

Analytical method for trifludimoxazin (BAS 850 H) and its metabolites M850H001, M850H002, M850H003, and M850H004 in soil

Reports: ECM: EPA MRID No.: 50406037. Saha, M. 2018. Methods of Analysis of BAS 850 H and its Relevant Metabolites in Soil with Limit of Determination (LOD) Calculation (Method D1401/02). BASF Study Nos.: 411638 and 411638_01 (Appendix A, p. 8; Appendix B, p. 221). BASF Registration Document No.: 2018/7005593. Report prepared, sponsored and submitted by BASF Corporation, Research Triangle Park, North Carolina, North Carolina; 276 pages. Final report issued August 13, 2018.

ILV: EPA MRID No. 50406038. Sheng, L. 2017. Independent Laboratory Validation of BASF Analytical Method D1401/02: "Analytical Method for the Determination of Residues of BAS 850 H and its four metabolites, M850H001, M850H002, M850H003 and M850H004 in Soil by LC-MS/MS". BASF Study ID No.: 412300. BASF Registration Document No.: 2017/7008180. CRO Study No.: 137G1602. Report prepared by EPL Bio Analytical Services (EPL), Niantic, Illinois, sponsored and submitted by BASF Crop Protection, Research Triangle Park, North Carolina; 197 pages. Final report issued November 1, 2017.

Tier II Summary and Evaluation: EPA MRID No. 50406044. Kleppe, C. 2018. Multi-Lateral Submission for the Evaluation of the Active Substance BAS 850 H. Chapters 4.4. BASF Registration Document No.: 2018/7000014 US. Report submitted by BASF Corporation, Research Triangle Park, North Carolina; 24 pages. Final report issued March 1, 2018.

Document No.: MRIDs 50406037 & 50406038 & 50406044

Guideline: 850.6100

Statements: ECM: The study was not conducted in accordance with Good Laboratory Practice (GLP) standards since it was not a study (p. 3 of MRID 50406037). Signed and dated No Data Confidentiality and GLP statements were provided; Quality Assurance and Authenticity statements were not provided (pp. 2-3). The ECM was a compilation of the study reports for an analytical method and LOD determination (p. 5). These two study reports (BASF Study # 411638 and 411638_1) were conducted in accordance with USEPA FIFRA GLP standards, 40 CFR, Part 160 (Appendix A, p. 10; Appendix B, p. 260). Signed and dated No Data Confidentiality, GLP, Quality Assurance, and Authenticity statements were provided (Appendix A, pp. 9-12; Appendix B, pp. 222-225).

ILV: The study was conducted in accordance with USEPA FIFRA GLP standards, 40 CFR, Part 160 (p. 3 of MRID 50406038). Signed and dated No Data Confidentiality, GLP, Quality Assurance, and Authenticity statements were provided (pp. 2-5).

Tier II Summary and Evaluation: Signed and dated No Data Confidentiality statement was provided (p. 2 of MRID 50406044). No GLP, Quality Assurance, and Authenticity statements were provided.

Classification: This analytical method is classified as **supplemental**. The LOQ of 1.0 µg/kg is greater than the lowest toxicological level of concern (0.0219 µg/kg, MRID 50406453) in soil for trifludimoxazin and its four metabolites.

PC Code: 080800

EFED Final Reviewer: William Gardner, Ph.D.,
Environmental Scientist Signature:
Date: 11/2/2020

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This Data Evaluation Record is a modification of the Tier II DER submitted to the Environmental Fate and Effects Division (EFED) by the registrant. Statistical analyses have been performed according to EFED guidance, and the Executive Summary has been revised. The DER may have been altered by EFED personnel subsequent to signing by CDM/CSS-Dynamac JV personnel. The CDM/CSS-Dynamac Joint Venture role does not include establishing Agency policies.

Executive Summary

This analytical method, BASF Method D1401/02, is designed for the quantitative determination of trifludimoxazin (BAS 850 H), M850H001, M850H002, M850H003, and M850H004 in soil at the LOQ of 1.0 µg/kg using LC/MS/MS. The LOQ is greater than the lowest toxicological level of concern (0.0219 µg/kg, MRID 50406453) in soil for trifludimoxazin and its four metabolites. The ECM performed the method with two characterized soil matrices while the ILV performed the method with only one characterized soil matrix. All soil matrices were sourced from trifludimoxazin terrestrial field dissipation studies. The ILV validated BASF Method D1401/02 in one soil for all analytes during the first trial with modifications to the analytical instrument and parameters. The recommended changes were included in the ECM final report. All ILV and ECM data regarding repeatability, accuracy, precision, and specificity were satisfactory for all five analytes. ILV linearity was satisfactory for all analytes; ECM linearity was satisfactory for all analyses, except M850H002 and M850H004 in clay soil.

In a separate study, the method LOD of BASF Analytical Method No. D1401/02 was validated in accordance with the methodology set forth in 40 CFR Ch. 1 Part 136 Appendix B. The LOD evaluation for BASF analytical method D1401/02 was performed for both positive and negative modes. An MDL calculation and subsequent LOD evaluation were conducted for each mode

based on the least sensitive analyte. Based on the calculated MDLs for BAS 850 H and M850H004, the LOD was calculated as 0.0002 mg/kg for both the positive and negative modes.

Some data obtained directly from Tier II Summary and Evaluation: EPA MRID No. 50406044 was not cited.

All referenced page numbers for MRID 50406037 refer to those reported on the right-handed margin of the document pages.

Table 1. Analytical Method Summary – BASF Analytical Method D1401/02

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date (dd/mm/yyyy)	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Trifludimoxazin (BAS 850 H; Reg. No. 5654329)	50406037 ¹	50406038		Soil ^{2,3}	14/02/2018 ⁴	BASF Corporation	LC/MS/MS	0.001 mg/kg
M850H001 (Reg.No. 5749359)								
M850H002 (Reg.No. 5757725)								
M850H003 (Reg.No. 5757726)								
M850H004 (Reg.No. 5833884)								

1 A compilation of BASF Study #s 411638 and 411638_01 (Appendix A, pp. 8, 16 and Appendix B, p. 221 of MRID 50406037).

2 In the ECM, clay (0-3 inch soil depth; TRT SC-1 0-3"; 31% sand, 29% silt, 40% clay; pH 7.6 in saturated paste; 2.1% organic carbon – Walkley Black; 3.7% organic matter – Walkley Black) and loamy sand (0-3 inch soil depth; SC-10 0-3"; 86% sand, 12% silt, 2% clay; pH 5.4 in saturated paste; pH 4.9 in 0.01M CaCl₂, 1:1; 0.48% organic carbon – Walkley Black; 0.83% organic matter – Walkley Black) soil samples collected under trifludimoxazin terrestrial field dissipation studies (BASF Study 411690, Trial R130128, BASF Reg. Doc. No. 2016/7005812 and BASF Study 698742, Trial R140774, BASF Reg. Doc. No. 2016/7005814) were used in the study (USDA soil texture classification; p. 7 of MRID 50406044; Appendix A, Appendix A, pp. 55-56 of MRID 50406037). Test systems were characterized by Agvise Laboratories, Northwood, North Dakota. The soil textures were verified by the reviewer using USDA-NRCS technical support tools.

