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DATA EVALUATION REVIEW

I. Study Type: Laboratory Volatility

II. Citation: Kesterson, B.A., Steve Johnson, and Lowell J. Lawrence. 1990. Laboratory Volatility of [¹⁴C] 2,4-D 2 Butoxyethyl Ester. performed by Pharmacology and Toxicity Research East (PTRL), Richmond, Kentucky and submitted by DowElanco, Midland, Michigan. MRID# 417180-01.

III. Reviewer:

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V. Conclusions:

This study provides unacceptable data on the volatility of 2,4-dichlorophenoxyacetic acid-2-butoxyethyl ester (2,4-D BEE) from soil. The study cannot fulfill the soil volatility (163-2) data requirement for the following reason:

- The volatile residues were not identified by a confirmatory analytical technique; total ¹⁴C analysis does not provide identification of 2,4-D BEE. EFGWB prefers that [¹⁴C]-residues be separated by chromatographic methods (TLC, HPLC, or GC) with solvent systems of different polarity, and that specific compounds isolated by chromatography be identified using a confirmatory method such as MS in addition to comparison with Rf or HPLC retention times of referenced standards.

Based on unacceptable data, the rate of 2,4-BEE volatilization was 2.4 to 1.3 X 10⁻⁴ μg cm⁻² hr⁻¹. (Please note the volatilization rates are expressed as 2,4-D equivalents.) The volatile [¹⁴C]-residues were distributed in the polyurethane foam plug trap (0.4% of applied) and NaOH solution trap (0.1% of applied). The soil [¹⁴C]-residues were extracted in methanol (≈90% of applied) and acidified methanol (≈4.5% of applied). The major soil extractable degradate was 2,4-D.

EFGWB believes the reported volatility rate for parent 2,4-D BEE may be incorrect. Because 2,4-D BEE rapidly hydrolyzes in alkaline environments (MRID# 414831-01) such as neutral to alkaline soils, the volatility rate of [¹⁴C]-residues may represent the major hydrolytic degradate, 2,4-D, rather than 2,4-D BEE. This hypothesis cannot be confirmed because the volatile

[¹⁴C]-residues were not identified using a confirmatory analytical technique. In the future, the soil volatility studies should be conducted under soil conditions which minimize 2,4-D BEE degradation.

VI. Materials and Methods:

Sand soil (pH 7.9, 0.62 % O.M., CEC 3 meq 100⁻¹, FC (_{1/3} bar) 8.7%) was sieved through a 2mm sieve and then autoclaved for 1 hour. Subsamples of sterile soil (50 gm) were each amended with radiolabeled 2,4-D BEE (SA 6.5 mCi mmole⁻¹, solubility 10 µg ml⁻¹, uniformly labeled ring, radiopurity 94.6% purified by TLC to 98,8%) to produce a nominal soil concentration of 7.8 µg ml⁻¹.

The soil samples were adjusted to field capacity and then placed into an incubation system. The incubation system consisted of an erlenmeyer flask, which was connected to polyurethane foam plug, ethylene glycol, and 10% NaOH gas traps. The volatile residues were collected by purging the incubation system with humidified air (144.8 ± 15 ml minute⁻¹.) into the gas traps. The experiment was conducted at a temperature of 24.9±0.5°C.

Solution trap samples were taken 5, 9, 16, 23, and 30 days post-treatment. Additionally, after the 30 day sampling, soil samples were taken for chemical analyses.

Analytical

The soil samples were sequentially extracted with methanol:water :1N HCl (94:5:1, v:v:v) and methanol:water (2:1, v:v). These extracts were pooled for chemical analyses.

Residues in soil extracts and the solution traps were separated by HPLC using a Supelc LC-18 ion pair reverse phase column with a linear gradient solvent system (acetonitrile/dihydrogen potassium buffer-adjusted at pH 3); and a UV detector set at 254 nm and Radiomatic Flo-One Beta detector. The separated residues were identified by co-chromatography with 2,4-D and 2,4-D BEE. In addition, the total ¹⁴C content of the extracted soil was determined by combustion.

The volatility rate of 2,4-D BEE, expressed as 2,4-D equivalents, was based ¹⁴C accumulation rates on the polyurethane foam plug.

VII. Study Author's Results and/or Conclusions

1. The rate of 2,4-D BEE volatilization was estimated to be 2.4 to 1.3 X 10⁻⁴ µg cm⁻² hr⁻¹ (Figure V). (Please note the volatilization rates are expressed as 2,4-D equivalents.)
2. A mass balance of [¹⁴C]-residues accounted for >98% of the applied 2,4-D BEE. The volatile [¹⁴C]-residues were distributed in the polyurethane foam plug trap (0.4% of applied) and NaOH solution trap (0.1% of applied); and the soil residues were

distributed in methanol extractable ($\approx 90\%$ of applied) and acidified methanol ($\approx 4.5\%$ of applied) fractions.

3. The extractable soil residues of 2,4-D BEE were identified as 2,4-D (87.6% extractable), 2,4-D BEE (6.3% extractable), and unidentified degradates (1.7% extractable). Additionally, the major volatile degradate was tentatively identified as CO_2 (% 0.1 applied).

VIII. Reviewer Comments

1. The volatile residues were not identified by a confirmatory analytical technique; total ^{14}C analysis does not provide identification of 2,4-D BEE. EFGWB prefers that [^{14}C]-residues be separated by chromatographic methods (TLC, HPLC, or GC) with solvent systems with different polarity, and that specific compounds isolated by chromatography be identified using a confirmatory method such as MS in addition to comparison with Rf or HPLC retention times of referenced standards. In future studies, the residues should be identified at least two analytical methods.

2. EFGWB believes the reported volatility rate for parent 2,4-D BEE may be incorrect. Because 2,4-D BEE rapidly hydrolyzed in alkaline environments (MRID# 414831-01) such as neutral to alkaline soils, the estimated volatility rate of [^{14}C]-residues may represent the major hydrolytic degradate, 2,4-D, rather than 2,4-D BEE. This hypothesis cannot be confirmed because the volatile [^{14}C]-residues were not identified using a confirmatory analytical technique. In the future, the soil volatility studies should be conducted under soil conditions which minimize 2,4-D BEE degradation.

3. The [^{14}C]-residues in the NaOH gas trap was tentatively identified as CO_2 . In future studies, the [^{14}C]- CO_2 content should be identified by an appropriate chemical procedure.

2,4-D EFED Review

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