TEXT SEARCHABLE DOCUMENT 2011

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

Data Requirement: EPA Guideline: 835.6100
OECD Data Point: IIA 4.4

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:
Date:

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

ANALYTICAL METHOD: EPA MRID No. 47700035. Class, T., and I-C. Beck. 2008. Validation of analytical method L0091 for the determination of residues of BAS 650 F and its metabolites M650F01, M650F02, M650F03 and M650F04 in soil 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 Unpublished study performed by PTRL Europe GmbH, Ulm, Germany; sponsored by BASF SE, Limburgerhof, Germany; and submitted by BASF Corporation, Research Triangle Park,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; 49 pages (pp. 1-3). PTRL Europe Report and Study No.: P 1481 G (pp. 1, 3). BASF Report No.: 250804 and Registration Document No.:

2008/1017003 (p. 1). Experimental start date April 28, 2008, and completion date June 25, 2008 (p. 3). Final report issued July 16, 2008 (p. 1).

INDEPENDENT LABORATORY VALIDATION: EPA MRID No. 47700025. Zhang, X. 2009. Independent laboratory validation of BASF analytical method L0091: "The determination of residues of BAS650F and its metabolites, M650F01, M650F02, M650F03, and M650F04 in soil samples". Unpublished study performed by Alliance Pharma Inc., Malvern, Pennsylvania; sponsored and submitted by BASF Corporation, Research Triangle Park, North Carolina; 81 pages (pp. 1-2). Alliance Pharma Study No.: 090802. BASF Study No.: 375546 and Registration Document No.: 2009/7006098. Experimental start date September 22, 2009, and completion date September 23, 2009 (p. 7). Final report issued October 26, 2009 (p. 1).

EXECUTIVE SUMMARY

This method is designed for the quantitative determination of residues of BAS 650F and its products M650F01, M650F02, M650F03 and M650F04 in soil. The method was created by BASF Corporation and validated by PTRL Europe GmbH. An independent laboratory validation (ILV), performed by Alliance Pharma Incorporated was submitted with this method. The original method was not provided with either the validation (ECM) or ILV reports.

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

Method Summary: Analytes are extracted from soil by shaking with acetonitrile followed by acetonitrile:water (50:50, v:v). Extracts are filtered, combined and an aliquot diluted with 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 0.01 mg/kg and 0.002 mg/kg, respectively. The ECM report LOD was 0.001 mg/kg.

METHOD ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS

No method deficiencies were apparent; however, the original BASF method was not provided for comparison.

For the ECM report, recovery results for parent BAS 650F could not be verified from the information provided. Additionally, recovery results determined by the primary reviewer for product M650F02 differed from reported results by 21%.

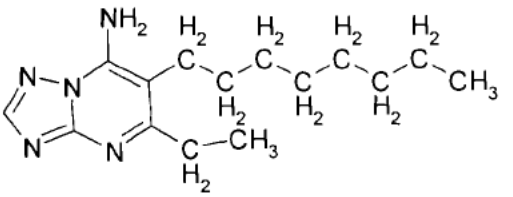
COMPLIANCE

This method was 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 (pp. 1-3 of MRID 47700035). Signed and dated statements of No Data Confidentiality, GLP and Quality Assurance were provided.

A. BACKGROUND INFORMATION

No information regarding mode of action of BAS 650F or purpose of the end-use product were provided.

