



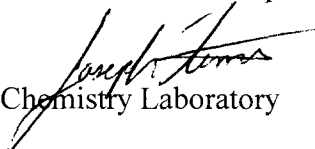
**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**  
**OFFICE OF PESTICIDE PROGRAMS**  
**ENVIRONMENTAL CHEMISTRY LABORATORY**  
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November 9, 2006

MEMORANDUM

EPA DP Barcode: 302078, 332765

**SUBJECT:** Flucarbazone-sodium Method In Soil Review Report No. ECM0222S1-S4

**FROM:** Joseph B Ferrario, Chief  
OPP/BEAD/Environmental Chemistry Laboratory  


**To:** Cara Dzubow, Program Analyst  
OPP/Environmental Fate and Effects Division  
Information and Support Branch (7507C)

The Environmental Fate and Effects Division (EFED) has requested an Environmental Chemistry Method Review on flucarbazone-sodium (MKH 6562) and its metabolites NODT, sulfonic acid, and sulfonamide in Soil using the method submitted by Bayer CropScience in accordance with the registration of MRID No. 462237-02. The method validation data was reviewed and the conclusions included in the attached Environmental Chemistry Method Review Report.

The following report includes an overview of the method and the method completeness, statements of adherence to EPA regulations, a presentation of results and a discussion of problems found in the registrant method. A statement of method acceptability is also included.

If you have questions concerning this report, please contact Shanda L Bennett at (228) 688 - 3251.

Attachments

cc: Dr. Christian Byrne, QA Officer  
BEAD/ECL

Shanda L Bennett, Chemist  
BEAD/ECL

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**Data Requirement:** PMRA Data Code: NA  
EPA DP Barcode: - 302078,332765  
OECD Data Point: NA  
EPA Guideline: ECM 0222S1-S4

**Test material:**

Common name: Flucarbazono-sodium  
Chemical name: 1H-1, 2, 4-triazole-carboxamide, 4, 5-dihydro-3-methoxy-4-methyl-5-oxo-N-((2-(trifluoromethoxy) phenyl) sulfonyl)-sodium salt  
IUPAC: sodium N-(2-trifluoromethoxy-sulfonyl)-4, 5-dihydro-3-methoxy-4-methyl-5-oxo-1H-1, 2, 4-triazole-1-carboxamide

**Primary Evaluator:** Shanda Bennett **Date:** 11/06/06  
Shanda Bennett, Chemist, EPA/OPP/BEAD/ECB  
**Peer Reviewer:** Charles Kennedy **Date:** 11/06/06  
Charles Kennedy, EPA/OPP/BEAD/ECB  
**QA Officer:** Dr. Christian Byrne **Date:** 11/06/06  
Dr. Christian Byrne, EPA/OPP/BEAD/ECB

**ANALYTICAL METHOD:** 462237-02, Lam, C. K., Qadri, S. S. December 15, 2003. "Analytical Method for the Determination of MKH 6562 and metabolites NODT, sulfonic acid, and sulfonamide in Soil by High Performance Liquid Chromatography Tandem Mass Spectrometry (LC-MS/MS)". The unpublished study was performed by Bayer CropScience, Bayer Research Park, 17745 South Metcalf Avenue, Stillwell, Kansas 66085-9104. The study was sponsored by Arvesta Corporation, 100 First Street, Suite 1700, San Francisco, California 94105. Pages 1-43.

**EXECUTIVE SUMMARY**

The method is applicable for the quantitative determination of residues, Flucarbazono-sodium (MKH 6562) and its metabolites MKH 6562 sulfonamide, MKH 6562 sulfonic acid (ammonium salt) and NODT (N, O-dimethyltriazolinone).

The method was submitted to EPA by Arvesta Corporation to support the registration of the herbicide – Flucarbazono-sodium. The method was performed by Bayer CropScience in Stillwell, KS and sponsored by Arvesta Corporation in San Francisco, CA. This method was conducted in the spirit of EPA's Good Laboratory Practice Standards, Title 40 Code of Federal Regulations Part 160. An independent laboratory validation was submitted with this method. It was entitled, "Independent Laboratory Validation of Method 200748: 'Analytical Method for the Determination of MKH 6562 and Metabolites NODT, Sulfonic acid, and Sulfonamide in Soil by High

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Performance Liquid Chromatography Tandem Mass Spectrometry (LC-MS/MS' ". The ILV was performed by ALS Laboratory Group, Environmental Division in Edmonton, Alberta Canada. ECB finds this method acceptable as submitted.