3 In the ILV, clay (30-36 inch soil depth; TRT SC-7 30-36"; 29% sand, 19% silt, 52% clay; pH 8.0 in saturated paste; 0.52% organic carbon – Walkley Black; 0.90% organic matter – Walkley Black) soil collected under a trifludimoxazin terrestrial field dissipation study (BASF Study 411690, Trial R130128, BASF Reg. Doc. No. 2016/7005812) was characterized, provided by BASF, and used in the study (p. 20 of MRID 50406044; p. 20; Appendix E, p. 185 of MRID 50406038). The soil texture was verified by the reviewer using USDA-NRCS technical support tools.

4 Method dates were November 28, 2017 (Original) and July 30, 2018 (Amended) for BASF Study # 411638 and November 16, 2017 for BASF Study # 411638_01 (Appendix A, p. 8, and Appendix B, p. 221 of MRID 50406037).

I. Principle of the Method

Method D1401/02

Residues of trifludimoxazin in soil samples (5 g each) are extracted by shaking twice with methanol:water with 0.1% formic acid (70:30, v/v). For the analysis of parent and M850H001, residues in an aliquot of the combined extracts are concentrated to an aqueous remainder and partitioned with cyclohexane:ethyl acetate (90:10, v/v); residues in an aliquot of the organic phase are then evaporated to dryness, reconstituted in methanol:water with 0.1% formic acid (20:80, v/v) and analyzed. For the analysis of M850H002, M850H003 and M850H004, residues in a separate aliquot of the combined extracts are concentrated to an aqueous remainder and diluted to a final volume of methanol:water with 0.1% formic acid (20:80, v/v) and analyzed.

After sample extraction and clean-up, residues of trifludimoxazin are determined by liquid chromatography (LC) electrospray ionization tandem mass spectrometry (MS/MS-ESI), monitoring in the positive ion mode the transitions 413→74 and 413→134 for trifludimoxazin and 397→114 and 397→141 for M850H001, and monitoring in the negative ion mode the transitions 373→193 and 373→323 for M850H002, 357→307 and 357→137 for M850H003 and 385→103 and 385→255 for M850H004. The results are calculated by direct comparison of the sample peak responses to those of external standards.

For validation, untreated soil samples were fortified with each analyte and analyzed according to the established method validation guidelines. The analytical sets consisted of a reagent blank, two controls, five replicates fortified with analyte at the method limit of quantitation, 0.001 mg/kg, and five replicates fortified at a higher level, corresponding to 10X the limit of quantitation, 0.01 mg/kg. For each analyte, the two mass transitions described above were evaluated. In conjunction with the subject study, matrix- and solvent-matched standards were analyzed in a separate experiment to evaluate any potential matrix effects.

Summary parameters for the analytical method are listed in the table shown below (**Table 2**).

Table 2: Summary Parameters for the Analytical Method Used for the Quantitation of Residues of Trifludimoxazin in Soil

Method ID	BASF method D1401/02
Analyte(s)	Residues of trifludimoxazin, including the metabolites M850H001, M850H002, M850H003 and M850H004, in soil
Extraction Solvent/technique	Residues of trifludimoxazin in soil samples (5 g each) are extracted by shaking twice with methanol:water with 0.1% formic acid (70:30, v/v). For the analysis of parent and M850H001, residues in an aliquot from the combined extracts are concentrated to an aqueous remainder and partitioned with cyclohexane:ethyl acetate (90:10, v/v); residues in an aliquot of the organic phase are then evaporated to dryness, reconstituted in methanol:water with 0.1% formic acid (20:80, v/v) and analyzed. For the analysis of M850H002, M850H003 and M850H004, residues in a separate aliquot from the combined extracts are concentrated to an aqueous remainder and diluted to a final volume of methanol:water with 0.1% formic acid (20:80, v/v) and analyzed
Cleanup Strategies	Centrifugation and liquid/liquid partitioning

Instrument	In the subject study, analyses for parent and M850H001 were performed with an Agilent 1290 UPLC system equipped with a Waters Xbridge C18 column (4.6 x 50 mm, 2.5 µm particle size) and a Sciex Instruments API 5500 detector and using a mobile phase gradient of (A) water with 1% formic acid and (B) acetonitrile with 0.1% formic acid, 70:30 to 50:50 to 1:99, v/v, over 8.0 minutes (flow rate 800 uL/minute). For the remaining analytes, analyses were performed with the same system described above using a mobile phase gradient of (A) water with 1% formic acid and (B) acetonitrile with 0.1% formic acid, 70:30 to 40:60 to 1:99, v/v, over 3.45 minutes (flow rate 800 uL/minute).	
Detector	AB Sciex 5500 Mass Spectrometer	
Analyte	Quantitation (<i>m/z</i>)	Confirmation (<i>m/z</i>)
BAS 850 H	413→74	413→134
M850H001	397→114	397→141
M850H002	373→193	373→323
M850H003	357→307	357→137
M850H004	385→103	385→255
Ionization Mode	Positive mode for trifludimoxazin and M850H001; Negative mode for M850H002, M850H003 & M850H004	
Standardization Method	Linear regression (1/x weighting). Direct comparison of the sample peak responses to those of external standards.	
Stability of Std Solutions	The available standard stability data indicate that each analyte is stable in stock and mixed intermediate (fortification) standards prepared in acidified methanol for at least ~3 months (≥81 days) and in mixed calibration standard solutions prepared by serial dilution of the intermediate standards using acidified methanol:water (20:80, v/v) for at least ~2 months (≥61 days), each when held under refrigeration.	
Retention times (approx. minutes)	Parent trifludimoxazin, ~7.4 and M850H001, ~4.7 (1 st injection) M850H002, ~3.2; M850H003, ~2.5; and M850H004, ~3.3 (2 nd injection)	

Table obtained from Table 1, pp. 8-9 of MRID 50406044.

Instrument/Detector for Confirmatory Method: Accurate quantitation is possible using one chromatographic method and two different mass transitions; therefore, no additional confirmatory techniques are required. The MRM transitions used to identify trifludimoxazin and its metabolites were determined by product ion scan, which were included in the report.

A Method Flow Chart was provided (Appendix A, p. 86 of MRID 50406037).

ILV

The ILV performed the ECM method as written with modifications of the analytical method; the ILV recommended these method modifications (p. 29 of MRID 50406038). The ILV parameters are reported in **Table 3** below.