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.
CAS #	865318-97-4.
Structure	

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

B. MATERIALS AND METHODS

B.1. Principle of Method

Analytes are extracted from soil by mechanical shaking with acetonitrile followed by acetonitrile:water (50:50, v:v). Extracts are filtered, combined and an aliquot diluted with acetonitrile:water (10:90, v:v). 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 ID	BASF method L0091, version 01, TP (technical procedure) 02 (p. 4 of MRID 47700035).
Analyte(s)	BAS 650F, M650F01, M650F02, M650F03, M650F04.
Extraction solvent/technique	Soil (5 g dry wt.) extracted with acetonitrile (10 mL) via mechanical shaking for 30 minutes (p. 11 of MRID 4700035). Extract separated from soil by centrifugation and decanted. Extraction repeated twice more with acetonitrile:water (50:50, v:v, 20 mL per extraction).
Cleanup strategies	Extracts filtered (folded filter paper), combined and brought to volume with acetonitrile:water (10:90, v:v; pp. 9, 11 of MRID 4700035). Aliquot (0.1 mL) diluted with acetonitrile:water (10:90, 1 mL) for analysis.
Instrument/Detector	Agilent 1200 HPLC system, equipped with a Waters XTerra C18 column (4.6 x 50 mm, 3.5 µm) preceded by a Phenomenex C18 RP (3 x 4 mm) guard column, and Applied Biosystems MDS Sciex API 4000 triple quadrupole LC/MS/MS system with Turbolonspray (ESI) source and MRM.

C. RESULTS AND DISCUSSION

C.1. Recovery Results Summary

TABLE C.1. Recovery Results from Method Validation for the Determination of Residues in Two Soils

Analyte	Spiking Level (mg/kg)	Mean Recoveries Obtained (%)	Relative Standard Deviation
Sandy Loam[1]			
BAS 650F	0.01	79	3
	0.10	73	2
M650F01	0.01	89	4
	0.10	86	2
M650F02	0.01	80	3
	0.10	71	3
M650F03	0.01	109	4
	0.10	106	3
M650F04	0.01	93	4
	0.10	90	3
Clay[1]			
BAS 650F	0.01	77	5
	0.10	72	4
M650F01	0.01	85	2
	0.10	77	3
M650F02	0.01	88	4
	0.10	77	4
M650F03	0.01	88	5
	0.10	83	3
M650F04	0.01	79	4
	0.10	76	2
Soils Combined[2]			
BAS 650F	0.01	79	4
	0.10	73	3
M650F01	0.01	86	6
	0.10	82	7
M650F02	0.01	84	6
	0.10	75	5
M650F03	0.01	100	9
	0.10	95	12
M650F04	0.01	87	8
	0.10	83	8

1 Quantitation ion results from Tables 1-2, pp. 17-18 of MRID 4700035.
2 Combined soil results (quantitation and confirmation ions) from DER Attachment 2.

C.1.1. Method Characteristics

TABLE C.2. Method Characteristics	
Parameter	**Value**
Analyte(s)	BAS 650F, M650F01, M650F02, M650F03, M650F04.
Limit of Quantitation (LOQ)	0.01 mg/kg.
Limit of Detection (LOD)	0.001 mg/kg.
Accuracy/Precision at LOQ	Acceptance criteria (EFED-ECM 2, Version 1, December 2010, p. 5) were met with matrix spike recoveries ranging from 74% to 113% (quantitation ion results) and relative standard deviations of ≤5% (Tables 1-2, pp. 17-18 of MRID 4700035).
Reliability of the Method/[ILV]	The method was successfully validated (ILV) in one trial.
Linearity	BAS 650F (quadratic regression): r = 1.0000. M650F01, M650F02, M650F03 and M650F04 (linear regression): r = 0.9998-1.0000.
Specificity	Method separately quantifies BAS 650F and its products M650F01, M650F02, M650F03 and M650F04.

C.2. Independent Laboratory Validation (ILV)

The ILV was conducted in compliance with FDA GLP 21 CFR part 58; no additional regulatory guidelines were cited (p. 3 of MRID 4700025).

