UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

MAY 8 1996

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OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM:

SUBJECT: **Oxyfluorfen.** Residue Trial Studies for Horseradish: GLN 171-4(k). Case No. 2490. Chemical No. 111601. MRID No. 43973701. DP Barcode: D225110. CBRS No. 17121.

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THRU: Ed Zager, Chief
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TO: Paula Deschamp, Section Chief
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BACKGROUND

IR-4 has submitted residue trial data in support of the use of oxyfluorfen on horseradish. These residue trials were proposed by the registrant and found acceptable by CBRS (memo dated 12/21/93, S. Funk, CBRS No. 12933). The registrant proposed to conduct field trials on horseradish in IL/WI (1) and MD/NJ (1). These data are reviewed here.

Tolerances are established for residues of the herbicide oxyfluorfen [2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-trifluoromethyl)benzene] and its metabolites containing the diphenyl ether linkage in or on various commodities including artichokes, avocados, bananas, broccoli, cabbage, cauliflower, cocoa beans, coffee, corn grain, cottonseed, dates, feijoa, figs, grapes, kiwifruit, olives, onions, persimmons, pistachios, pome fruits group, pomegranates, soybeans, stone fruits group, and tree nuts at 0.05 ppm (40 CFR 180.381(a)). Tolerances with regional

(1)

registration are established for residues of oxyfluorfen and its metabolites containing the diphenyl ether linkage in the raw agricultural commodities: guava, papaya, and taro (corms and leaves) at 0.05 ppm (40 CFR 180.381(b)).

Food additive tolerances are established for residues of oxyfluorfen and its metabolites containing the diphenyl ether linkage in the processed commodities: cottonseed oil, mint oil (peppermint and spearmint) and soybean oil (40 CFR 180.4600).

Adequate methodology is available for the enforcement of tolerances for oxyfluorfen residues in or on plant and animal commodities. Two GLC/electron capture detector (ECD) methods are listed in Pesticide Analytical Manual (PAM) Vol. II as Methods I and II for the determination of oxyfluorfen residues in or on soybean grain, milk, and the fat, meat, and meat byproducts of cattle (Pesticide Registration Section 180.381). Using Method I, recovery of combined residues of oxyfluorfen and its metabolites containing a diphenyl ether linkage from soybean grain samples was 55-63%. Recoveries of oxyfluorfen residues from milk and meat using Method II were 55-75% and 56-70%, respectively. Similar GLC/ECD techniques have been used for collection of data concerning residues in or on almond hulls and nuts, oranges, soybean hay, and wheat. The Pestrak data base dated 12/13/89 indicates that oxyfluorfen is completely recovered (>80%) using PAM Vol. I Multiresidue Protocols D and E (for non-fatty foods); recovery of oxyfluorfen metabolites containing a diphenyl ether linkage using Multiresidue Protocols A, D, and E is unlikely.

The nature of the residue in plants is adequately understood. The residue to be regulated is oxyfluorfen, per se, (S. Knizner, 4/8/94, CBRS No. 12513, 12522, 13212, and 13228).

CONCLUSIONS

1. Horseradish root samples treated at a 1X application rate (0.5 lbs. a.i./A) with GOAL 1.6E containing the active ingredient (a.i.) oxyfluorfen had nondetectable residues (<0.01 ppm). The existing tolerance for oxyfluorfen residues on horseradish is 0.05 ppm. These residue data support the existing tolerance.

2. Storage stability data are required to support this study.

3. The analytical method used to collect data for this study is adequate. An adequate enforcement method and accompanying independent laboratory validation (ILV) for the determination of residues of oxyfluorfen, per se, in plants is needed.

RECOMMENDATIONS

The registrant should be advised that the submitted horseradish field trial study is adequate and satisfies GLN 171-4(k) for horseradish. The submitted residue data support the existing

tolerance. The registrant should submit the required storage stability data in support of this study and these conclusions and recommendations. The registrant should be advised to propose an enforcement method for the determination of residues of oxyfluorfen, per se, in plants. An accompanying ILV for the proposed enforcement method should also be submitted.

DETAILED CONSIDERATIONS

Directions for Use

REFS lists two products containing the active ingredient oxyfluorfen registered for use on horseradish: GOAL 1.6E and GOAL 2XL. The existing label for the 1.6E product allows a seasonal maximum application of 0.5 lbs. a.i./A as a single broadcast spray to horseradish roots after they have been planted, but before the plants emerge.

Test System and Materials

Field trials were conducted in MD(1) and WI(1) consisting of one treated plot and one untreated, control plot. GOAL 1.6E containing the active ingredient oxyfluorfen was applied in a single broadcast application to horseradish after the roots had been planted, but prior to emergence of the plants. The roots were harvested at normal maturity at 141 to 144 days after the last application of GOAL 1.6E on 9/26/94 (MD) and 10/11/94 (WI). Two samples of the root were collected from the treated and control plots for analysis. Collected samples were stored frozen for up to 4.5 months before delivery to the laboratory for analysis. Samples were extracted and analyzed on 8/4 - 5/95. The samples were stored for a total of 10 months prior to extraction and analysis.

Analytical Method

The analytical method used in this study for data collection was modified from Technical Report No. 23-73-5, "A Residue Analytical Method for RH-915 (Parent Compound: Oxyfluorfen). In brief, ten g of horseradish sample is initially extracted with acetonitrile:water, followed by a liquid/liquid partition of this initial extract with water and petroleum ether. The aqueous layer is discarded and the acetonitrile fraction is evaporated to dryness. The remaining residue is dissolved in petroleum ether, passed through a Florisil clean-up column, and rinsed from the column with petroleum ether:ethyl acetate. The collected ether eluate is evaporated to dryness and the residue is taken-up in hexane. The final extract is analyzed to determine oxyfluorfen by a gas chromatograph equipped with dual electron capture detectors. One detector was equipped with the primary chromatographic column and the second detector is equipped with a different column for confirmatory analysis. Adequate sample chromatograms and calculations were provided. The limit of quantitation (LOQ) was 0.01 ppm. The method is adequate for data collection.

The data collection method was validated using control samples fortified with oxyfluorfen at

0.01 ppm level were 90% and 100%. Recoveries on the two samples fortified at the 0.10 ppm level were 81% and 80%.

The existing single residue plant analytical method in PAM Vol. II is for the determination of oxyfluorfen and metabolites containing the diphenyl ether linkage. An enforcement method and accompanying ILV for the determination of oxyfluorfen, per se, in plants for inclusion into PAM Vol. II is needed.

Residue Trial Results

Two samples from each plot were analyzed for oxyfluorfen residues along with two untreated control samples. All treated and untreated, control samples had nondetectable residues of oxyfluorfen (<0.01 ppm).

Storage Stability

No storage stability data were submitted with these residue field trial data. The registrant referenced a storage stability study (MRID No. 43859801). These data have been submitted, but have not been received for review at this time. The maximum period of frozen storage for samples analyzed in this study was 10 months.

cc: Mark Wilhite, Chemical Review Manager
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