Table 3: Summary Parameters for the Analytical Method D1401/02 Used in the ILV for the Quantitation of Residues of Trifludimoxazin in Soil

Method ID	BASF method D1401/02
Analyte(s)	Residues of trifludimoxazin, including the metabolites M850H001, M850H002, M850H003 and M850H004, in soil
Extraction Solvent/technique	Residues of trifludimoxazin in soil samples (5 g each) are extracted by

	shaking twice with methanol:water with 0.1% formic acid (70:30, v/v). For the analysis of parent and M850H001, residues in an aliquot from the combined extracts are concentrated to an aqueous remainder and partitioned with cyclohexane:ethyl acetate (90:10, v/v); residues in an aliquot of the organic phase are then evaporated to dryness, reconstituted in methanol:water with 0.1% formic acid (20:80, v/v) and analyzed. For the analysis of M850H002, M850H003 and M850H004, residues in a separate aliquot from the combined extracts are concentrated to an aqueous remainder and diluted to a final volume of methanol:water with 0.1% formic acid (20:80, v/v) and analyzed	
Cleanup Strategies	Centrifugation and liquid/liquid partitioning	
Instrument	In the subject study, analyses for parent and M850H001 were performed with an Agilent 1290 UPLC system equipped with a Waters Xbridge C18 column (4.6 x 50 mm, 2.5 µm particle size) and a Sciex 6500 Triple Quad detector and using a mobile phase gradient of (A) water with 1% formic acid and (B) acetonitrile with 0.1% formic acid, 70:30 to 50:50 to 1:99, v/v, over 8.0 minutes (flow rate 800 uL/minute). For the remaining analytes, analyses were performed with the same system described above using a mobile phase gradient of (A) water with 1% formic acid and (B) acetonitrile with 0.1% formic acid, 70:30 to 40:60 to 1:99, v/v, over 3.45 minutes (flow rate 800 uL/minute).	
Detector	Sciex 6500 Triple Quad Mass Spectrometer	
Analyte	Quantitation (m/z)	Confirmation (m/z)
BAS 850 H	413→74	413→134
M850H001	397→114	397→141
M850H002	373→193	373→323
M850H003	357→307	357→137
M850H004	385→103	385→255
Ionization Mode	Positive mode for trifludimoxazin and M850H001; Negative mode for M850H002, M850H003 & M850H004	
Standardization Method	Linear regression (1/x weighting). Direct comparison of the sample peak responses to those of external standards.	
Stability of Std Solutions	The available standard stability data indicate that each analyte is stable in stock and mixed intermediate (fortification) standards prepared in acidified methanol for at least ~3 months (≥81 days) and in mixed calibration standard solutions prepared by serial dilution of the intermediate standards using acidified methanol:water (20:80, v/v) for at least ~2 months (≥61 days), each when held under refrigeration.	
Retention times (approx. minutes)	Parent trifludimoxazin, 7.3 and M850H001, 4.7 (1 st injection) M850H002, 2.8; M850H003, 2.1; and M850H004, 2.9 (2 nd injection)	

Table obtained from Table 5, p. 21 of MRID 50406044.

Instrument/Detector for Confirmatory Method: Accurate quantitation is possible using one chromatographic method and two different mass transitions; therefore, no additional confirmatory techniques are required.

Methodology to Evaluate MDL and LOD

Evaluation of LOD of BASF Analytical Method No. D1401/02 required the experimental determination of MDL as defined by 40 CFR Ch.1 Part 136 Appendix B (Reference 2). A brief description of the methodology to determine MDL is as follows:

1. Injections of standards containing all analytes were injected using LC-MS/MS parameters from D1401/02. All transitions were monitored according to the method. The least sensitive transition of the least sensitive analyte for each method injection (there are two, as noted earlier) was determined qualitatively through visual inspection of factors such as peak height, relative background level, area count, etc. Once the appropriate analytes and transitions were selected, an estimation was made to what level a sample in matrix would produce a S/N of 2-10.
2. Using BASF Analytical Method D1401/02, seven control samples (5 g each) were extracted twice with methanol-water with 0.1% formic acid (70:30, v/v) and taken to the final volumes for both injections. An aliquot (0.99 mL) from the extract was diluted with a calibration standard (methanol-water with 0.1% formic acid (20:80, v/v, 0.01 mL) to an appropriate concentration level to make the post-extraction fortified control samples for LOD determination. The sample was then syringe filtered (0.45µm PTFE) directly into HPLC injection vials, passing the first approximately 0.2 - 0.3 mL to waste. These seven matrix spiked samples were injected twice on the LC-MS/MS with bracketing calibration standards for quantitation.
3. Using the standard curve to calculate the concentrations of the seven matrix-spiked samples, the results were put into the equation shown below:

$$MDL = S \times t_{(N-1, 1-\alpha=.99)}$$

MDL = Method detection limit

S = Standard deviation of the matrix-spiked sample set concentrations

$t_{(N-1, 1-\alpha=.99)}$ = Critical t value from a student t-test table at 99% confidence

The acceptance criteria for the MDL calculation were:

- a. The calculated MDL must be able to be seen on the instrument with S/N of ≥ 2 .
 - b. The concentration of the matrix-spiked samples must be no greater than 10X the calculated MDL.
4. A post-extraction fortified control sample at the MDL was injected on the LC-MS/MS (no standard curve required) to verify that the MDL can be seen with a S/N ≥ 2 .

Loamy sand soil (0-3 inch) soil collected under a trifludimoxazin terrestrial field dissipation study (BASF Study 698742, Trial R140774, BASF Reg. Doc. No. 2016/7005814) was used for the LOD determination (p. 9 of MRID 50406044).

LOQ/LOD

The LOQ was defined by the lowest fortification level successfully tested. The validated LOQ for residues of BAS 850 H and its metabolites in soil is 0.001 mg/kg for each analyte (p. 4 of MRID 50406044). The limit of determination is set to be 0.0002 mg/kg for each analyte in soil.

II. Recovery Findings

Method D1401/02

Tier II Summary and Evaluation (MRID 50406044)

ECM (MRID 50406037/Tier II): Mean recoveries and relative standard deviations (RSDs) were within guideline requirements (mean 70-120%; RSD \leq 20%) for analysis of trifludimoxazin (BAS 850 H; Reg.No. 5654329), M850H001 (Reg.No. 5749359), M850H002 (Reg.No. 5757725), M850H003 (Reg.No. 5757726), and M850H004 (Reg.No. 5833884) in two soil matrices at fortification levels of 0.001 mg/kg (LOQ) and 0.01 mg/kg (10 \times LOQ; Table 2, pp. 11-12 of MRID 50406044). Analytes were identified using two ion transitions; performance data (recovery results) from primary and confirmatory analyses were comparable. Recovery results were corrected for residues quantified in the controls; however, no residues were quantified (Appendix A, p. 85; Appendix A, Appendix C, pp. 91-110 of MRID 50406037). Solvent-based calibration standards were used. The clay (0-3 inch soil depth; TRT SC-1 0-3"; 31% sand, 29% silt, 40% clay; pH 7.6 in saturated paste; 2.1% organic carbon – Walkley Black; 3.7% organic matter – Walkley Black) and loamy sand (0-3 inch soil depth; SC-10 0-3"; 86% sand, 12% silt, 2% clay; pH 5.4 in saturated paste; pH 4.9 in 0.01M CaCl₂, 1:1; 0.48% organic carbon – Walkley Black; 0.83% organic matter – Walkley Black) soil samples collected under trifludimoxazin terrestrial field dissipation studies (BASF Study 411690, Trial R130128, BASF Reg. Doc. No. 2016/7005812 and BASF Study 698742, Trial R140774, BASF Reg. Doc. No. 2016/7005814) were used in the study (USDA soil texture classification; p. 7 of MRID 50406044; Appendix A, Appendix A, pp. 55-56 of MRID 50406037). Test systems were characterized by Agvise Laboratories, Northwood, North Dakota. The soil textures were verified by the reviewer using USDA-NRCS technical support tools.