TABLE C.3. Recovery Results of the Method Obtained by an Independent Laboratory Validation for the Determination of Residues in Soil[1,2]

Analyte	Spiking Level (units)	Mean Recoveries Obtained (%)	Relative Standard Deviation
BAS 650F	0.01	108.3	4.8
	0.10	117.9	3.2
M650F01	0.01	95.0	4.7
	0.10	101.6	5.3
M650F02	0.01	104.0	5.8
	0.10	102.9	2.7
M650F03	0.01	100.1	4.7
	0.10	110.4	3.7
M650F04	0.01	88.0	6.0
	0.10	96.9	3.6

1 Soil was described as both a sandy loam and a clay loam; a soil characterization was not provided (p. 10; Appendix A, pp. 80-81 of MRID 4700025).
2 Quantitation ion results from Appendix A, pp. 80-81 of MRID 4700025.

D. CONCLUSION

This method is designed for the quantitative determination of residues of BAS 650F and its products M650F01, M650F02, M650F03 and M650F04 in soil. 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 limits of detection for BAS 650F and its products are 2x higher than those 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 soil samples

APPENDIX A

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

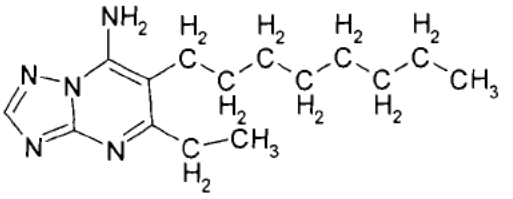
BAS 650F; EPA PC Code 119210 EPA MRID Numbers 47700035 (ECM)/47700025 (ILV)

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

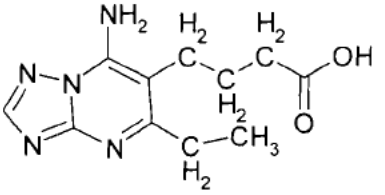
Referenced page numbers are from MRID 47700035, except where noted otherwise.

I. Background Information

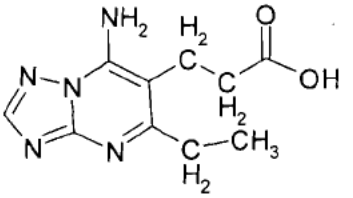
A.	**Title of Method**	BASF Analytical method L0091 "The determination of residues of BAS 650 F and its metabolites, M650F01, M650F02, M650F03 and M650F04 in soil samples" (BASF Document Number 2008/1017003); BASF method L0091, version 01, TP (technical procedure) 02 (pp. 4, 8 in MRID 4700035; pp. 7, 10, 12 in MRID 4700025).	
B.	**ECM No.**	[*Leave blank. This is for BEAD ECB's use.*]	
C.	**MRID No.**	47700035.	
D.	**Matrix**	Soil.	
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.



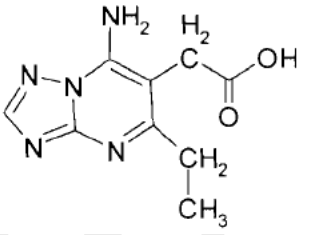
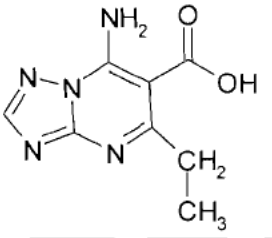
Metabolite 01:	
Common name:	M650F01
IUPAC name:	4-(7-Amino-5-ethyl[1,2,4]triazolo[1,5-α]pyrimidin-6-yl)butanoic acid.
CAS name:	Not reported.
CAS No:	Not reported.
Synonyms:	Reg. No. 5178872.



