

Environmental Chemistry Method for Detection of Triallate in Soil

Reports: Analytical Residue Method for Triallate and the Sodium Salt of 2,3,3-Trichloroprop-2-ene Sulfonic Acid in Soil

Document No.: [MRIDs 40117901]

Guideline: 850.6100 [U.S.], 8.2.2.3 [Soil];

Statements: The study did not claim in compliance with any Good Laboratory Practice (GLP) Regulations.

Classification: The ECM is classified as Invalid for monitoring triallate and TCPSA in soil.

Deficiencies: The ECM fails to report the following parameters: 1) calibration curve with r^2 ; 2) limit of detection (LOD), and limit of quantification (LOQ); 3) % soil matrix recovery; 4) the linearity, repeatability and reproducibility of the method; 5) examples using this method to perform the soil sample analysis for triallate and TCPSA; and 6) lack of corresponding Independent Laboratory Validation (ILV) Study.

PC Code: 078802

Reviewer: He Zhong, Ph.D.
Biologist, EFED, EPA

Signature: 
Date: 08/19/2014

Executive Summary

This environmental chemistry method (ECM), MRID 40117901, has reported an analytical procedure via GC-ECD for analysis of triallate [S-(2,3,3-trichloroallyl) diisopropyl-thiocarbamate] and 2,3,3-trichloroprop-2-ene sulfonic acid (TCPSA) in soil with 0.01 ppm as the limit of validation (LOV) for each compound. However, it is uncertain how the LOV value was obtained and the relationship to the limit of detection (LOD) and limit of the quantification (LOQ), which were not reported (Table 1). In addition, there were no quality control and quality assurance (QA/QC) parameters (calibration curve, r^2 and % matrix spike recovery, etc.) associated with this ECM report.

Table 1. Triallate and TCPSA Analytical Method Summary

Analyte(s) by Pesticide	MRID	Matrix	Method Date (m/d/y)	Registrant	Instrument	Limit of Detection ($\mu\text{g}/\text{Kg}$)	Limit of Quantitation ($\mu\text{g}/\text{Kg}$)
Environmental Chemistry Method	40117901	Soil	N/A	N/A	GC-ECD	N/A	N/A

I. Principle of the Method

Soil samples with triallate and/or TCSPA are extracted with acetonitrile/water using a mechanical shaker and filtered through a Buchner funnel. An aliquot of the extract is diluted with water and partitioned with isoctane to separate triallate. The isoctane fraction is cleaned up, concentrated and quantitated by a GC-ECD. The method also documented the preparation of 8 levels triallate standards ranging from 2.5 $\mu\text{g}/\text{L}$ to 35 $\mu\text{g}/\text{L}$ in isoctane solution. The aqueous fraction, containing any TCPSA is partitioned with methylene chloride following the cleanup. The methylene chloride fraction is concentrated and passed through a cation exchange column and derivatized with triethylorthoformate in a 100 °C oil bath for 1 hr, and analyzed using GC-ECD.

II. Recovery Findings

The mean % recoveries and relative standard deviations (RSD) of triallate and TCPSA were not reported. The method only reported the purity data of neat compound for triallate and TCPSA using NMR and GC/MS method (Appendix G).

III. Method Characteristics

The method reported the apparatus and equipment for the triallate and TCPSA residue analysis. It also reported the GC instrument conditions, standard preparations, triallate and TCPSA analytical procedures such as, florisil cleanup, quantification, partition, column chromatography, derivatization and quantification and standard purity determination with few example chromatograms. However, the method did not report the results such as calibration curve with r^2 , LOD, and LOQ. The linearity, repeatability and reproducibility were not established.

IV. Method Deficiencies and Reviewer's Comments

No QA/QC information were found in this ECM, such as the calibration curve (r^2), LOD, LOQ, and the % matrix spike recovery for soil samples. No results for soil sample analysis using this method were reported. The ECM cannot be validated by the EPA reviewer without this QA/QC

data for the ECM as well as a corresponding Independent Laboratory Validation (ILV) Study containing similar ILV QA/QC data.

V. References

Allan, J.M. (Year -N/A). Analytical Residue Method for Triallate and the Sodium Salt of 2,3,3-trichloroprop-2-ene Sulfonic Acid in Soil. MRID 40117901