed by PTRL Europe GmbH, Ulm, Germany; sponsored by BASF SE, Limburgerhof, Germany; and submitted by BASF Corporation, Research Triangle Park, North Carolina; 56 pages (pp. 1-2, 5). PTRL Europe Report and Study No.: P 1483 G (pp. 1, 4). BASF Report No.: 250801 and Registration Document No.: 2008/1017005 (p. 1). Experimental start date June 3, 2008, and completion date July 30, 2008 (p. 5). Final report issued July 31, 2008 (p. 1).

INDEPENDENT LABORATORY VALIDATION: EPA MRID No. 48435701. Perez, R. and S. Perez. 20011. Independent laboratory validation of BASF analytical methods 574/0 and L0113, "Method for determination of residues of BAS 650 F in water samples" and "Methods for the determination of BAS 650 F metabolites M650F01, M650F02, M650F03 and M650F04 in water samples". Unpublished study performed by ADPEN Laboratories, Inc., Jacksonville, Florida; sponsored and submitted by BASF Corporation, Research Triangle Park, North Carolina; 221 pages (p. 1). ADPEN Study No.: 2K10-ADPEN-903-0817. BASF Study No.: 382027 and Registration Document No.: 2010/7014027. Experimental start date September 21, 2010, and completion date January 6, 2011 (p. 7). Final report issued March 3, 2011 (p. 1).

EXECUTIVE SUMMARY

These methods are designed for the quantitative determination of residues of BAS 650F and its products M650F01, M650F02, M650F03 and M650F04 in drinking and surface water. The methods were created by BASF Corporation and validated by PTRL Europe GmbH. An independent laboratory validation (ILV), performed by ADPEN Laboratories, Inc., was submitted with the methods.

The Agency finds that this method meets the criteria for a scientifically valid method and is **acceptable** for (applicable residues).

Method Summary: BAS 650F is extracted from water by shaking with methylene chloride, with an aliquot of the organic phase then taken to dryness and the residues reconstituted in acetonitrile:water (50:50, v:v). BAS 650F products M650F01, M650F02, M650F03 and M650F04 are extracted from water using Strata-X-C solid-phase cartridges and elution with acetonitrile:25% ammonia. The resulting eluate is taken to dryness and the residues reconstituted in acetonitrile:water (10:90, v:v). BAS 650F and its products, M650F01, M650F02, M650F03 and M650F04, are quantified by LC/MS/MS. The ILV utilized limits of quantitation (LOQ) and detection (LOD) of 0.05 µg/kg and 0.01 µg/kg, respectively, for BAS 650F and its products (p. 15 of MRID 48435701). The ECM LODs were 0.0025 µg/kg (BASF) and 0.005 µg/kg (PTRL) for parent BAS 650F and 0.01 µg/kg for M650F01, M650F02, M650F03 and M650F04 (pp. 12, 31 of MRID 47700037; pp. 14, 53 of MRID 47700038).

METHOD ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS

No significant method deficiencies were apparent.

COMPLIANCE

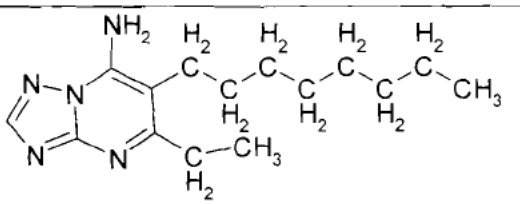
The methods were conducted in compliance with German Principles of Good Laboratory Practice (GLP); Anhang 1 zu § 19a des Chemikalien-gesetzes (ChemG) i.d.F. vom 20.06.2002, which are in accordance with USEPA GLP Standards 40 CFR, Part 160 (p. 3 of MRIDs 47700037 and 47700038). Signed and dated statements of No Data Confidentiality, GLP, Quality Assurance and Compliance to Guidelines and Certification were provided (pp. 2-5 of MRIDs 47700037 and 47700038).

A. BACKGROUND INFORMATION

BAS 650F is a new fungicide developed for use against *Phytophthora* in tomatoes and *Plasmopara* in grapes (p. 25 of MRID 47700037).

