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DATA EVALUATION RECORD

STUDY IDENTIFICATION:

Bade, T. R. 1992. Stability of SAN-582 H and Its Metabolites in Stored Frozen Soil Samples QAU #89/11/27. Performed by Analytical Sciences-Residue Chemistry of Sandoz Crop Protection Corporation, Des Plaines, Illinois. MRID No. 422662-06.

TYPE OF STUDY: Soil Storage Stability

REVIEWED BY:

George Tompkins, Entomologist
Review Section 1, EFGWB, EFED

Signature: *George Tompkins*
Date: NOV 24 1992

APPROVED BY:

Paul J. Mastradone, Section Chief
Review Section 1, EFGWB, EFED

Signature: *Paul J. Mastradone*
Date:

CONCLUSIONS:

1. This study provides useful information on the storage stability of both SAN 582 H and oxalamide to support the field dissipation studies (MRID Nos. 422662-02, 422662-03, 422662-04, and 422662-05). The information provided indicates that fortified soil samples of both SAN 582 H and oxalamide were stable when stored frozen in the soil for up to 29 months (870 days). After 29 months of storage the recovery of parent SAN 582 H ranged from 78-89.5%, and oxalamide recoveries ranged from 97.5-105% in soils from California, Wisconsin, and Minnesota.

MATERIALS AND METHODS:

The following analytical standards were used to measure residue levels in the fortified soil samples (See Figure 1 for structures):

- 1) SAN-582 H; 2-chloro-N-((1-methyl-2-methoxy)ethyl)-N-(2,4-dimethyl-thienyl)-acetamide, Lot #RS-582-10688 with a purity of 99.1%, Lot #RS-SAN-110389 with a purity of 99.6%,
- 2) Oxalamide metabolite of SAN-582 H; N-((1-methyl-2-methoxy)ethyl)-N-(2,4-dimethyl-thienyl)-oxalamide, Lot #RS-OXA-020289 with a purity of 94.8%. Lot #RS-5820XA-101689-2 with a purity of 99.85%,
- 3) Methyl Ester of the Oxalamide metabolite of SAN-582 H; N-((1-methyl-2-methoxy)ethyl)-N(2,4-dimethyl-thienyl)-oxalamide, methyl ester; Lot #RS-5820ME-061589 with a purity of 97.5%, Lot #AR-28306 with a purity of 94.7%.

The following analytical standards were used in the aerobic soil metabolism study:

SAN 582 H; Lot #RS-582-10688, with a purity of 99.1%
¹⁴C SAN-582 H (14-C labeled in the thienyl ring); Lot # RA-683-1, specific activity = 43.3mCi/mmole, radiochemical purity = 99.3%.

The soil used in the metabolism study was a Kenyon loam soil which was characterized as having 34% sand, 41% silt, 25% clay, 3.8% organic matter, pH of 6.2, CEC of 20.4 meq/100 gm. This soil was sieved through a 2 mm sieve, homogenized and brought to 75% of its moisture capacity at 1/3 bar. A mixture of the standard SAN-582 H and the radioactively labeled analytical standard was thoroughly homogenized with 5000 g of field moist soil to give a final concentration of 2.29 ppm.

In the fortified soil stability study three soils were used and consisted of: 1) a silt loam soil from Wantonwan County, Minnesota, which was 38% sand, 50% silt, 12% clay, and 7% organic matter; 2) a sandy loam soil from Hugson, California, which was 78% sand, 14% silt, 8% clay, and 0.4% organic matter; 3) a clay loam soil from Sheboygan, Wisconsin, which was 67% sand, 33% clay, and 8% organic matter.

These soils were all sieved through a 2 mm sieve, homogenized and stored in a field moist condition. Aliquots of the three soils were weighed into 4-oz glass bottles. Half of these aliquots were fortified at a 0.2 ppm level, with both SAN-582 H and Oxalamide, and half were left unfortified to use for control analyses and laboratory fortification analyses performed at the time of each stability interval. After fortification (13 June 1989) all of the jars were placed in a cardboard box and placed in a freezer for their entire storage period. For day 0 analyses, two samples from each soil type that had been fortified and one unfortified sample from each soil type were analyzed for SAN-582 H and oxalamide. On subsequent stability intervals (1, 3, 6, 12, 18, and 29 months) two fortified samples from each soil type and two unfortified samples from each soil type were removed from the freezer. One unfortified sample from each soil type was retained as a control sample and the other was used as the laboratory fortified control sample. The stability analyses performed on the 0, 1, 3, and 18 month storage samples utilized the procedure described in residue method AM-0830-0290-2 (Appendix III, procedure enclosed) in which after extraction the residues were dissolved in toluene and analyzed by gas chromatography (GC) using a mass selective detector. The stability analyses performed on the 29 month samples utilized the residue method AM-0865-0791-0 (Appendix III) in which after extraction the residues are dissolved in toluene for GC analysis using a Nitrogen Phosphorous Detector (NPD) or a Mass Selective Detector (MSD).

REPORTED RESULTS:

1. The limit of detection for measurement of SAN 582 H and its oxalamide metabolite with method AM-0830-0290-2 and method AM-0865-0791-0 was 0.01 ppm.
2. The average normalized recovery of SAN 582 H from three different soils ranged from 78% (Wisconsin soil) to 89.5% (California soil) after 29 months frozen storage. The average normalized stability recovery for oxalamide from the same three soils ranged from 97.5% (Wisconsin soil) to 105% (California soil) after 29 months in frozen storage.
3. The recoveries using method AM-0830-0290-2 for SAN 582 H from laboratory fortified and stability fortified samples averaged $97.7 \pm 12.9\%$ and $93.8 \pm 15.2\%$, respectively. Recoveries for oxalamide from laboratory and stability fortified samples averaged $87.9 \pm 20.8\%$ and $86.3 \pm 19.4\%$, respectively (Table I).
4. Recoveries using method AM-0865-0791-0, for SAN 582 H from laboratory fortified and stability fortified samples averaged $94.7 \pm 18.0\%$. The recoveries for oxalamide from laboratory and stability fortified samples averaged $75.0 \pm 20.0\%$ and $76.8 \pm 17.9\%$, respectively (Table 1).

DISCUSSION:

In using method AM-0830-0290-2 the difference between the average laboratory recovery and the average stability recovery was less than 4% for both SAN 582 H and oxalamide. This method appeared to have much less variability in the recoveries in the laboratory fortified and stability fortified samples than did method AM-0865-0791-0, in which the average recoveries ranged by as much as 15% for SAN 582 H.

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