ILV (MRID 50406038/Tier II): Mean recoveries and RSDs were within guideline requirements for analysis of trifludimoxazin (BAS 850 H; Reg.No. 5654329), M850H001 (Reg.No. 5749359), M850H002 (Reg.No. 5757725), M850H003 (Reg.No. 5757726), and M850H004 (Reg.No. 5833884) in one soil matrix at fortification levels of 0.001 mg/kg (LOQ) and 0.01 mg/kg (10 \times LOQ; Table 6, pp. 27-30 of MRID 50406044). Analytes were identified using two ion transitions; performance data (recovery results) from primary and confirmatory analyses were comparable. Solvent-based calibration standards were used. The clay (30-36 inch soil depth; TRT SC-7 30-36"; 29% sand, 19% silt, 52% clay; pH 8.0 in saturated paste; 0.52% organic carbon – Walkley Black; 0.90% organic matter – Walkley Black) soil collected under a trifludimoxazin terrestrial field dissipation study (BASF Study 411690, Trial R130128, BASF Reg. Doc. No. 2016/7005812) was characterized, provided by BASF, and used in the study (p. 20

of MRID 50406044; p. 20; Appendix E, p. 185 of MRID 50406038). The soil texture was verified by the reviewer using USDA-NRCS technical support tools. The ILV validated BASF Method D1401/02 in one soil for BAS 850 H, M850H002, M850H003, and M850H004 analytes during the first trial with modifications to the analytical instrument and parameters (pp. 23, 29; Appendix A, p. 133 of MRID 50406038; p. 22 of MRID 50406044). Due to the low signals of the confirmatory transitions for M850H003 (m/z 357 \rightarrow 137) and M850H004 (m/z 385 \rightarrow 255), the recommended 10 μ L injection volume was not suitable for quantitation during the ILV study; therefore, the 40 μ L injection volume was used to obtain the acceptable sensitivity. The recommended changes were included in the ECM final report (p. 33 of MRID 50406037).

Table 1: Method Validation Recoveries for Residues of Trifludimoxazin (BAS 850 H) in Fortified Control Soil Samples Using BASF Analytical Method D1401/02¹

Matrix	Fortification Levels (mg/kg)	n	Recovery (%)	Average Recovery (%)	Standard Deviation	%RSD ¹
North Dakota Soil (Clay, 0-6") ²	BAS 850 H: Primary (<i>m/z</i> 413→74)					
	0.001	5	90, 84, 75, 77, 77	80	6.20	7.70
	0.01	5	116, 96, 122, 118, 120	114	10.7	9.30
	Overall	10	Range: 75–122	97	19.8	20.3
	BAS 850 H: Confirmatory (<i>m/z</i> 413→134)					
	0.001	5	99, 102, 93, 101, 83	95	7.70	8.10
	0.01	5	120, 93, 122, 116, 121	115	12.3	10.8
	Overall	10	Range: 83–122	105	14.0	13.3
	M850H001: Primary Quantitation (<i>m/z</i> 397→114)					
	0.001	5	111, 108, 112, 113, 94	108	7.80	7.30
	0.01	5	107, 87, 111, 118, 106	106	11.5	10.9
	Overall	10	Range: 87–118	107	9.30	8.70
	M850H001: Confirmatory Quantitation (<i>m/z</i> 397→141)					
	0.001	5	120, 130, 121, 115, 100	117	10.9	9.30
	0.01	5	96, 81, 112, 117, 96	100	14.4	14.3
	Overall	10	Range: 81–130	109	14.9	13.7
	M850H002: Primary Quantitation (<i>m/z</i> 373→193)					
	0.001	5	106, 89, 109, 119, 109	106	10.8	10.1
	0.01	5	79, 89, 104, 100, 91	93	10.0	10.8
	Overall	10	Range: 79–119	100	12.1	12.2
	M850H002: Confirmatory Quantitation (<i>m/z</i> 373→323)					
	0.001	5	107, 98, 109, 105, 101	104	4.30	4.10
	0.01	5	72, 88, 99, 84, 83	85	9.80	11.5
	Overall	10	Range: 72–109	95	12.2	12.9
	M850H003: Primary Quantitation (<i>m/z</i> 357→307)					
	0.001	5	98, 100, 114, 115, 112	108	8.10	7.60
	0.01	5	87, 119, 106, 106, 99	103	11.7	11.3
	Overall	10	Range: 87–119	106	9.80	9.30
	M850H003: Confirmatory Quantitation (<i>m/z</i> 357→137)					
	0.001	5	104, 92, 112, 115, 117	108	10.4	9.70
0.01	5	66, 105, 103, 116, 110	100	19.6	19.6	
Overall	10	Range: 66–117	104	15.3	14.7	
M850H004: Primary Quantitation (<i>m/z</i> 385→103)						
0.001	5	100, 99, 104, 106, 118	105	7.60	7.20	
0.01	5	84, 99, 100, 100, 90	95	7.10	7.50	
Overall	10	Range: 84–118	100	8.90	8.90	
M850H004: Confirmatory Quantitation (<i>m/z</i> 385→255)						
0.001	5	103, 92, 111, 120, 102	106	10.5	9.90	
0.01	5	90, 103, 94, 91, 118	99	11.5	11.6	
Overall	10	Range: 90–120	102	10.9	10.7	

Table 1: Method Validation Recoveries for Residues of Trifludimoxazin (BAS 850 H) in Fortified Control Soil Samples Using BASF Analytical Method D1401/02¹ (continued)

