

Independent Laboratory Validation of Method for Tribenuron Methyl and Metabolites in Soil


Data Requirement: EPA Guideline: OCSPP 850.6100**Test Material:**

Common name: Tribenuron-methyl

Chemical name:

CAS name: methyl 2-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)methylamino]carbonyl]amino]sulfonyl]benzoate

CAS No.: 101200-48-0

Reviewer: James Lin 
Environmental Engineer
Environmental Risk Branch 2
Environmental Fate and Effects Division

Date: February 20, 2014

INDEPENDENT LABORATORY VALIDATION: MRID 49124302, Pope, C. (2013) Independent Laboratory Validation of DuPont-35446 “Analytical Method for the Determination of Tribenuron Methyl and Metabolites in Soil Using LC/MS/MS”. Project Number: DUPONT/36230, 130317, AP130317. Alliance Phama, Inc. 52p.

EXECUTIVE SUMMARY

An independent laboratory validation (ILV) (MRID 49124302) was performed by Alliance Phama, Inc. The Agency finds that the ILV does not meet the criteria for a scientifically valid method, since only one metabolite is tested and not for all potential metabolites.

COMPLIANCE

Signed and dated statements that this method and study were conducted in accordance with the requirements for Good Laboratory Practice Standards, 40 CFR 160 were present in the method. Also present was a statement of non-confidentiality on the basis of the method falling with the scope of FIFRA Section 10(d)(1)(A), (B), or (C).

A. METHODS

The environmental chemistry method (ECM) described in the report, Henze, R. and Stry, J. (2012) Analytical Method for the Determination of Tribenuron Methyl and Metabolites in Soil Using LC/MS/MS. Project Number: DUPONT/35446 (MRID 49124301), was used for independent laboratory validation (ILV) purpose.

B. RECOVERY FINDINGS

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The recovery results based on the Independent Laboratory Validation are shown in Table 1. The mean recoveries and relative standard deviations (RSD) were within the guideline requirements (mean 70-120%; RSD \leq 20%) for soil samples.

Table 1. Independent Validation Method Recoveries for Analytes in Soil

Analyte	Spike Level (ppm)	Sample Size (N)	Range of Recoveries (%)	Mean% \pm SD	RED (%)
Tribenuron Methyl	0.001	5	76 – 82	97 \pm 2.4	3
	0.01	5	76 – 86	81 \pm 4.5	6
	Overall	10	76 – 86	80 \pm 3.6	4
Tribenuron Methyl	0.001	5	73 – 84	79 \pm 4.3	5
	0.01	5	75 – 85	80 \pm 4.3	5
	Overall	10	73 – 85	79 \pm 4.1	5
IN-L5296	0.001	5	97 – 108	104 \pm 4.6	4
	0.01	5	101 – 107	103 \pm 2.7	3
	Overall	10	97 – 108	103 \pm 3.5	3
IN-L5292	0.001	5	97 – 108	105 \pm 4.4	4
	0.01	5	99 – 105	103 \pm 2.3	2
	Overall	10	97 - 108	104 \pm 3.5	3

C. CONCLUSION

This environmental chemistry method (ECM) (MRID 49124301) is designed for tribenuron methyl (DPX-L5300) and its metabolites in soil including IN-L5296, IN-A4098, IN-D5119, IN-00581, IN-GN815, IN-GK521, IN-B5528, and IN-L5296. This independent laboratory validation effort only tests on tribenuron methyl and IN-L5292. There are still seven metabolites have not been tested; therefore the ILV does not meet the requirements.