TABLE A.1. Test Compound Nomenclature

Parameter	Value
Common name	BAS 650F.
Company experimental name	M650F00.
IUPAC name	5-Ethyl-6-octyl[1,2,4]triazolo[1,5-α]pyrimidin-7-amine.
CAS Name	5-Ethyl-6-octyl[1,2,4]triazolo[1,5-a]pyrimidin-7-amine.

TABLE A.1. Test Compound Nomenclature	
Parameter	**Value**
CAS #	865318-97-4.
Structure	

Information obtained from Appendix 1, p. 38 in MRID 47700035.

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound	
Parameter	**Value**
Molecular formula	$C_{15}H_{25}N_5$
Molecular weight	275.4 g/mol.
Melting point/range (°C)	Not reported.
pH	Not reported.
Density (g/cm^3)	Not reported.
Water solubility at 20 °C (mg/L)	Not reported.
Solvent solubility at 20 °C (mg/L)	Not reported.
Vapor pressure at __ °C (torr)	Not reported.
Dissociation constant (pK_a)	Not reported.
Octanol/water partition coefficient	Not reported.
UV/visible absorption spectrum (nm)	Not reported.

Information obtained from p. 20 of MRID 47700037.

B. MATERIALS AND METHODS

B.1. Principle of Method

BAS 650F is extracted from water by shaking with methylene chloride. An aliquot of the organic phase, containing BAS 650F, is taken to dryness and the residues reconstituted in acetonitrile:water (50:50, v:v) for analysis. BAS 650F products M650F01, M650F02, M650F03 and M650F04 are extracted from water using Strata-X-C solid-phase extraction (SPE) cartridges eluted with acetonitrile:25% ammonia. The eluate, containing all four products, is taken to dryness and the residues reconstituted in acetonitrile:water (10:90, v:v) for analysis. Analytes are separated and quantified by LC/MS/MS using a Waters XTerra C18 LC column, electrospray ionization (ESI^+) and multiple reaction monitoring (MRM). Per analyte, two MRM parent-daughter ions for quantitation and confirmation are monitored.

TABLE B.1. Summary Parameters for the Analytical Method Used for the Quantitation of Chemical Residues in Matrices Studied	
Parameter	**Value**
Method IDs	BAS 650F: BASF method 574/0 (p. 8 of MRID 47700037). BAS 650F products M650F01, M650F02, M650F03, M650F04: BASF method L0113 (p. 10 of MRID 47700038).
Analyte(s)	Method 574/0: BAS 650F. Method L0113: M650F01, M650F02, M650F03, M650F04.
Extraction solvent/technique	BAS 650F: Water aliquot (100 g) is treated with sodium chloride (10 g; p. 29 of MRID 47700037). Following dissolution, water is extracted with methylene chloride (25 mL) via mechanical shaking for 30 minutes. BAS 650 products: Water adjusted to *ca.* pH 7 with HCl or NH_3, as needed (p. 51 of MRID 47700038). Water aliquot (10 g) then loaded onto Strata-X-C SPE cartridge preconditioned with acetonitrile followed by water. Loaded cartridge is dried under vacuum, then analytes are eluted with acetonitrile:25% NH_3 (90:10, v:v, 10 mL).
Cleanup strategies	BAS 650F: Following phase separation, organic phase aliquot (10 mL) is concentrated to dryness via rotary evaporation (*ca.* 40°C). Resulting residues are reconstituted in acetonitrile:water (50:50, v:v) for analysis. BAS 650 products: SPE eluate is concentrated via rotary evaporation (*ca.* 50°C), then taken to dryness under nitrogen. Resulting residues are reconstituted in acetonitrile:water (10:90, v:v) for analysis.
Instrument/Detector	Agilent HPLC system, equipped with a Waters XTerra C18 column (4.6 x 50 mm, 3.5 µm) and Applied Biosystems Sciex API triple quadrupole LC/MS/MS system with TurboIonspray (ESI^+) source and MRM. For parent BAS 650F analysis, LC column preceded by a Phenomenex C18 RP (3 x 4 mm) guard column.