Matrix	Fortification Levels (mg/kg)	n	Recovery (%)	Average Recovery (%)	Standard Deviation	%RSD ¹
North Carolina Soil (Loamy Sand, 0-3'') ³	BAS 850 H: Primary (<i>m/z</i> 413→74)					
	0.001	5	100, 94, 103, 113, 100	102	7.00	6.90
	0.01	5	119, 120, 97, 119, 120	115	10.2	8.90
	Overall	10	Range: 94–120	109	10.7	9.80
	BAS 850 H: Confirmatory (<i>m/z</i> 413→134)					
	0.001	5	94, 89, 102, 97, 104	97	6.10	6.20
	0.01	5	114, 106, 87, 100, 108	103	10.1	9.80
	Overall	10	Range: 87–114	100	8.40	8.40
	M850H001: Primary Quantitation (<i>m/z</i> 397→114)					
	0.001	5	104, 103, 110, 114, 112	109	4.70	4.30
	0.01	5	96, 103, 77, 98, 100	95	10.2	10.7
	Overall	10	Range: 77–114	102	10.4	10.2
	M850H001: Confirmatory Quantitation (<i>m/z</i> 397→141)					
	0.001	5	98, 104, 107, 94, 125	106	12.1	11.5
	0.01	5	100, 105, 70, 103, 94	94	14.2	15.1
	Overall	10	Range: 70–125	100	13.8	13.8
	M850H002: Primary Quantitation (<i>m/z</i> 373→193)					
	0.001	5	100, 117, 116, 104, 100	108	8.30	7.70
	0.01	5	101, 106, 99, 108, 111	105	5.10	4.90
	Overall	10	Range: 99–117	106	6.60	6.20
	M850H002: Confirmatory Quantitation (<i>m/z</i> 373→323)					
	0.001	5	107, 111, 104, 94, 112	105	7.30	6.90
	0.01	5	105, 104, 91, 109, 107	103	7.30	7.10
	Overall	10	Range: 91–112	104	7.00	6.70
	M850H003: Primary Quantitation (<i>m/z</i> 357→307)					
	0.001	5	101, 113, 101, 103, 119	107	8.10	7.50
	0.01	5	119, 96, 97, 103, 101	103	9.30	9.00
	Overall	10	Range: 96–119	105	8.50	8.00
	M850H003: Confirmatory Quantitation (<i>m/z</i> 357→137)					
	0.001	5	117, 124, 109, 105, 110	113	7.50	6.60
0.01	5	120, 114, 103, 92, 109	108	10.9	10.1	
Overall	10	Range: 92–124	110	9.20	8.40	
M850H004: Primary Quantitation (<i>m/z</i> 385→103)						
0.001	5	111, 108, 105, 82, 109	103	12.1	11.8	
0.01	5	102, 103, 94, 102, 97	100	4.00	4.00	
Overall	10	Range: 82–111	101	8.70	8.60	
M850H004: Confirmatory Quantitation (<i>m/z</i> 385→255)						
0.001	5	121, 122, 107, 76, 95	104	19.5	18.7	
0.01	5	127, 93, 94, 96, 117	105	15.5	14.7	
Overall	10	Range: 76–127	105	16.6	15.9	

¹ Relative Standard Deviation = (Standard Deviation ÷ Average Recovery) × 100

² Clay soil (reported as clay loam in study) from BASF Study 411690, Trial R130128 (BASF Reg. Doc. No. 2016/7005812).

³ Loamy sand soil from BASF Study 698742, Trial R140774 (BASF Reg. Doc. No. 2016/7005814)

Table obtained from Table 2, pp. 11-12 of MRID 50406044. Recovery results were corrected for residues quantified in the controls; however, no residues were quantified (Appendix A, p. 85 of MRID 50406037). **91-110**

1 Clay (0-3 inch soil depth; TRT SC-1 0-3"; 31% sand, 29% silt, 40% clay; pH 7.6 in saturated paste; 2.1% organic carbon – Walkley Black; 3.7% organic matter – Walkley Black) and loamy sand (0-3 inch soil depth; SC-10 0-3"; 86% sand, 12% silt, 2% clay; pH 5.4 in saturated paste; pH 4.9 in 0.01M CaCl₂, 1:1; 0.48% organic carbon – Walkley Black; 0.83% organic matter – Walkley Black) soil samples collected under trifludimoxazin terrestrial field dissipation studies (BASF Study 411690, Trial R130128, BASF Reg. Doc. No. 2016/7005812 and BASF Study 698742, Trial R140774, BASF Reg. Doc. No. 2016/7005814) were used in the study (USDA soil texture classification; p. 7 of MRID 50406044; Appendix A, Appendix A, pp. 55-56 of MRID 50406037). Test systems were characterized by Agvise Laboratories, Northwood, North Dakota. The soil textures were verified by the reviewer using USDA-NRCS technical support tools.

Table 5: Independent Laboratory Validation Summary Results of Method D1401/02: Trifludimoxazin and Metabolites in Fortified Control Soil Samples¹

Matrix	Fortification Levels (mg/kg)	n	Recovery (%)	Average Recovery (%)	Standard Deviation	%RSD ^a
Clay Soil ^b	BAS 850 H					
	Quantitation (<i>m/z</i> 413→74)					
	0.001	5	87.1, 91.2, 91.6, 94.5, 95.2	91.9	3.18	3.46
	0.010	5	106, 105, 97.8, 105, 102	103	3.33	3.23
	Overall	10	Range: 87.1–106	97.5	6.62	6.79
	Confirmation (<i>m/z</i> 413→134)					
	0.001	5	92.1, 95.3, 98.0, 91.6, 102	95.9	4.48	4.67
	0.010	5	104, 114, 98.8, 112, 107	107	6.05	5.65
	Overall	10	Range: 91.6–114	102	7.79	7.67
	M850H001					
	Quantitation (<i>m/z</i> 397→114)					
	0.001	5	85.2, 91.1, 88.2, 94.7, 93.1	90.5	3.80	4.20
	0.010	5	103, 102, 97.9, 106, 98.7	102	3.31	3.26
	Overall	10	Range: 85.2–106	96.0	6.74	7.02
	Confirmation (<i>m/z</i> 397→141)					
	0.001	5	93.2, 97.1, 86.4, 96.1, 98.3	94.2	4.77	5.06
	0.010	5	102, 101, 102, 103, 98.4	101	1.91	1.88
	Overall	10	Range: 86.4–103	97.8	5.05	5.17
	M850H002					
	Quantitation (<i>m/z</i> 373→193)					
	0.001	5	89.3, 120, 99.0, 102, 102	102	11.1	10.8
	0.010	5	89.2, 106, 110, 116, 111	106	10.3	9.63
	Overall	10	Range: 89.2-120	104	10.3	9.86
	Confirmation (<i>m/z</i> 373→323)					
	0.001	5	87.8, 117, 103, 105, 104	103	10.3	9.94
	0.010	5	92.0, 108, 114, 117, 112	109	9.84	9.07
	Overall	10	Range: 87.8-117	106	9.87	9.32
	M850H003					
	Quantitation (<i>m/z</i> 357→307)					
	0.001	5	91.0, 110, 102, 101, 102	101	6.88	6.80
	0.010	5	91.6, 106, 109, 110, 105	104	7.27	6.98
	Overall	10	Range: 91.0-110	103	6.87	6.69
Confirmation (<i>m/z</i> 357→137)						
0.001	5	95.3, 104, 104, 97.0, 98.7	99.6	3.79	3.81	
0.010	5	93.2, 108, 107, 109, 106	105	6.57	6.27	
Overall	10	Range: 93.2-109	102	5.73	5.61	
M850H004						
Quantitation (<i>m/z</i> 385→103)						
0.001	5	86.6, 105, 95.4, 104, 95.6	97.3	7.57	7.78	
0.010	5	92.9, 108, 113, 112, 114	108	8.79	8.13	
Overall	10	Range: 86.6-114	103	9.57	9.32	
Confirmation (<i>m/z</i> 385→255)						
0.001	5	83.6, 104, 95.4, 102, 96.7	96.3	8.02	8.32	
0.010	5	92.2, 109, 116, 112, 113	108	9.37	8.65	
Overall	10	Range: 83.6-116	102	10.4	10.1	