**Method Summary:** Residues of flucarbazono-sodium were extracted from fortified soil (from study sites at Washington and Oklahoma) with an acetonitrile/ 0.2 M acidified ammonium acetate solution (4:1, v/v) at room temperature. The soil and solution was transferred into a sample vial, capped, shaken, and centrifuged. An aliquot of the extracts was transferred into a culture tube and the internal standards were added. The solution was evaporated to dryness and reconstituted with an water/ammonium acetate/methanol solution. The extract was filtered into a vial and stored in the freezer until LC-MS/MS analysis.

**METHOD ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS**

Based on the parameters set in the *Ecological Effects Test Guidelines, OPPTS 850.7100, Data Reporting for Environmental Chemistry Methods*; "Public Draft." (U.S. Environmental Protection Agency. Office of Prevention, Pesticides, and Toxic Substances (7101). U.S. Government Printing Office: Washington, DC, 1996, EPA-712-C-96-348), ECL finds this method acceptable.

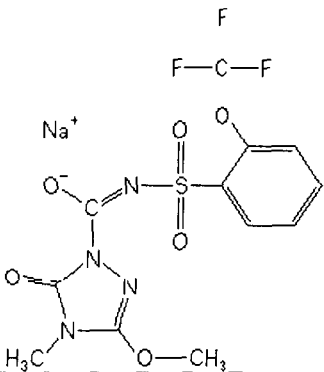
**COMPLIANCE**

Signed and dated statements that this method was conducted in the spirit of the Good Laboratory Practice Standards, 40 CFR 160 were present in the method. Also, a statement of non-confidentiality on the basis of the method falling within the scope of FIFRA Section 10 (d)(1)(A)(B), or (C) was signed and dated along with information on the Quality Assurance inspection dates and signatures.

**A. BACKGROUND INFORMATION**

Flucarbazono-sodium, sodium N-(2-trifluoromethoxy-sulfonyl)-4, 5-dihydro-3-methoxy-4-methyl-5-oxo-1H-1, 2, 4-triazole-1-carboxamide is a herbicide that is under development by Arvesta Corporation in San Francisco, CA. It controls annual grasses and some broadleaf weeds in wheat.

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<b>TABLE A.1. Test Compound Nomenclature</b>	
Compound	Chemical Structure 
Common name	Flucarbazono-sodium
Company experimental name	MKH 6562
IUPAC name	sodium N-(2-(trifluoromethoxy-sulfonyl)-4, 5-dihydro-3-methoxy-4-methyl-5-oxo-1H-1, 2, 4-triazole-1-carboxamide
CAS Name	1H-1, 2, 4-triazole-carboxamide, 4, 5-dihydro-3-methoxy-4-methyl-5-oxo-N-((2-(trifluoromethoxy) phenyl) sulfonyl)-sodium salt
CAS #	181274-17-9

<b>TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound</b>	
Parameter	Value
Melting point/range	200°C (with decomposition)
pH	NA
Density	NA
Water solubility (20 °C)	3.1% (w/w) for flucarbazono-sodium
Solvent solubility (mg/ml at 20 °C)	44mg/L
Vapor pressure at ___ °C	1 x 10 <sup>-10</sup> mm Hg at 20°C
Dissociation constant (pK <sub>a</sub> )	NA
Octanol/water partition coefficient	NA
UV/visible absorption spectrum	NA

## MATERIALS AND METHODS

### B.1. Principle of Method

Ten grams of soil is weighed into a 60 mL vial and twenty milliliters of a solution of acetonitrile/0.2 M acidified ammonium acetate (4:1, v:v) added and mechanically shaken for 1 hour. The mixture is centrifuged at approximately 2300 rpm for 10-15

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minutes. An 8 milliliter aliquot is sampled and a mixed internal standard solution added and mixed. This solution is reduced to dryness and reconstituted to 1 mL with a solution of 95:5 (water:100 mM ammonium acetate in methanol). This solution is vortexed and filtered through a 0.45  $\mu$ m filter disc into a HPLC vial and stored until LC-MS/MS analysis.

