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

JUL 2 1996

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM:

SUBJECT: Oxyfluorfen. Storage Stability on Various Crops: GLN 171-4(e). Case No. 2490. Chemical No. 111601. MRID Nos. 43859801. DP Barcode: D221585. CBRS No. 17259.

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TO: Paula Deschamp, Section Head
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BACKGROUND

Rohm and Haas Company has submitted storage stability data on various crops in support of the reregistration of oxyfluorfen. These data were required variously in Phase IV and Phase V reviews of oxyfluorfen under reregistration activities. These data are reviewed here.

CONCLUSIONS

1. The submitted storage stability study support the submitted residue trial data for: almonds (hulls and nutmeats), artichokes, avocado, banana, broccoli, cabbage, cauliflower, corn, cottonseed, figs and dates, grapes, guava, horseradish, kiwi, mint hay, olives, onions, persimmons, pistachios, pome fruits, pomegranates, soybeans, stone fruits, and tree nuts.

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2. Residues of oxyfluorfen, per se, are stable in a variety of crops for at least 3 years.

RECOMMENDATIONS

The registrant should be advised that GLN 171-4(e) is satisfied for residues of oxyfluorfen, per se, on a variety of crops for which residue trial data exists. The submitted storage stability study (MRID 43859801) support the integrity of the existing residue trial data.

A processing study remains outstanding for coffee.

DETAILED CONSIDERATIONS

Test System and Material

Samples of whole fruit and vegetable were chopped and stored frozen prior to fortification with oxyfluorfen (2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl) benzene), the 5CF3 isomer (2-chloro-1-(3-ethoxy-4-nitrophenoxy)-5-(trifluoromethyl) benzene), the 6' NO₂ isomer (2-chloro-1-(3-ethoxy-6-nitrophenoxy)-4-(trifluoromethyl) benzene), the 2' NO₂ isomer (2-chloro-1-(3-ethoxy-2-nitrophenoxy)-4-(trifluoromethyl) benzene), and the 4'H un-nitrated isomer (2-chloro-1-(3-ethoxyphenoxy)-4-(trifluoromethyl) benzene). To prepare fortified samples for aging, ten gram crop samples were fortified with 1.0 ppm oxyfluorfen and four of its isomers (listed above) and stored at or below -10°C until analyzed periodically at 0, 3, 6, 9, 12, 18, 24, and 36 months. For each storage period, a total of 5 samples were analyzed. The analysis set included: 2 aged, previously fortified samples representing the particular storage period, 2 freshly fortified samples used as fresh spikes on the day of extraction, and 1 unspiked control sample.

Analytical Method

Analyses were performed using the analytical method described in the method entitled, "Analytical Method for the Determination of GOAL[®] Herbicide (Oxyfluorfen and its Major Isomers Residues in Crops and Soil", Technical Report 34-92-15. The method determines oxyfluorfen and three of its isomers as heptafluorobutyramide derivatives. A fourth isomer is quantified by itself (underivatized) by gas chromatography coupled with electron capture detection. Adequate sample chromatograms and calculations were provided. The limit of quantitation (LOQ) was 0.01 ppm. The method is adequate for data collection. This method was referenced in CBRS No. 17194 (CAE, 06/18/96, DP Barcode: D225680) and is briefly described below.

After processing the crop sample, the target compounds are extracted by the following procedure: a portion of the processed sample is weighed out and extracted by high speed blending with methanol, then centrifuged. An aliquot of the supernatant is placed in a Bleidner

apparatus with sodium hydroxide solution and metallic aluminum. The compounds of interest are reduced to their corresponding amines and simultaneously steam distilled and extracted into hexane. The hexane extract is removed from the Bleidner apparatus and reacted with heptafluorobutyric acid anhydride to form the analytical derivatives. After concentration, the extract is fractionated using a Florisil column. The appropriate fraction collected from the Florisil column is concentrated, then brought to volume with isoctane for analysis by electron capture gas chromatography using ⁶³Ni electron capture detector.