C. RESULTS AND DISCUSSION

C.1. Recovery Results Summary

TABLE C.1. Recovery Results from Method Validation for the Determination of Residues in drinking and surface water			
Analyte	**Spiking Level (µg/kg)**	**Mean Recoveries Obtained (%)**	**Relative Standard Deviation**
Drinking (Tap) Water[1]			
BAS 650F	0.05	102	5
	0.50	107	4
M650F01	0.05	83	9

		YES	NO	REVIEW FURTHER
C.	**Are all steps in the method scientifically sound?**	x		
D.	**Is a confirmatory method or technique provided?**	x		
E.	**Check the category below which best describes this ECM.** [*Is the data supplied in the method package satisfactory or deficient in any way? If there are deficiencies, are the deficiencies major or minor? Note whether deficiencies are with the method procedure, whether they are with respect to guidelines, and whether they affect the review classification.*]			
1.	**Satisfactory**	x		
2.	**Major Deficiencies**			
3.	**Minor Deficiencies**			

IX. Recommendations

- Section ***V. Representative Chromatograms*** **A. 4.** Linear regression analysis was used to determined calibration curves for both quantitation and confirmation ions of BAS 650F, M650F01, M650F02 and M650F04 and the confirmation ion of M650F03; however, quadratic regression analysis was used to determine the calibration curve for the quantitation ion of M650F03.

  The ILV used linear regression analysis for both quantitation and confirmation ions of BAS 650F and it products to determine calibration curves.

- Section ***V. Representative Chromatograms*** **A. 5.** Recovery results for quantitation ion of M650F03 could not be verified by the primary reviewer. Using quadratic regression analysis is atypical, but was used to obtain a "better coefficient" (Figure 3, p. 21 of MRID 47700038. Using linear regression analysis (R^2 = 1.0000) for the calibration curve and the provided chromatogram results, percent M650F03 (176 m/z) recoveries for samples P1483-37 (0.05 µg/kg surface water) and P1483-42 (0.5 µg/kg surface water) were 92% and 106%, respectively,

as compared to 94% and 105%, respectively, in the ECM report (Figure 15, p. 35 of MRID 47700038; DER Attachment 2).

Recovery results for BAS 650F in 0.05 and 0.5 µg/kg fortified drinking (tap) water and M650F01, M650F02, M650F03 (204 m/z) and M650F04 in 0.05 and 0.5 µg/kg fortified surface (river) water, for provided chromatograms, were verified by the primary reviewer (DER Attachment 2).

ILV recovery results for BAS 650F and its products in 0.05 and 0.50 µg/kg fortified surface (pond) water, for provided chromatograms, were verified by the primary reviewer (DER Attachment 2).

- Section ***VI. Good Laboratory Practice (GLP) Standards*** **A.** The studies were conducted in compliance with German Principles of GLP; Anhang 1 zu § 19a des Chemikalien-gesetzes (ChemG) i.d.F. vom 20.06.2002, which are in accordance with FIFRA GLP standards (p. 3 of MRID 47700037 and 47700038).

- Section ***VII. Independent Lab Validation (ILV)*** **D.** The ILV limit of detection (LOD) for BAS 650F is 2-4x higher than LODs stated in the ECM report.

Name and Dated Signature of Primary Reviewer

Lynne Binari

5/2/11

Name(s) and Dated Signature(s) of Secondary Reviewer(s)

Kathleen P. Ferguson

5/3/11

TABLE C.1. Recovery Results from Method Validation for the Determination of Residues in drinking and surface water

Analyte	Spiking Level (µg/kg)	Mean Recoveries Obtained (%)	Relative Standard Deviation
	0.50	84	4
M650F02	0.05	88	4
	0.50	94	4
M650F03	0.05	99	2
	0.50	101	6
M650F04	0.05	88	7
	0.50	91	2
Surface (River) Water[1]			
BAS 650F	0.05	100	2
	0.50	106	2
M650F01	0.05	81	6
	0.50	84	2
M650F02	0.05	95	4
	0.50	90	3
M650F03	0.05	96	5
	0.50	110	3
M650F04	0.05	99	1
	0.50	94	2
Water Combined[2]			
BAS 650F	0.05	101	3
	0.50	106	3
M650F01	0.05	83	7
	0.50	85	3
M650F02	0.05	91	5
	0.50	92	4
M650F03	0.05	100	6
	0.50	105	6
M650F04	0.05	93	8
	0.50	92	3

1 Quantitation ion results from Tables 1-2, pp. 14-15 of MRID 47700037 and Tables 1-2, pp. 17-18 of MRID 4700038.
2 Combined water results (quantitation and confirmation ions) from DER Attachment 2.