^a Relative Standard Deviation = (Standard Deviation ÷ Average Recovery) × 100

^bClay soil (reported as clay loam in study) from BASF Study 411690, Trial R130128; (30-36 inch)

Table obtained from Table 6, p. 23 of MRID 50406044. Uncorrected recovery results were reported (Appendix C, pp. 397-424 of MRID 50406038).

¹Clay (30-36 inch soil depth; TRT SC-7 30-36"; 29% sand, 19% silt, 52% clay; pH 8.0 in saturated paste; 0.52% organic carbon – Walkley Black; 0.90% organic matter – Walkley Black) soil collected under a trifludimoxazin terrestrial field dissipation study (BASF Study 411690, Trial R130128, BASF Reg. Doc. No. 2016/7005812) was characterized, provided by BASF, and used in the study (p. 20 of MRID 50406044; p. 20; Appendix E, p. 185 of MRID 50406038). The soil texture was verified by the reviewer using USDA-NRCS technical support tools.

III. Method Characteristics

The LOQ was defined as the lowest fortification level tested (p. 4 of MRID 50406044). The LOQ for residues of BAS 850 H (including parent compound and its metabolites M850H001, M850H002, M850H003, and M850H004) in soil was 0.001 mg/kg. The method LOD was defined as the lowest analyte concentration injected as a calibration solution, resulting in an LOD of 0.0002 mg/kg (20% of the LOQ). The LOD for trifludimoxazin and M850H001 residues in soil was shown to be detectable as the absolute amount of analyte injected (0.002 ng) into the LC-MS/MS when the lowest calibration standard was analyzed (0.25 ng/mL). The method LOD for M850H002, M850H003 and M850H004 in soil was shown to be detectable as the absolute amount of analyte injected (0.0002 ng) into the LC-MS/MS when the lowest calibration standard was analyzed (0.025 ng/mL). All analytes demonstrated acceptable signal to noise ratio (S/N > 3:1).

The LOD evaluation for BASF analytical method D1401/02 was performed for both positive and negative modes.

To determine the least sensitive analyte and transition for the purpose of LOD determination, solvent based standard solutions were injected using the validated analytical LC-MS/MS method (all transitions were evaluated) from method D1401/02. It was determined qualitatively that the MS/MS ion transition for trifludimoxazin (m/z 413 \rightarrow m/z 134) was the least sensitive transition and, therefore, the best candidate to conduct the LOD evaluation for the positive mode.

The MDL was determined to be 0.000008 ng on-column for trifludimoxazin. To determine this value, the seven (7) control samples were run through the extraction procedure and were fortified with standard solution prior to the LC-MS/MS determination step to achieve a concentration of 0.0002 ng/mL, and 0.10 mL (100 μ L, 0.02 ng on-column) of each sample was injected on the LC-MS/MS according to method D1401/02. Calculation of MDL for trifludimoxazin was conducted according to the table provide in 40 CFR Ch. 1 Part 136 appendix B.

Based on this calculated MDL, the LOD for trifludimoxazin was set at 0.025 ng on-column (i.e. 0.25 ng/mL injected at 0.10 mL [100 μ L]). This increase from the MDL to the LOD is to account for variability in the residue method, natural drift of the LC-MS/MS instrumentation, potential contamination issues, untested matrix effects, and potential unseen background interferences. Below is a table that details all the results for this injection.

Table 6: Calculation of MDL for Trifludimoxazin (m/z 413 → m/z 134) for Method D1401/02, using the Methodology Set Forth in 40 CFR Ch 1 Part 136 Appendix B

0.0002 ng/mL Matrix-Spike Replicate	Calculated Concentration (ng/mL)
1	0.00019
2	0.00017
3	0.00023
4	0.00020
5	0.00024
6	0.00023
7	0.00019
Standard Deviation (S) =	0.000026
N-1 =	6
Critical t value (t) =	3.143
Injection Volume (μL)	100
MDL (ng/mL) =	0.00008
MDL (ng on-column) =	0.000008
LOD (ng on-column) =	0.025
LOD Equivalent to incurred sample at =	0.0002 mg/kg

$$\text{MDL on-column (ng)} = \text{MDL (ng/mL)} \times \text{Injection Volume (mL)}$$

Table obtained from Table 3, p. 13 of MRID 50406044.

It was determined qualitatively that the MS/MS ion transition for M850H004 (m/z 385 → m/z 255) was the least sensitive transition and, therefore, the best candidate to conduct the LOD evaluation for the negative mode.

The MDL was determined to be 0.000002 ng on-column for M850H004. To determine this value, the seven (7) control samples were run through the extraction procedure and were fortified with standard solution prior to the LC-MS/MS determination step to achieve a concentration of 0.0002 ng/mL and 0.02 mL (20 μL, 0.004 ng on-column) of each sample was injected on the LC-MS/MS according to method D1401/02. Calculation of MDL for M850H004 was conducted according to the table provide in 40 CFR Ch. 1 Part 136 appendix B.

Based on this calculated MDL, the LOD for M850H004 was set at 0.0005 ng on-column (i.e. 0.025 ng/mL injected at 0.02 mL [20 μL]). This increase from the MDL to the LOD is to account for variability in the residue method, natural drift of the LC-MS/MS instrumentation, potential contamination issues, untested matrix effects, and potential unseen background interferences. Below is a table that details all the results for this injection.

Table 7: Calculation of MDL for M850H004 (m/z 385 → m/z 255) for Method D1401/02, using the Methodology Set Forth in 40 CFR Ch 1 Part 136 Appendix B

0.0002 ng/mL Matrix-Spike Replicate	Calculated Concentration (ng/mL)
1	0.00024
2	0.00017
3	0.00017
4	0.00023
5	0.00015
6	0.00023
7	0.00024
Standard Deviation (S) =	0.000039
N-1 =	6
Critical t value (t) =	3.143
Injection Volume (μL) =	20
MDL (ng/mL) =	0.00012
MDL (ng on-column) =	0.000002
LOD (ng on-column) =	0.0005
LOD Equivalent to incurred sample at =	0.0002 mg/kg

$$\text{MDL on-column (ng)} = \text{MDL (ng/mL)} \times \text{Injection Volume (mL)}$$

Table obtained from Table 4, p. 14 of MRID 50406044.