Storage Stability Results

Storage stability data for residues of oxyfluorfen and four of its isomers on various crops for oxyfluorfen, per se, are presented in Table 1. Oxyfluorfen, per se, is the only regulated residue of oxyfluorfen (CBRS Nos. 12513, 12522, 13212, and 13228, SAK, 4/8/94). However, the isomer data can be obtained in the study submitted (MRID 43859801), if necessary. The crops tested for stability of oxyfluorfen residues were: apples, alfalfa, almond nuts and hulls, banana pulp, cabbage, cottonseeds, onions, oranges, peaches, strawberries, and wheat grain. Recoveries from fortified, aged samples (stored samples) have been corrected for method recoveries from freshly fortified samples analyzed concurrently with the aged fortified samples. Corrected recoveries were calculated as the average of the recoveries from the aged fortified samples divided by the average of the recoveries from the two freshly fortified samples multiplied by 100.

Table 1. Recoveries from aged and fresh crop samples fortified at 1.0 ppm with oxyfluorfen.					
Storage Period	Fresh Fortification %Recovery		Apparent % Recovery in Aged Samples		Corrected* % Recovery
Alfalfa					
0 day	90.7	88.4	83.9	253.1**	94
3 months	80.3	81.0	88.4	92.9	112
6 months	81.0	77.0	93.4	83.5	111
9 months	75.6	89.6	81.2	84.5	100
12 months	85.4	82.2	85.6	87.0	103
18 months	75.6	76.0	80.3	81.2	107
24 months	73.4	72.6	66.8	70.7	94
36 months	69.7	73.0	67.4	76.3	100
Almond hulls					
0 day	79.2	69.2	79.2	70.9	101

Table 1. Recoveries from aged and fresh crop samples fortified at 1.0 ppm with oxyfluorfen.

Storage Period	Fresh Fortification %Recovery		Apparent % Recovery in Aged Samples		Corrected* % Recovery
3 months	65.9	69.3	73.8	78.2	112
6 months	47.4	104.0	0.5	42.3	56
9 months	68.9	67.4	69.5	72.5	104
12 months	78.1	74.2	81.0	86.9	110
18 months	65.2	53.9	54.4	55.9	93
24 months	58.5	55.8	53.7	62.0	101
36 months	66.2	56.1	58.1	52.8	91
Almond Meats					
0 day	83.8	88.1	90.3	89.0	104
3 months	75.2	81.2	74.9	82.4	100
6 months	87.3	93.3	94.0	89.1	101
9 months	71.7	74.3	71.0	85.2	107
12 months	93.5	99.5	104.1	92.5	101
18 months	79.4	83.6	82.3	75.1	96
24 months	76.7	69.9	69.4	72.4	96
36 months	55.7	70.6	80.3	76.7	124
Apple					
0 day	82.4	63.3	82.5	83.2	114
3 months	77.4	71.0	86.5	83.3	114
6 months	73.2	71.8	69.9	79.7	103
9 months	70.8	67.9	74.1	68.3	83
12 months	76.7	75.3	72.8	88.2	106
18 months	69.0	63.9	72.8	69.2	107
24 months	65.2	64.8	68.1	68.4	105
36 months	71.0	74.3	75.6	77.1	105

Table 1. Recoveries from aged and fresh crop samples fortified at 1.0 ppm with oxyfluorfen.

Storage Period	Fresh Fortification %Recovery		Apparent % Recovery in Aged Samples		Corrected* % Recovery
Banana					
0 day	0.0	84.8	86.8	89.2	104
3 months	85.8	82.7	83.6	79.4	97
6 months	62.8	30.6	48.1	63.4	119
9 months	82.0	78.6	82.5	84.9	104
12 months	91.7	84.6	91.0	100.8	109
18 months	71.1	77.2	73.6	77.8	102
24 months	80.7	72.7	81.0	71.6	99
36 months	68.8	63.2	75.5	79.3	117
Cabbage					
0 day	73.7	75.1	76.8	79.6	105
3 months	51.8	45.6	55.4	52.0	110
6 months	57.3	56.6	59.7	57.6	103
9 months	73.5	70.5	73.3	67.0	97
12 months	75.2	74.9	80.7	82.0	108
18 months	71.2	69.5	72.8	72.6	103
24 months	78.7	76.9	64.4	63.9	82
36 months	59.7	67.2	73.2	71.3	117
Cotton					
0 day	68.9	59.2	68.0	56.5	98
3 months	106	7.5	58.6	82.3	91
6 months	67.8	71.5	61.1	74.2	95
9 months	74.5	73.8	74.2	74.8	100
12 months	79.7	93.1	79.1	94.3	100
18 months	71.5	73.1	67.9	66.8	94

Table 1. Recoveries from aged and fresh crop samples fortified at 1.0 ppm with oxyfluorfen.