C.1.1. Method Characteristics

TABLE C.2. Method Characteristics	
Parameter	**Value**
Analyte(s)	Method 574/0: BAS 650F. Method L0113: M650F01, M650F02, M650F03, M650F04.
Limit of Quantitation (LOQ)	Both methods: 0.05 µg/kg.
Limit of Detection (LOD)	Method 574/0: 0.0025 µg/kg (BASF, p. 31 of MRID 47700037), 0.005 µg/kg (PTRL, p. 11 of MRID 47700037). Method L0113: 0.01 µg/kg (BASF, PTRL; pp. 15, 53 of MRID 47700038).
Accuracy/Precision at LOQ	Acceptance criteria (EFED-ECM 2, Version 1, December 2010, p. 5) were met with matrix spike recoveries ranging from 77% to 108% (quantitation ion results) and relative standard deviations of ≤9% (Tables 1-2, pp. 14-15 of MRID 47700037; Tables 1-2, pp. 17-18 of MRID 47700038).
Reliability of the Method/[ILV]	Each method was successfully validated (ILV) in one trial (p. 20 of MRID 48435701).
Linearity	Linear regression with both quantitation and confirmatory ions for BAS 650F, M650F01, M650F02, M650F04 and confirmatory ion (204 m/z) for M650F03: r = 0.9992-0.9999 (Figure 1, p. 16 of MRID 47700037; Figures 1-4, pp. 19-22 of MRID 47700038). Quadratic regression with quantitation ion (176 m/z) for M650F03: r = 0.9999 (Figure 3, p. 21 of MRID 47700038).
Specificity	Method 574/0: quantifies BAS 650F. Method L0113: separately quantifies M650F01, M650F02, M650F03 and M650F04.

C.2. Independent Laboratory Validation (ILV)

The ILV was conducted in accordance with EPA OPPTS Guidelines 860.1340, 835.6200 and 835.7100, Subdivision N Guideline §162-4 and European Commission SANCO/3029/99 rev. 4 (2000), and in compliance with EPA GLP Standards, 40 CFR part 160 (pp. 12, 21 of MRID 48435701).

TABLE C.3. Recovery Results of the Method Obtained by an Independent Laboratory Validation for the Determination of Residues in surface (pond) water[1]

Analyte	Spiking Level (µg/kg)	Mean Recoveries Obtained (%)	Relative Standard Deviation
BAS 650F	0.05	94.5	6.3
	0.50	105.2	5.5
M650F01	0.05	102.4	5.3
	0.50	107.7	3.6

TABLE C.3. Recovery Results of the Method Obtained by an Independent Laboratory Validation for the Determination of Residues in surface (pond) water[1]

Analyte	Spiking Level (µg/kg)	Mean Recoveries Obtained (%)	Relative Standard Deviation
M650F02	0.05	106.9	4.5
	0.50	102.4	6.5
M650F03	0.05	107.8	6.3
	0.50	106.8	5.7
M650F04	0.05	77.9	9.7
	0.50	79.3	11.9

1 Quantitation ion results from Appendix B, pp. 106, 108, 110, 112, 114 of MRID 48435701.

D. CONCLUSION

BASF method 574/0 is designed for the quantitative determination of residues of parent BAS 650F and BASF method L0113 for the quantitative determination of residues of BAS 650 F products M650F01, M650F02, M650F03 and M650F04 in water. The Agency finds that this method meets the criteria for a scientifically valid method and is **acceptable** for applicable residues. However, the independent laboratory validation limit of detection (LOD) for BAS 650F is 2-4x higher than LODs stated in the ECM report. Attachment 1 contains the method review checklist to determination of residues of BAS 650 F and its metabolites, M650F01, M650F02, M650F03 and M650F04 in water samples.