<b>TABLE B.1.1.</b>	<b>Summary Parameters for the Analytical Method Used for the Quantitation of Chemical Residues in Matrices Studied</b>
Method ID	ECM0222S1-S4
Analyte(s)	Flubarbazone-sodium (MKH 6562), NODT, Sulfonic acid, and Sulfonamide
Extraction solvent/technique	Soil extracted in acetonitrile/acidified ammonium acetate solution on a mechanical shaker table, then centrifuged.
Cleanup strategies	Filter extract through a 0.45- $\mu$ m filter disc
Instrument/Detector	TSQ 7000 LC/Tandem Mass Spectrometer with ESI or APCI interface and gradient HPLC

## C. RESULTS AND DISCUSSION

### C.1. Recovery Results Summary

<b>TABLE C.1.1. Recovery Results from Method Validation of [matrices]</b>			
Matrix	Spiking Level (conc. units)	% Recoveries	Relative Standard Deviation
<b>Washington Soil</b>			
MKH 6562		88.5	11.0
MKH sulfonic acid	0.5 ppb	93.9	13.6
MKH sulfonamide		90.4	7.4
NODT		95.4	4.5
<b>Oklahoma Soil</b>			
MKH 6562		95.3	6.4
MKH sulfonic acid	0.5 ppb	78.1	12.7
MKH sulfonamide		75.9	13.7
NODT		98.5	6.3
<b>Washington Soil</b>			
MKH 6562		103.2	5.3
MKH sulfonic acid	5.0 ppb	104.5	3.0
MKH sulfonamide		106.4	3.0
NODT		89.7	2.4
<b>Oklahoma Soil</b>			
MKH 6562		104.7	3.5
MKH sulfonic acid	5.0 ppb	93.6	5.3
MKH sulfonamide		109.4	5.3
NODT		95.2	2.5

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**C.1.2. Method Characteristics**

<b>TABLE C.1.2. Method Characteristics</b>	
Analyte	MKH 6562 (Flucarbazono-sodium)
Limit of Quantitation	0.5 ppb
Limit of Detection (LOD)	MDL ( <b>Washington soil</b> ) MKH 6562 – 0.153 MKH 6562 sulfonic acid – 0.201 MKH 6562 sulfonamide – 0.150 NODT – 0.068 MDL ( <b>Oklahoma soil</b> ) MKH 6562 – 0.095 MKH 6562 sulfonic acid – 0.156 MKH 6562 sulfonamide – 0.163 NODT – 0.098
Accuracy/Precision at LOQ	LOQ – ( <b>Washington soil</b> ) MKH 6562 – 88.5% ± 9.7% MKH 6562 sulfonic acid – 93.9% ± 12.8% MKH sulfonamide – 90.4% ± 6.7% NODT – 95.4% ± 4.3% LOQ – ( <b>Oklahoma soil</b> ) MKH 6562 – 95.3% ± 6.1% MKH 6562 sulfonic acid – 78.1% ± 9.9% MKH sulfonamide – 75.9% ± 10.4% NODT – 98.5% ± 6.2%
Reliability of the Method/ [ILV]	An independent laboratory method validation [ILV], (MRID No. 469261-01), was conducted to verify the reliability of method for the determination of MKH 6562 (Flucarbazono-sodium) and its metabolites NODT, Sulfonic acid, and Sulfonamide in soil. The values obtained indicated that the registrant method is acceptable according to <i>OPPTS 850.7100 Guidelines</i> .
Linearity	The method was found to be linear over the standard range from 0.25 to 25 ppb for all analyses with the exception of NODT. The coefficients of determination ( $r^2$ ) ranged from 0.9959 to 0.9995. NODT was not linear and was quantified with quadratic $1/x^2$ . Its coefficient of determination was 0.9943.
Specificity	The analytical method employs a highly specific and selective detector; therefore, a confirmatory method is not necessary.

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**C.2. Independent Laboratory Validation (ILV)**

The ILV was conducted in accordance with the *OPPTS 850.7100 Guidelines*.

<b>TABLE C.2.1. Recovery Results Obtained by an Independent Laboratory Validation of the Method for the Determination of Flucarbazone-sodium in Soil</b>			
Compound	Spiking Level (ppb)	Average Recoveries Obtained (%)	Relative Standard Deviation (%)
MKH 6562	0.500	91	12
NODT		97	7.0
MKH 6562 Sulfonamide		100	7.6
MKH 6562 Sulfonic acid		108	13
MKH 6562	5.00	91	6.9
NODT		92	3.2
MKH 6562 Sulfonamide		89	5.7
MKH 6562 Sulfonic acid		92	4.7

**D. CONCLUSION**

From a review of this method, “Lam, C. K., Qadri, S. S. December 15, 2003, “*Analytical Method for the Determination of MKH 6562 and metabolites NODT, sulfonic acid, and sulfonamide in Soil by High-Performance Liquid Chromatography Tandem Mass Spectrometry (LC-MS/MS)*”, ECL concludes that this method is acceptable as submitted.