Storage Period	Fresh Fortification %Recovery		Apparent % Recovery in Aged Samples		Corrected* % Recovery
24 months	78.0	74.7	0.8**	68.2	89
36 months	60.4	47.8	60.1	60.9	107
Onion					
0 day	55.2	52.3	49.1	80.2	110
3 months	77.5	78.9	83.1	84.4	107
6 months	79.9	72.0	78.1	89.7	106
9 months	75.8	67.9	77.2	79.0	106
12 months	67.9	64.9	88.0	98.1	137
18 months	74.6	70.8	89.5	85.9	120
24 months	59.9	57.7	67.4	73.9	118
36 months	73.4	71.4	74.4	75.4	103
Oranges					
0 day	86.9	81.9	88.6	83.6	102
3 months	76.0	73.4	84.3	88.7	114
6 months	74.8	81.6	80.6	68.6	99
9 months	82.0	82.7	85.5	81.9	103
12 months	84.8	87.6	94.6	90.8	109
18 months	82.2	84.3	89.1	83.0	105
24 months	78.6	72.1	74.4	75.0	98
36 months	71.2	70.4	77.3	84.8	112
Peach					
0 day	74.7	69.9	74.8	77.0	103
3 months	82.5	75.6	81.1	69.7	96
6 months	76.5	68.6	72.9	72.1	98
9 months	76.6	69.8	73.7	77.3	101

Table 1. Recoveries from aged and fresh crop samples fortified at 1.0 ppm with oxyfluorfen.					
Storage Period	Fresh Fortification %Recovery		Apparent % Recovery in Aged Samples		Corrected* % Recovery
	12 months	81.7	82.4	82.7	
18 months	81.1	71.7	81.3	76.7	102
24 months	73.7	73.9	79.2	82.5	109
36 months	59.3	73.9	72.2	64.8	109
Strawberry					
0 day	80.6	77.8	81.3	79.2	101
3 months	80.5	79.1	72.8	83.4	95
6 months	69.5	77.3	63.7	80.8	96
9 months	84.1	79.8	85.2	82.6	102
12 months	73.1	65.5	67.4	73.3	98
18 months	78.2	78.0	82.4	82.5	106
24 months	69.2	69.4	72.2	68.1	102
36 months	72.1	70.1	73.5	66.6	100
Wheat Grain					
0 day	80.8	79.9	80.2	74.9	97
3 months	95.6	99.1	79.9	81.5	83
6 months	83.3	80.1	69.0	71.4	85
9 months	90.9	84.9	82.6	79.4	92
12 months	84.4	80.4	76.4	74.6	91
18 months	81.2	78.5	77.3	72.8	94
24 months	87.5	81.5	78.5	81.2	93
36 months	61.9	60.0	51.3	45.5	81

* Recoveries in this column have been corrected for concurrent method recoveries from fresh samples fortified on the day of analysis. ** Value not included in correction calculation for percent recovery.

Results from the storage stability study for apples, alfalfa, almond nutmeats and hulls, banana pulp, cabbage, cottonseeds, onions, oranges, peaches, strawberries, and wheat grain indicate that

residues of oxyfluorfen, per se, are stable in all of these crops for up to 36 months (3 years). These data can be translated to all crops for which residue trial data exist and support the integrity of those residue trial data.

Storage intervals for a variety of crops prior to their analysis for oxyfluorfen residues varied from 34 to 1123 days (3 years). The submitted storage stability data adequately support the submitted residue trial data for residues of oxyfluorfen, per se, on field trial samples. Specific storage intervals for the following field trial crop samples were: avocado (272 days), cabbage (11 months), cauliflower (11 months), corn (209 days), cotton (24 months), figs and dates (306 days), grapes (6-24 months), guava (2-3 months), kiwi (268 days), mint hay (2-13 months), olives (288 days), onions (11 months), pistachios (10 months), pome fruits (269 to 287 days), pomegranates (268 days), soybeans (2 years), stone fruits (1123 days), tree nuts (18 months), horseradish (10 months), artichokes (444 days). All samples were stored at -10°C, except guava which was stored at 5°C. A separate storage stability study with guava was conducted and determined that residues of oxyfluorfen, per se, were stable on guava for at least 4 months at 5°C. (See Phase IV review, S. Funk, 12/04/90; CBRS Nos. 16313, 16374, 17049, 17106, 17121, CAE & SAK).

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