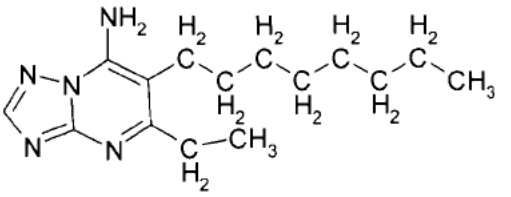
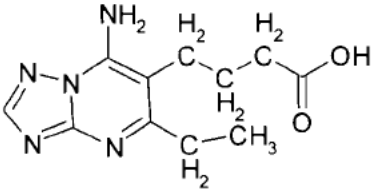
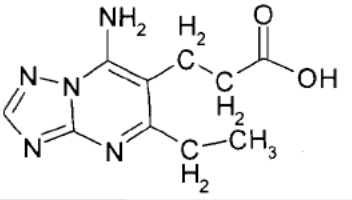
ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

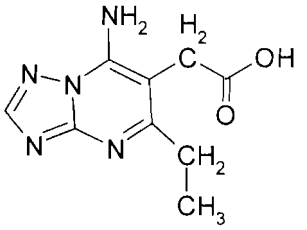
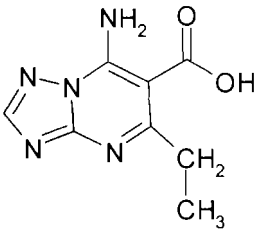
ENVIRONMENTAL CHEMISTRY METHOD (ECM) STANDARD EVALUATION PROCEDURE (SEP) CHECKLIST: BACKGROUND AND INITIAL REVIEW INFORMATION

For MRIDs 47700037 and 47700038, referenced page numbers, except for page 1, are located at the bottom, right-hand corner of the page; *i.e.* "page * of 50" for MRID 47700037 and "page * of 56" for MRID 47700038. For MRID 48435701, referenced page numbers, except for page 1, are located at the top, right-hand corner of the page; *i.e.* "page * of 221".

I. Background Information

A.	**Title of Methods**	Technical procedure of method no. 574/0 for the determination of reg. no. 4993353 residues in water by HPLC/MS (p. 21 in MRID 47700037). Technical procedure: method for the determination of BAS 650 F metabolites M650F01, M650F02, M650F03 and M650F04 in water samples (p. 43 in MRID 47700038).	
B.	**ECM No.**	[*Leave blank. This is for BEAD ECB's use.*]	
C.	**MRID No.**	47700037 (parent BAS 650F). 47700038 (BAS 650F products M650F01, M650F02, M650F03 and M650F04).	
D.	**Matrix**	Water.	
E.	**Analyte(s) detected**	**Parent:**	
		Common name:	BAS 650F.
		IUPAC name:	5-Ethyl-6-octyl[1,2,4]triazolo[1,5-α]pyrimidin-7-amine.
		CAS name:	5-Ethyl-6-octyl[1,2,4]triazolo[1,5-a]pyrimidin-7-amine.
		CAS No:	865318-97-4.
		Synonyms:	Ametoctradin; M650F00; Reg. No. 4993353.

	
Metabolite 01:	
Common name:	M650F01
IUPAC name:	4-(7-Amino-5-ethyl[1,2,4]triazolo[1,5-α]pyrimidin-6-yl)butanoic acid.
CAS name:	Not reported.
CAS No:	Not reported.
Synonyms:	Reg. No. 5178872.
	
Metabolite 02:	
Common name:	M650F02.
IUPAC name:	3-(7-Amino-5-ethyl[1,2,4]triazolo[1,5-α]pyrimidin-6-yl)propanoic acid.
CAS name:	Not reported.
CAS No:	Not reported.
Synonyms:	Reg. No. 5178871.
	
Metabolite 03:	
Common name:	M650F03.
IUPAC name:	(7-Amino-5-ethyl[1,2,4]triazolo[1,5-α]pyrimidin-6-yl)acetic acid.
CAS name:	Not reported.

CAS No:	Not reported.
Synonyms:	Reg. No. 5178870.
	
Metabolite 04:	
Common name:	M650F04.
IUPAC name:	7-Amino-5-ethyl[1,2,4]triazolo[1,5-α]pyrimidine-6-carboxylic acid.
CAS name:	Not reported.
CAS No:	Not reported.
Synonyms:	Reg. No. 5211623.
	

