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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 Onions: GLN 171-4(k). Case No. 2490. Chemical No. 111601. MRID No. 43965501. DP Barcode: D224847. CBRS No. 17106.

FROM: Catherine Eiden, Chemist *Catherine Eiden*
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THRU: Ed Zager, Chief *Edward Zager*
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TO: Paula Deschamp, Section Chief
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BACKGROUND

The Phase IV review of oxyfluorfen (S. Funk, 3/16/91) considered previously submitted field trials for onions and found them to be adequate provided that storage stability data to support the trials reflecting the maximum temperature and period of storage were submitted. Storage stability data have been submitted and are under review. Amendments to the labels were required to clarify when to apply oxyfluorfen to onions. These amendments have been made (F. Fort, 4/16/92, CBRS No. 9024). In a memo regarding Replacement of Craven Laboratory Residue Chemistry Data (S. Funk, 10/10/91, CBRS No. 8548) the registrant for oxyfluorfen was directed to replace residue trial data for onions. The registrant responded and agreed to conduct 7 new onion trials in CA(1), TX(1), OR/WA (1), CO(1), ID(1), MI(1), and NY(1). CBRS found the registrant's proposal acceptable (S. Funk, 12/21/93, CBRS No. 12933). The residue data submitted and reviewed here are the Craven replacement data for onions.

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In response to the need and the requirement to replace Craven data for oxyfluorfen residues on onions, and support the reregistration of oxyfluorfen, IR-4 has submitted crop field trial data for oxyfluorfen residues on onions (MRID No. 43965501). 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 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. Onion 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 onions 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 onion field trial studies are adequate and satisfy GLN 171-4(k) for onions. 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: GOAL 1.6E (EPA Reg. No. 707-174) and GOAL 2XL (EPA Reg. No. 707-243). A copy of the existing GOAL 1.6E label was included in the report. GOAL 1.6E is an herbicide recommended for post-emergence application to onions that have at least 2 true leaves. Single applications at 0.12 to 0.25 lbs. a.i./A are recommended, and the seasonal maximum application rate is 0.5 lbs. a.i./A. The established pre-harvest interval (PHI) is 45 days. There are no feeding/grazing restrictions associated with bulb onions on the label. There are no feedstuffs listed for onions in Table II: September 1995.

Test System and Material

Seven field trials were conducted in CA(1), CO(1), ID(1), MI(1), NY(1), OR(1), and TX(1). Each trial had one treated and one control (untreated) plot. Two post-emergence broadcast spray applications were made to each of the plots when the planted onions had 2 to 3 true leaves. The second and last application was applied 45 to 48 days before harvest, except in the NY trial, where onions were harvested 54 days after the last application. Two samples of onions were collected from each trial and shipped frozen and stored at -18°C until extracted and analyzed.

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 onion 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 residuum 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 rinse is evaporated to dryness and the residuum is taken-up in hexane. The final extract is analyzed to determine oxyfluorfen by gas chromatograph equipped with an dual electron capture detector. One detector is equipped with a primary chromatographic column and the second detector was 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 to 0.10 ppm in duplicate. These fortified samples were analyzed concurrently with the treated field samples. Table 1 below provides the percentage recoveries from these fortified control samples.

Table 1. Recoveries of Oxyfluorfen Residues from Fortified Control Onion Samples.

Fortification Level (ppm)	Percent Recovery (%)	Fortification Level (ppm)	Percent Recovery (%)
TX, MI, OR, CA		ID, CO, NY	
0.01	90	0.01	130
0.01	110	0.01	110
0.01	130	0.01	120
0.01	110	0.01	110
Average (\pm SD)	110 \pm 16.3	Average \pm SD	117.5 \pm 9.6
0.10	71	0.10	92
0.10	88	0.10	101
0.10	92	0.10	99
0.10	101	0.10	96
Average (\pm SD)	88 \pm 12.5	Average \pm SD	97 \pm 3.9

The existing single residue method in PAM Vol. II is for the determination of oxyfluorfen and metabolites containing the diphenyl ether linkage in plants. 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 and are under review. The maximum period of frozen storage for samples analyzed in this study was 11 months.

cc: Mark Wilhite, Chemical Review Manager
Accelerated Reregistration Branch
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RF, SF, List B File, C. Eiden, Circ.

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