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

MAY 8 1996

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM:

SUBJECT: Oxyfluorfen. Residue Trial & Processing Study for Corn: GLN 171-4(k & l).
Case No. 2490. Chemical No. 111601. MRID No. 43944801. DP Barcode:
D224356. CBRS No. 17049.

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

The Phase IV review on oxyfluorfen (S. Funk, 3/16/91) required that a processing study be conducted for corn bearing weathered residues of oxyfluorfen processed into starch, crude and refined oil from wet milling, and grain dust, grits, meal, flour, and crude and refined oil from dry milling processes. The registrant was advised that if residue trial samples from plots treated at exaggerated rates equal to the maximum theoretical concentration factor resulting from processing had no detectable residues on the rac, a processing study would not be required.

Previously submitted field trials for corn were found to be adequate provided that the pre-harvest interval (PHI) for corn was amended to 60 days on the label. Storage stability data for corn field trials reflecting 24 months of storage at -10°C were required. The label has been changed to reflect a 60-day PHI. Storage stability data are being developed. Finally, the

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Phase IV review determined that the registrant must propose fodder and forage tolerances for corn treated with oxyfluorfen. CBRS has since concluded that corn fodder and forage tolerances are not warranted, in this case, because oxyfluorfen is used in NC and SC only to eradicate "witchweed" (*Striga asiatica*) and the forage and fodder are not fed to livestock to avoid the spread of the weed (F. Fort, 4/16/92, CBRS No. 9024, DP Barcode No. D171996).

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 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 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 meal 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. Corn samples treated at a 1X and a 25X application rate with GOAL 1.6E containing the active ingredient (a.i.) oxyfluorfen had nondetectable residues (<0.01 ppm). The existing tolerance for oxyfluorfen residues on corn is 0.05 ppm. These residue data support the

existing tolerance.

2. Because corn rac samples treated at the 25X rate had nondetectable residues of oxyfluorfen even after treatment at an exaggerated rate equivalent to the maximum theoretical concentration factor expected from processing, corn was not processed into meal, grain, starch, grits, flour, grain dust, and oils. No 409 tolerance or Section 701 MRL is warranted.
3. Storage stability data are required to support this study.
4. 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.
5. The label for the GOAL 2XL product allows a 30-day pre-harvest interval (PHI). Available field trial data support a 60-day PHI (Phase VI review, S. Funk, 3/16/91).

RECOMMENDATIONS

The registrant should be advised that the submitted corn field trial/processing study is adequate and satisfies GLN 171-4(1) for corn. The submitted data support the existing tolerance. The registrant should submit the required storage stability data in support of this study and these conclusion 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.

The registrant should be advised to amend the GOAL 2XL product label to reflect a 60-day PHI for use on corn. Otherwise, field trials to support the 30-day PHI will be required.

DETAILED CONSIDERATIONS

Directions for Use

As of 4/22/96, REFS lists one product registered for use on field corn: GOAL 1.6E containing the active ingredient (a.i.) oxyfluorfen @ 1.6 lbs. of the a.i. (EPA Reg. No. 707-174). The use is limited to use in NC and SC for the eradication of witchweed in conjunction with the USDA program there. The seasonal maximum use rate is 1.25 lbs. a.i./A, to be applied as a first application at a rate of 0.5 to 0.75 lbs. a.i./A when the corn has achieved 24 inches in height, and as a second application at a rate of 0.25 to 0.5 lbs. a.i./A, if necessary. The PHI is 60 days for the GOAL 1.6E product. There is a feeding restriction on fodder and forage to prevent the spread of witchweed.

SRRD also lists the GOAL 2XL product as registered for use on corn. The GOAL 2XL label allows the same use pattern, seasonal maximum rate, and feeding restrictions as GOAL 1.6E, and a 30-day PHI for use on corn.