II. Information about the Laboratory

A.	**Name**	PTRL Europe GmbH (p. 3 in MRIDs 47700037, 47700038).
B.	**Address**	Helmholtzstr. 22, Science Park, D-89081 Ulm, Germany.
C.	**Telephone No.**	(++49)-(0)731-400693-14 (Study Director).
D.	**Name of the Study Director**	Thomas Class.
E.	**Name of the Lead Chemist**	Iris-Constanze Beck.
F.	**Laboratory Validation:**	Yes, at LOQ and 10xLOQ.

III. Method Summary Information for Analyte(s): **BAS 650F and its metabolites M650F01, M650F02, M650F03 and M650F04.**

A.	**Statement of Data Confidentiality**	Yes.
1.	**Is the Method Classified or Confidential?**	No.
2.	**Submitted Prior to 2008 with a Non-Standard Claim of Confidentiality?**	No.
B.	**Sample Preparation**	Parent BAS 650F: none reported. BAS 650F products: water adjusted to *ca.* pH 7 with HCl or NH_3, as needed (p. 51 in MRID 47700038).
C.	**Sample Extraction**	Parent BAS 650F: water aliquot (100 g) weighed into glass bottle; add NaCl (10 g); following NaCl dissolution, add methylene chloride (25 mL); shake mechanically (horizontal shaker, *ca.* 225 rpm) for 30 minutes (pp. 27, 29 in MRID 47700037). BAS 650F products: water (*ca.* pH 7) aliquot (10 g) loaded (flow rate *ca.* 1-2 mL/minute) onto Strata-X-C solid phase extraction (SPE) cartridge preconditioned with acetonitrile (6 mL) followed by water (6 mL); dry loaded cartridge under vacuum; elute (*ca.* 1-2 mL/minute) analytes with acetonitrile:25% NH_3 (90:10, v:v, 10 mL; p. 51 in MRID 47700038).
D.	**Sample Cleanup**	Parent BAS 650F: organic phase aliquot (10 mL) concentrated to dryness via rotary evaporation (*ca.* 40°C); residues reconstituted in acetonitrile:water (50:50, v:v; pp. 28-29 in MRID 47700037). BAS 650F products: eluate concentrated via rotary evaporation (*ca.* 50°C), then taken to dryness under nitrogen; residues reconstituted in acetonitrile (0.2 mL), and water (1.8 mL; p. 51 in MRID 47700038).
E.	**Sample Derivatization (if applicable)**	None reported.
F.	**Sample Analysis**	LC/MS/MS (p. 9 in MRID 47700037; p. 10 in MRID 47700038).

1.	**Instrumentation**	Agilent 1100 (BAS 650F) or 1200 (products) HPLC system and Applied Biosystems Sciex API 3000 (BAS 650F) or 4000 (products) triple quadrupole LC/MS/MS system with Turbolonspray (ESI, positive) source (p. 9 in MRID 47700037; p. 52 in MRID 47700038).
2.	**Primary Column**	Waters XTerra C18 column (4.6 x 50 mm, 3.5 µm; p. 9 in MRID 47700037; p. 52 in MRID 47700038). For parent BAS 650F analysis, LC column preceded by a Phenomenex C18 RP (3 x 4 mm) guard column (p. 9 in MRID 47700037).
3.	**Confirmatory Column (If Any)**	None reported.
4.	**Detector**	MRM (multiple reaction monitoring).
5.	**Other Confirmatory Techniques (If Any)**	Per analyte, two MRM parent-daughter ions for quantitation (Q) and confirmation (C) were monitored.
6.	**Other Relevant Information**	LC conditions: gradient mobile phase combining (A) 0.1% aqueous acetic acid and (B) 0.1% acetic acid in acetonitrile [percent A:B at 0.0 min. 90:10 (v:v), 7.0-9.0 min. 0:100, 9.1-11.0/12.0 min. 90:10], column temperature 30°C, injection volume 50 µL, flow rate 0.4/0.5 mL/minute (p. 9 in MRID 47700037; p. 52 in MRID 47700038). Retention times were ~8.3, ~3.6, ~2.5, ~2.0 and ~2.9 minutes for BAS 650F, M650F01, M650F02, M650F03 and M650F04, respectively (Figures 2-4, pp. 17-19 in MRID 47700037; Figures 5-16A, pp. 23-38 in MRID 47700038). MS conditions: ion transitions monitored (amu) were 276.3→149.1 (Q) and 176.2 (C) for BAS 650F; 250.2→232.0 (Q) and 149.1 (C) for M650F01; 236.0→176.1 (Q) and 218.2 (C) for M650F02; 222.2→176.1 (Q) and 204.1 (C) for M650F03; and 208.2→190.2 (Q) and 123.0 (C) for M650F04 (p. 9 in MRID 47700037; p. 52 in MRID 47700038).
G.	**Detection and Quantitation Limits**	
1.	**Limit of Quantitation (LOQ)**	