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).
B.	**Address**	Helmholtzstr. 22, Science Park I, 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**	Soil 2-mm sieved; soil moisture adjusted to *ca.* 40% of maximum water holding capacity (p. 9).
C.	**Sample Extraction**	Soil aliquot (5 g dry wt.) weighed into centrifuge bottle; add 10 mL acetonitrile; shake mechanically (horizontal shaker, *ca.* 225 rpm) for 30 minutes (p. 11). Extract separated from soil by centrifugation (4,000 rpm, 5 minutes) and decanted. Repeat extraction twice more with 20 mL acetonitrile:water (50:50, v:v) per extraction.
D.	**Sample Cleanup**	Extracts filtered (folded filter paper, 110 mm i.d., Machery-Nagel), combined and adjusted to 50 mL with acetonitrile:water (10:90, v:v; pp. 9, 11). Aliquot (0.1 mL) diluted to 1 mL with acetonitrile:water (10:90) for analysis.
E.	**Sample Derivatization (if applicable)**	None reported.
F.	**Sample Analysis**	LC/MS/MS (p. 11).
1.	**Instrumentation**	Agilent 1200 HPLC system and Applied Biosystems MDS Sciex API 4000 triple quadrupole LC/MS/MS system with Turbolonspray (ESI) source (pp. 11-12).
2.	**Primary Column**	Waters XTerra C18 column (4.6 x 50 mm, 3.5 µm) preceded by a Phenomenex C18 RP (3 x 4 mm) guard column (p. 11).
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 min. 90:10], column temperature 30°C, injection volume 50 µL, flow rate 0.5 mL/minute (p. 11). Retention times were ~8.4, ~3.8-3.9, ~3.0-3.1, ~2.3-2.4 and ~2.9-3.0 minutes for BAS 650F, M650F01, M650F02, M650F03 and M650F04, respectively (Figures 4-18, pp. 23-37). 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. 12).										
G.	**Detection and Quantitation Limits**											
1.	**Limit of Quantitation (LOQ)**											
	Claimed in Method	0.01 mg/kg (p. 8).			**Estimated**	0.01 mg/kg (Figures 9-18, pp. 28-37).						
2.	**Limit of Detection (LOD)**											
	Claimed in Method	0.001 mg/kg.			**Estimated**	<0.001 mg/kg.						

H. Recovery (Accuracy) /Precision Data; percent recovery (mean ± SD, n = 5)

Level	Cmpd	BAS 650F		M650F01		M650F02		M650F03		M650F04	
mg/kg	Ion m/z	149 Q	176 C	232 Q	149 C	176 Q	218 C	176 Q	204 C	190 Q	123 C
Sandy loam (LUFA 2.3)											
0.01	Range	75-80	76-83	87-93	86-93	77-83	78-87	101-113	98-112	87-97	84-96
	Mean	79	80	89	91	80	81	109	104	93	90
	SD	2	3	3	3	2	4	5	5	4	5
	RSD	3	4	4	3	3	5	4	5	4	5

0.10	**Range**	71-75	72-76	83-88	84-90	68-73	70-74	102-111	99-108	87-93	86-93
	Mean	73	74	86	88	71	73	106	104	90	89
	SD	2	2	2	2	2	2	4	4	2	2
	RSD	2	3	2	3	3	2	3	4	3	3

		Clay (LUFA 6S)									
0.01	**Range**	74-83	75-84	83-87	77-83	85-93	83-93	85-94	92-101	75-83	80-92
	Mean	77	80	85	79	88	88	88	97	79	86
	SD	4	3	2	2	3	4	5	4	3	5
	RSD	5	4	2	3	4	4	5	4	4	6
0.10	**Range**	69-77	69-76	74-80	74-81	74-82	74-84	81-87	82-86	74-79	76-82
	Mean	72	72	77	78	77	79	83	84	76	78
	SD	3	3	3	3	3	4	2	2	2	2
	RSD	4	4	3	4	4	4	3	2	2	3
Q = Quantitation ion; C = Confirmation ion; SD = Standard Deviation; RSD = Relative Standard Deviation. Results obtained from Tables 1-2, pp. 17-18 of the study report.											