Environmental Chemistry Method for Tribenuron Methyl and Its Degradates in Soil


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CAS name: methyl 2-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)methylamino]carbonyl]amino]sulfonyl]benzoate

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ANALYTICAL METHOD: MRID 49124301, Henze, R. and Stry, J. (2012) Analytical Method for the Determination of Tribenuron Methyl and Metabolites in Soil Using LC/MS/MS. Project Number: DUPONT/35446, 172p.**EXECUTIVE SUMMARY**

This environmental chemistry method (ECM) (MRID 49124301) is designed for the quantitative determination of residues of tribenuron methyl (DPX-L5300) and its degradates in soil including IN-L5296, IN-A4098, IN-D5119, IN-00581, IN-GN815, IN-GK521, IN-B5528, and IN-L5296. The method was created by DuPont Crop Protection. in accordance with EPA's Good Laboratory Practice Standards, Title 40 Code of Federal Regulations Part 160. The Agency finds that the ECM meets the criteria for a scientifically valid method and is acceptable.

COMPLIANCE

Signed and dated statements that this method and study were conducted in accordance with the requirements for Good Laboratory Practice Standards, 40 CFR 160 were present in the method. Also present was a statement of non-confidentiality on the basis of the method falling with the scope of FIFRA Section 10(d)(1)(A), (B), or (C).

A. METHODS

Tribenuron methyl and metabolites were extracted from soil samples using a solution of 4:1 acetone/ 0.1 M aqueous ammonium carbonate. Aliquots of the extracts were removed and transferred into a clean centrifuge tubes. The aliquots were evaporated under a stream of nitrogen until the extract composition was approximately 1:1 acetone/0.1 M aqueous ammonium carbonate. The extracts were filtered through a carbon solid phase extraction cartridge. The extracts were then evaporated until only the aqueous phase remained. The volumes of the extracts were adjusted to 6-mL using HPLC grade water. Tribenuron methyl

Environmental Chemistry Method for Tribenuron Methyl and Its Degradates in Soil

and metabolites were separated from co-extracts by reversed phase liquid chromatography (LC) and were detected by turbospray mass spectrometry/mass spectrometry (MS/MS). Due to the number of analytes two LC/MS/MS runs were necessary. The compounds IN-D5119, IN-00581, IN-A4098, IN-GK521, IN-GN815, and IN-R9803 were detected using one set of chromatographic conditions. The compounds IN-B5528, IN-R9805, IN-L5296, and tribenuron methyl were detected using alternative chromatographic conditions.

B. RECOVERY FINDINGS

The results of percent recovery and relative standard deviations (RSD) are shown in Table 1. The recovery data from these samples demonstrated that residues of tribenuron methyl and metabolites were stable during extraction, clean-up and analysis.

Table 1. Initial Validation Method Recoveries for Analytes in Two Soils

Percent Recovery (%RSD)					
Analyte	Speyer Soil (LOQ)	Speyer Soil (10X LOQ)	Lleida Soil (LOQ)	Lleida Soil (10X LOQ)	N
IN-A4098	99% (14)	98% (5.5)	104% (11)	94% (9.1)	5
IN-D5119	94% (12)	103% (7.9)	92% (15)	108% (6.6)	5
IN-00581	106% (13)	100% (8.9)	93% (4.3)	103% (6.3)	5
IN-GN815	92% (2.7)	101% (4.6)	93% (3.6)	96% (6.1)	5
IN-GK521	88% (3.2)	93% (4.4)	85% (4.5)	83% (13)	5
IN-B5528	92% (5.0)	87% (2.9)	92% (13)	90% (6.5)	5
IN-R9805	106% (4.4)	92% (9.2)	97% (11)	94% (7.1)	5
IN-L5296	105% (2.1)	95% (4.5)	100% (8.7)	94% (7.1)	5
Tribenuron methyl	87% (3.6)	90% (6.5)	91% (16)	72% (11)	5

C. CONCLUSION

The mean recoveries and relative standard deviations (RSD) were within the guideline requirements (mean 70-120%; RSD ≤20%) for soil samples fortified at 1X and 10X the LOQ. The LOQ (Limit of Quantitation) of 1.0 µg/kg (ppb) was defined as the lowest concentration tested at which acceptable recoveries were obtained with an RSD of ≤20%. The LOD (Limit of Detection) in soil was estimated to be 0.3 µg/kg (ppb).