	Claimed in Method	0.05 µg/kg (pp. 11, 31 in MRI7D 47700037; pp. 13, 53 in MRID 47700038).	**Estimated**	0.05 µg/kg (Figures 3-4, pp. 18-19 in MRID 47700037; Figures 9-16A, pp. 27-38 in MRID 47700038).
2.	**Limit of Detection (LOD)**			
	Claimed in Method	Parent BAS 650F: 0.0025 µg/kg (BASF), 0.005 µg/kg (PTRL; pp. 12, 31 in MRID 47700037). BAS 650F products: 0.01 µg/kg (pp. 14, 53 in MRID 47700038).	**Estimated**	Parent BAS 650F: <0.005 µg/kg (Figures 3-4, pp. 18-19 in MRID 47700037). BAS 650F products: <0.01 µg/kg (Figures 9-16A, pp. 27-38 in MRID 47700038).

H. **Recovery (Accuracy) /Precision Data; percent recovery (n = 5, except where noted otherwise)**

Level	Cmpd	BAS 650F		M650F01		M650F02		M650F03		M650F04	
µg/kg	Ion m/z	149 Q	176 C	232 Q	149 C	176 Q	218 C	176 Q	204 C	190 Q	123 C
Drinking (Tap) Water											
0.05	**Range**	95-108	97-105	77-95	73-85	86-94	79-97	96-100	99-118[1]	81-97	81-106
	Mean	102	101	83	80	88	90	99	106	88	90
	SD	5	3	7	5	3	7	2	9	6	9
	RSD	5	3	9	6	4	8	2	8	7	10
0.50	**Range**	100-110	99-111	79-87	80-89	89-98	90-96	93-108	91-106	89-94	86-93
	Mean	107	105	84	85	94	94	101	100	91	90
	SD	4	4	3	3	3	3	6	6	2	2

	RSD	4	4	4	4	4	3	6	6	2	3
Surface (River) Water											
0.05	**Range**	97-102	98-103	75-86	85-94	92-101	90-97	92-103	94-103	98-100	87-100
	Mean	100	102	81	89	95	92	96	100	99	95
	SD	2	2	5	4	4	3	5	4	1	5
	RSD	2	2	6	4	4	3	5	4	1	6
0.50	**Range**	103-107	105-109	82-85	84-86	87-92	84-92	105-115	106-114	91-96	94-96
	Mean	106	107	84	85	90	89	110	110	94	95
	SD	2	2	1	1	2	3	4	3	2	1
	RSD	2	2	2	1	3	4	3	3	2	1

Q = Quantitation ion; C = Confirmation ion; SD = Standard Deviation; RSD = Relative Standard Deviation.
BAS 650F results obtained from Tables 1-2, pp. 14-15 of MRID 47700037. Product results obtained from Tables 1-2, pp. 17-18 of MRID 47700038.
1 n = 4 (Table 1, p. 17 of MRID 47700038).

IV. Detailed Information about the Method

		YES	NO	REVIEW FURTHER
A.	**Does the method require spiking with the analytes(s) of interest?**	x		
B.	**If the method requires explosive or carcinogenic reagents, are proper precautions explained?**			Not applicable.
C.	**Is the following information supplied?**			
1.	**Detailed stepwise description of:**			
a.	**The sample preparation procedure?**	x		
b.	**The sample spiking procedure?**	x		
c.	**The extraction procedure?**	x		
d.	**The derivatization procedure?**			Not applicable.