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.
e.	**The clean-up procedure?**	x		
f.	**The analysis procedure?**	x		
2.	**Procedures for:**			
a.	**Preparation of standards?**	x		

		YES	NO	REVIEW FURTHER
b.	**Calibration of instrument?**	x		
3.	**List of glassware and chemicals**	Chemicals	Glassware	Glassware only described as "typical" (p. 9).
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	
b.	**Is it a scientifically accepted procedure?**			Not applicable.
c.	**Is the matrix blank free of interferences(s) at the retention time, wavelength, *etc.*, of the analyte(s) of interest?**	x		Figures 9-18, pp. 28-37.
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		

		YES	NO	REVIEW FURTHER
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 (Appendix 2, pp. 43-46)		

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 9-18, pp. 28-37).		See section ***IX. Recommend-ations*** below.
2.	**Method blanks?**		x	
3.	**Matrix blanks?**	x (Figures 9-18, pp. 28-37).		
4.	**Standard curves?**	x (Figures 1-3, pp. 20-22).		See section ***IX. Recommend-ations*** below.
a.	**Do the standard curves have acceptable linearity?**	x; r = 0.9998-1.0000).		
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?**	Q ion for BAS 650F. Q and C ions for the four products.	C ion for BAS 650F.	For BAS 650F C ion, mean ± RSD of 122.8 ± 4.8%.
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	See section ***IX. Recommend-ations*** below.
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.*]			
C.	**Are all steps in the method scientifically sound?**			
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**			
2.	**Major Deficiencies**	x		
3.	**Minor Deficiencies**	x		

IX. Recommendations

- Section ***V. Representative Chromatograms*** **A. 1.** In some instances, M650F03 gave a slightly split peak, but this reportedly did not hinder quantification (p. 15; Figure 12, p. 31; Figure 17, p. 36).

- Section ***V. Representative Chromatograms*** **A. 4.** Quadratic regression analysis was used to determine calibration curves for BAS 650F, while linear regression analysis was used for the products M650F01, M650F02, M650F03 and M650F04.

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

- Section ***V. Representative Chromatograms*** **A. 5.** Recovery results for BAS 650F could not be verified by the primary reviewer. Using quadratic regression analysis is atypical and no explanation was provided as to why linear regression analysis was not employed for determining parent BAS 650F calibration curves. Using linear regression analysis (R^2 = 1.0000) for the calibration curves and the provided chromatogram results, percent BAS 650F recoveries for sample P1481-77 were 76% and 78% for ions 149 m/z and 176 m/z, respectively, as compared to 74% and 76%, respectively, in the ECM report (Table 1, p. 17; Figure 9, p. 28; DER Attachment 2).

  Recovery results for the BAS 650F products (M650F01, M650F02, M650F03 and M650F04) in 0.10 mg/kg fortified clay soil, for provided chromatograms, were verified by the primary reviewer (DER Attachment 2). However, recovery results for the products in 0.10 mg/kg fortified sandy loam soil, except for M650F03 at ion 204 m/z, could not be verified. Recovery results determined by the primary reviewer differed by 1-4% from ECM reported values for M650F01, M650F03 (ion 176) and M650F04, but differed by 21% for M650F02.

  ILV recovery results for BAS 650F and its products in 0.01and 0.10 mg/kg fortified soil, for provided chromatograms, were verified by the primary reviewer (DER Attachment 2).

- Section ***VI. Good Laboratory Practice (GLP) Standards*** **A.** The study was 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 (pp. 1-2).

- Section ***VII. Independent Lab Validation (ILV)*** **D.** The ILV limits of detection for BAS 650F and its products are 2x higher than those stated in the ECM report.

- Section ***VIII. Completeness*** **A.** Although PTRL validated the method for BASF and the study was reportedly "conducted as described in method L0091 version 01, technical procedure TP02", the original method was not provided for review for comparison. The actual title of the method was obtained from the ILV report.

- Section ***VIII. Completeness*** **E. 2. Major Deficiencies.** Recovery results for parent BAS 650F could not be verified from the information provided. Additionally, recovery results determined by the primary reviewer for product M650F02 differed from ECM reported results by 21%.

- Section ***VIII. Completeness*** **E. 3. Minor Deficiencies.** The original BASF method should be appended to 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