Table 8. Method Characteristics - Analytical Method D1401/02

Analyte		Trifludimoxazin (BAS 850 H; Reg.No. 5654329)	M850H001 (Reg.No. 5749359)	M850H002 (Reg.No. 5757725)	M850H003 (Reg.No. 5757726)	M850H004 (Reg.No. 5833884)	
Limit of Quantitation (LOQ)	ECM	0.001 mg/kg (0.030 µg/mL)					
	ILV						
Limit of Detection (LOD)	ECM	0.0002 mg/kg (20% of the LOQ)					
	ILV						
Linearity (calibration curve r and concentration range) ¹	ECM	Clay	r = 0.9971 (Q) r = 0.9991 (C)	r = 0.9979 (Q) r = 0.9983 (C)	r = 0.9901 (Q) r = 0.9908 (C)	r = 0.9974 (Q) r = 0.9972 (C)	r = 0.9923 (Q) r = 0.9922 (C)
		Loamy sand	r = 0.9984 (Q) r = 0.9979 (C)	r = 0.9991 (Q) r = 0.9985 (C)	r = 0.9954 (Q) r = 0.9970 (C)	r = 0.9995 (Q) r = 0.9972 (C)	r = 0.9950 (Q) r = 0.9967 (C)
		Range	0.25-10 ng/mL		0.025-1.25 ng/mL		
	ILV	Clay	r = 0.9998 (Q) r = 0.9987 (C)	r = 0.9997 (Q) r = 0.9990 (C)	r = 0.9997 (Q) r = 0.9998 (C)	r = 0.9997 (Q & C)	r = 0.9986 (Q) r = 0.9987 (C)
		Range	0.25-10 ng/mL		0.025-10 ng/mL		
	Repeatable	ECM ²	Yes at LOQ and 10×LOQ (two characterized soil matrices)				
ILV ^{3,4}		Yes at LOQ and 10×LOQ (one characterized soil matrix).					
Reproducible		Yes at LOQ and 10×LOQ					
Specific	ECM	Yes, no matrix interferences were observed. Some minor baseline noise near the analyte peak and non-uniform peak integration was noted in several LOQ and 10×LOQ representative chromatograms. Contaminant near the BAS 850 H peak was observed (RT 7.82 min.; peak height/area variable based on soil). ⁵		Yes, no matrix interferences were observed.			
		Loamy sand soil representative chromatograms contained less matrix interferences than those of clay soil.					
	ILV ⁷	Yes, no matrix interferences were observed. Contaminant near the BAS 850 H peak was	Yes, matrix interferences were <5% of the LOQ (based on peak area). Minor baseline noise interfered with analyte peak attenuation and integration.				

Analyte	Trifludimoxazin (BAS 850 H; Reg.No. 5654329)	M850H001 (Reg.No. 5749359)	M850H002 (Reg.No. 5757725)	M850H003 (Reg.No. 5757726)	M850H004 (Reg.No. 5833884)
	observed (<i>ca.</i> RT 7.75 min.; peak height/area <i>ca.</i> 5% LOQ). ⁶				

Data were obtained from MRID 50406044; Appendix A, Appendix E, pp. 91-110 (raw data sheets); Appendix A, Appendix J, pp. 140-145 (calibration curves) and Appendix A, Appendix J, pp. 154-239 (chromatograms) of MRID 50406037; Sections A-J, pp. 49-129 (calibration curves and chromatograms) and Appendix C, pp. 141-160 (raw data sheets) of MRID 50406038. Q = Quantitation ion transition; C = Confirmation ion transition or method.

- 1 Solvent-based calibration standards were used in the ECM and ILV. 5 A confirmation method is not usually required when LC/MS or GC/MS is the primary method to generate study data; therefore, linearity deficiencies in the confirmation analysis do not affect the validity of the method.
 - 2 In the ECM, clay (0-3 inch soil depth; TRT SC-1 0-3"; 31% sand, 29% silt, 40% clay; pH 7.6 in saturated paste; 2.1% organic carbon – Walkley Black; 3.7% organic matter – Walkley Black) and loamy sand (0-3 inch soil depth; SC-10 0-3"; 86% sand, 12% silt, 2% clay; pH 5.4 in saturated paste; pH 4.9 in 0.01M CaCl₂, 1:1; 0.48% organic carbon – Walkley Black; 0.83% organic matter – Walkley Black) soil samples collected under trifludimoxazin terrestrial field dissipation studies (BASF Study 411690, Trial R130128, BASF Reg. Doc. No. 2016/7005812 and BASF Study 698742, Trial R140774, BASF Reg. Doc. No. 2016/7005814) were used in the study (USDA soil texture classification; p. 7 of MRID 50406044; Appendix A, Appendix A, pp. 55-56 of MRID 50406037). Test systems were characterized by Agvise Laboratories, Northwood, North Dakota. The soil textures were verified by the reviewer using USDA-NRCS technical support tools.
 - 3 In the ILV, clay (30-36 inch soil depth; TRT SC-7 30-36"; 29% sand, 19% silt, 52% clay; pH 8.0 in saturated paste; 0.52% organic carbon – Walkley Black; 0.90% organic matter – Walkley Black) soil collected under a trifludimoxazin terrestrial field dissipation study (BASF Study 411690, Trial R130128, BASF Reg. Doc. No. 2016/7005812) was characterized, provided by BASF, and used in the study (p. 20 of MRID 50406044; p. 20; Appendix E, p. 185 of MRID 50406038). The soil texture was verified by the reviewer using USDA-NRCS technical support tools.
 - 4 The independent laboratory validation of BASF method (D1401/02) was successfully completed for all analytes in the first trial with modifications to the analytical instrument and parameters (pp. 23, 29; Appendix A, p. 133 of MRID 50406038; p. 22 of MRID 50406044). Due to the low signals of the confirmatory transitions for M850H003 (m/z 357 → 137) and M850H004 (m/z 385 → 255), the recommended 10 µL injection volume was not suitable for quantitation during the ILV study; therefore, the 40 µL injection volume was used to obtain the acceptable sensitivity. The recommended changes were included in the ECM final report (p. 33 of MRID 50406037).
 - 5 Based on Figures J.17-J.18, pp. 184-186 and Figures J.27-J.28, pp. 199-201 of MRID 50406037.
 - 6 Based on Section A, pp. 53-54, 60-61 of MRID 50406038. The reviewer noted that the contaminant in the C ion chromatograms were *ca.* 5XLOQ (reviewer-estimate); however, this did not affect the specificity of the method since it was isolated to the C analysis. A confirmation method is not usually required when LC/MS or GC/MS is the primary method to generate study data; therefore, deficiencies in the confirmation analysis of the performance data, linearity, and supporting chromatograms do not affect the validity of the method.
- Linearity is satisfactory when $r \geq 0.995$ [updated DER acceptance criteria (11/2019); Linearity criterion is consistent with Superfund analytical methods for inorganic analytes (National Functional Guidelines for Inorganic Superfund Methods Data Review, EPA-540-R-2017-001, January 2017. https://www.epa.gov/sites/production/files/201701/documents/national_functional_guidelines_for_inorganic_superfund_methods_data_review_01302_017.pdf)].