		YES	NO	REVIEW FURTHER
e.	**The clean-up procedure?**	x		
f.	**The analysis procedure?**	x		
2.	**Procedures for:**			
a.	**Preparation of standards?**	x		
b.	**Calibration of instrument?**	x		
3.	**List of glassware and chemicals**	x		
a.	**Are sources recommended?**	x		
b.	**Are they commercially available?**	x		
4.	**Name, model, *etc.*, of the instrument, column, detector, *etc.*, used?**	x		
a.	**Are sources recommended?**	x		
b.	**Are they commercially available?**	x		
5.	**LOD**			
a.	**Is there an explanation of how it was calculated?**	x		Instrument response at lowest standard concentration achieved signal/noise ratio of equal or better than 3/1 (p. 31 of MRID 47700037; p. 53 of MRID 47700038).
b.	**Is it a scientifically accepted procedure?**	x		
c.	**Is the matrix blank free of interferences(s) at the retention time, wavelength, *etc.*, of the analyte(s) of interest?**	x		Figures 3-4, pp. 18-19 of MRID 47700037. Figures 9-16A, pp. 27-38 of MRID 47700038.

		YES	NO	REVIEW FURTHER
6.	**LOQ**			
a.	**Is there an explanation of how it was calculated?**		x	
b.	**Is it a scientifically accepted procedure?**			Not applicable.
7.	**Precision and accuracy data**			
a.	**Were there an adequate number of spiked samples analyzed?**	x		Five replicates each at LOQ and 10xLOQ.
b.	**Are the mean recoveries between 70-120%?**	x		
c.	**Are the RSDs of the replicates 20% or less at or above the LOQ?**	x		
8.	**Description and/or explanation of:**			
a.	**Areas where problems may be encountered?**		x	
b.	**Steps that are critical?**		x	
c.	**Interferences that may be encountered?**		x	
9.	**Characterization of the Matrix(ces)?**	x		pp. 8-9 of MRID 47700037; p. 11 of MRID 47700038.

V. Representative Chromatograms

		YES	NO	REVIEW FURTHER
A.	**Are there representative chromatograms for:**			

1.	**Analyte(s) in each matrix at the LOQ and 10 x LOQ?**	x		Figures 3-4, pp. 18-19 of MRID 47700037; Figures 9-16A, pp. 27-38 of MRID 47700038.
2.	**Method blanks?**		x	
3.	**Matrix blanks?**	x		Figures 3-4, pp. 18-19 of MRID 47700037; Figures 9-16A, pp. 27-38 of MRID 47700038.
4.	**Standard curves?**	x		Figure 1, p. 16 of MRID 47700037; Figures 1-4, pp. 19-22 of MRID 47700038. See section ***IX. Recommend-ations*** below.
a.	**Do the standard curves have acceptable linearity?**	x; r = 0.9992-0.9999).		
5.	**Standards that can be used to recalculate some of the values for analyte(s) in the sample chromatograms?**	x		See section ***IX. Recommend-ations*** below.
B.	**Can the responses of the analytes(s) in the chromatograms of the lowest spiking level be accurately measured?**	x		

VI. Good Laboratory Practice (GLP) Standards

		YES	NO	REVIEW FURTHER
A.	**Is there a statement of adherence to the FIFRA GLP standards?**	x		See section ***IX. Recommend-ations*** below.

VII. Independent Lab Validation (ILV)

		YES	NO	REVIEW FURTHER
A.	**Was an ILV performed?**	x		
B.	**Was the validation independent?**	x		
C.	**Did the ILV's precision/accuracy data meet the criteria established in OPPTS Guideline 850.7100?**	x		Tables 1-5, pp. 23-32 of MRID 48435701.
D.	**Were recommendations of major or minor modifications to the method made by the independent lab performing the ILV? If major modifications were suggested, what were they?**		x	See section ***IX. Recommend-ations*** below.

VIII. Completeness

		YES	NO	REVIEW FURTHER
A.	**Has enough information been supplied to do a proper review?**	x		
B.	**Has enough information been supplied to do a laboratory evaluation, if requested?** [*This may be left blank, as it is a determination made by BEAD ECB.*]			