Test System and Materials

For this study, one field site was selected in NC(1). Three test plots were established at the site: one control plot, and two treated plots. The first treated plot received a total application of 1.25 lbs. a.i./A of GOAL 1.6E in two applications (the first @ 0.75 lbs. a.i./A once the corn has achieved a minimum height of 24 inches, 75 days prior to harvest, and the second @ 0.50 lbs. a.i./A 15 days later, 60 days prior to harvest). Treatment at this plot was equivalent to a 1X rate. The second treated plot received a total application of 31.25 lbs. a.i./A of GOAL 1.6E in two applications (the first @ 18.73 lbs. a.i./A once the corn has achieved a minimum height of 24 inches, 75 days prior to harvest, and the second @ 12.51 lbs. a.i./A 15 days later, 60 days prior to harvest). Treatment at this second treated plot was equivalent to a 25X rate. This is the maximum theoretical concentration factor for corn rac residues processed into corn oil (Pesticide Reregistration Rejection Rate Analysis Residue Chemistry: Maximum Theoretical Concentration Factors: February 1993).

Samples were harvested 60 days after the last application in bulk 10 pound samples and stored and shipped frozen to the laboratory for analysis. Samples were stored for approximately 7 months (209 days) @ -5°C to -25°C prior to analysis for oxyfluorfen.

Analytical Method

In brief, the analytical method used to analyze oxyfluorfen residues uses acetonitrile to extract residues initially. To further purify the extract with liquid-liquid partition (acetonitrile:petroleum ether and petroleum ether:acetonitrile water), silica gel chromatography and basic alumina solid phase extraction before quantitation using gas chromatography with electron-capture detection. Two columns were used for quantitation and confirmation of the analytical results. The limit of quantitation (LOQ) for the method is 0.01 ppm.

The analytical method used for analysis of the study samples was validated on representative nontreated grape and soybean samples prior to the analysis of the study samples. The method validation included analyses of nonfortified control samples and duplicate analyses of control samples fortified with oxyfluorfen at levels of 0.01 ppm, 0.10 ppm, and 0.50 ppm. Recoveries of oxyfluorfen from the fortified soybean samples ranged from 86.3% to 121% with an average recovery of $95.3\% \pm 22.0\%$ (n = 12). The soybean recoveries using a confirmatory gas chromatographic column ranged from 90.8% to 124%. Recoveries from fortified grape samples ranged from 77% to 109% with a mean recovery of $90.8\% \pm 10.5\%$ (n = 17). The grape recoveries using a confirmatory column ranged from 75.3% to 110%.

Control corn rac samples were fortified with oxyfluorfen ranging from 0.01 to 0.05 ppm and

analyzed concurrently with the treated samples. The concurrent recoveries for all fortified samples ranged from 88% to 99.5% with a mean recovery and standard deviation of 92.6% \pm 5.5% (n =4).

Table 1. contains all of the fortification and recovery data for soybeans, grapes and corn used to validate the analytical method.

Table 1. Recoveries of Oxyfluorfen from Fortified Racs			
Commodity	Chromatographic Column	Fortification Levels (ppm)	Recovery Range (%)
Soybeans	Rtx-200	0.01 - 0.50	86.3 - 121
Soybeans	Rtx-50	0.01 - 0.50	50 - 124
Grapes	Rtx-200	0.01 - 0.50	78.1 - 109
Grapes	Rtx-50	0.01 - 0.50	75.3 - 110
Corn	Rtx-200	0.01 - 0.02	88.0 - 99.5

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, for inclusion into PAM Vol. II is needed.

Residue Results

Three corn samples, one from each test plot, i.e., control, 1X-treatment, and 25X-treatment, were analyzed for oxyfluorfen residues. Duplicate samples were not analyzed. All samples had nondetectable residues of oxyfluorfen (<0.01 ppm).

Storage Stability

Samples analyzed for this study were stored for a maximum of 209 days at -5 to -25°C prior to extraction and analysis. Storage stability information were not provided to support these data. Storage stability data are being developed by the registrant.

Processing Study

Oxyfluorfen residues were nondetectable on corn rac samples collected and analyzed after

treatment at a 1X and a 25X rate with GOAL 1.6E. Because oxyfluorfen residues were nondetectable on corn rac samples after treatment at an exaggerated rate equivalent to the maximum theoretical concentration factor (25X) expected from processing, the rac samples were not processed into meal, flour, oils, grain, starch, and grits, and the processing study requirement for corn is waived.

cc: Mark Wilhite, Chemical Review Manager
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