IV. Method Deficiencies and Reviewer's Comments

1. The LOQ is greater than the lowest toxicological level of concern (IC25 of 0.022 µg/kg, MRID 50406453) in soil for trifludimoxazin and its four metabolites.
2. ECM linearity (quantitation ion analysis) was not satisfactory for M850H002 in clay soil ($r = 0.9901$) and M850H004 in clay soil ($r = 0.9923$; Appendix A, Appendix E, pp. 91-110 of MRID 50406037). Linearity is satisfactory when $r \geq 0.995$. The linearity of the confirmation analysis calibration curves was not satisfactory for M850H002 and M850H004 in clay soil; however, deficiencies in the confirmation analysis do not affect the validity of the method since a confirmation method is not usually required when LC/MS or GC/MS is the primary method to generate study data.

The ECM was performed under the following guidelines, in addition to OCSPP 850.6100: SANCO/3029/99 rev 4. (11/07/2000; p. 1 of MRID 50406037). Under these guidelines, the requirement for the linear correlation coefficient (r) is ≥ 0.99 . No deviation from these linearity requirements was noted.

3. Although the ECM and ILV studies were conducted at separate BASF laboratories, there was an unusually large amount of information sharing between the two entities both pre-study and during the analytical phases (Appendix G, p. 193 of MRID 50406037). However, these correspondences were transparent and do not suggest collusion. There was no evidence that the analysts, study director, equipment, instruments, and supplies of the two laboratories were not distinct.
4. The ECM and ILV soil matrices were sourced from trifludimoxazin terrestrial field dissipation studies; however, only one soil matrix was included in the ILV. Even though a certain number of soil matrices is not specified in the OCSPP guidelines, more than one soil/soil matrix would need to be included in an ILV in order to cover the range of soils used in the terrestrial field dissipation studies. The ECM included two soils, while the ILV only included one. The ILV should be a more rigorous test of the method, and therefore, should include at least as many test matrices as the ECM.
5. The reviewer noted the persistent, nearby contaminant in the ECM and ILV representative chromatograms of BAS 850 H (Figures J.17-J.18, pp. 184-186 and Figures J.27-J.28, pp. 199-201 of MRID 50406037; Section A, pp. 53-54, 60-61 of MRID 50406038).
6. It was reported for the ILV that one sample set of 13 samples required *ca.* 8 hours of work, excluding LC/MS/MS and calculation of results (p. 28 of MRID 50406038).

V. References

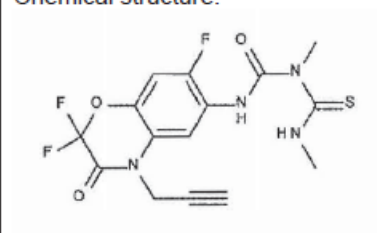
U.S. Environmental Protection Agency. 2012. Ecological Effects Test Guidelines, OCSPP 850.6100, Environmental Chemistry Methods and Associated Independent Laboratory Validation. Office of Chemical Safety and Pollution Prevention, Washington, DC. EPA 712-C-001.

40 CFR Part 136. Appendix B. Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.11, pp. 317-319.

MRID 50406044. Description of methods for analysis of soil (parent and metabolites), Summary and evaluation (Tier II), BASF Reg. Doc. # 2018/7000014 US. BASF Corporation 26 Davis Drive P.O. Box 13528 Research Triangle Park, NC 27709.

Attachment 1: Chemical Names and Structures

BASF Code Name:	BAS 850 H	Chemical structure:
BASF Registry Number:	5654329	
IUPAC Name:	1,5-dimethyl-6-thioxo-3-(2,2,7-trifluoro-3-oxo-4-(prop-2-yn-1-yl)-3,4-dihydro-2H-1,4-benzoxazin-6-yl)-1,3,5-triazinane-2,4-dione	
CAS Number:	1258836-72-4	
Molecular Formula:	C ₁₈ H ₁₁ F ₃ N ₄ O ₄ S	
Molecular Weight:	412.3 g/mol	
Batch No.:	L84-130	
Purity:	99.2%	Chemical structure:
BASF Code Name:	M850H001	
BASF Registry Number:	5749359	
IUPAC Name:	1,3-dimethyl-5-[2,2,7-trifluoro-3-oxo-4-(prop-2-yn-1-yl)-3,4-dihydro-2H-1,4-benzoxazin-6-yl]-1,3,5-triazinane-2,4,6-trione	
Molecular Formula:	C ₁₆ H ₁₁ F ₃ N ₄ O ₅	
Molecular Weight:	396.3 g/mol	
Batch No.:	L80-52	
Purity:	98.7%	Chemical structure:
BASF Code Name:	M850H002	
BASF Registry Number:	5757725	
IUPAC Name:	1,5-dimethyl-6-thioxo-3-(2,2,7-trifluoro-3-oxo-3,4-dihydro-2H-1,4-benzoxazin-6-yl)-1,3,5-triazinane-2,4-dione	
Molecular Formula:	C ₁₃ H ₉ F ₃ N ₄ O ₄ S	
Molecular Weight:	374.3 g/mol	
Batch No.:	L84-162	
Purity:	96.8%	Chemical structure:
BASF Code Name:	M850H003	
BASF Registry Number:	5757726	
Chemistry Name:	1,3-dimethyl-5-(2,2,7-trifluoro-3-oxo-3,4-dihydro-2H-1,4-benzoxazin-6-yl)-1,3,5-triazinane-2,4,6-trione	
Molecular Formula:	C ₁₃ H ₉ F ₃ N ₄ O ₅	
Molecular Weight:	358.2 g/mol	
Batch No.:	L84-160; L85-70	
Purity:	99.2%; 99.4%	
Expiry date:	August 1, 2017; April 1, 2018	

BASF Code Name:	M850H004	Chemical structure: 
BASF Registry Number:	5833884	
IUPAC Name:	N,N-dimethyl-N'-(2,2,7-trifluoro-3-oxo-4-(prop-2-yn-1-yl)-3,4-dihydro-2H-1,4-benzoxazin-6-yl)dicarbonimidothioicdiamide	
Molecular Formula:	C ₁₅ H ₁₃ F ₃ N ₄ O ₃ S	
Molecular Weight:	386.4 g/mol	
Batch No.:	L85-50	
Purity:	99.5%	
Expiry date:	April 1, 2018	

Figures from pp. 17-18 of MRID 50406037.