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2-8-93

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

FEB 8 1993

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Reregistration of Trifluralin. Corn grain processing studies. CBRS No. 10338. DP Barcode No. D181183. MRID Nos. 42403201. Chemical No. 036101.

FROM: Bonnie Cropp-Kohlligian, Environmental Scientist
Reregistration Section II
Chemistry Branch II: Reregistration Support
Health Effects Division [H7509C]

THRU: Edward Zager, Chief
Chemistry Branch II: Reregistration Support
Health Effects Division [H7509C]

TO: Lois Rossi/Walter Waldrop [PM-71]
Reregistration Branch
Special Review and Reregistration Division [H7508W]

Attached is the review of data submitted by DowElanco and the Trifluralin Data Development Consortium in response to reregistration requirements for corn grain processing data. This information was reviewed by Acurex Corporation under supervision of CBRS, HED and deemed adequate pending the submission of supporting data. The data assessment has undergone secondary review in the Branch and has been revised to reflect Branch policies.

It is recommended that a copy of this review be sent to the Registrant.

If you need additional input, please advise.

Attachment 1: Trifluralin CBRS No. 10338; DP Barcode D181183. Registrant's Response to Residue Chemistry Data Requirements.

cc: BLCKohlligian, Jonathan Fleuchaus (PTSD; LE-132P), Circulate, Trifluralin Reg. Std. File, SF, Acurex.
cc: RF (without attachment).
RDI: WHazel:1/27/93 MMetzger:2/8/93 EZager:2/8/93
H7509C:CBRS:BLCKohlligian:CM#2:Rm 803:703-305-7462:11/5/92.

TRIFLURALIN
(Chemical Code 036101)
(CBRS No. 10338; DP Barcode D181183)

TASK 3

Registrant's Response to Residue Chemistry Data Requirements

October 16, 1992

Contract No. 68-DO-0142

Submitted to:

U.S. Environmental Protection Agency
Arlington, VA 22202

Submitted by:

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TRIFLURALIN

(Chemical Code 036101)

(CBRS No. 10338; DP Barcode D181183)

REGISTRANT'S RESPONSE TO RESIDUE CHEMISTRY REQUIREMENTS

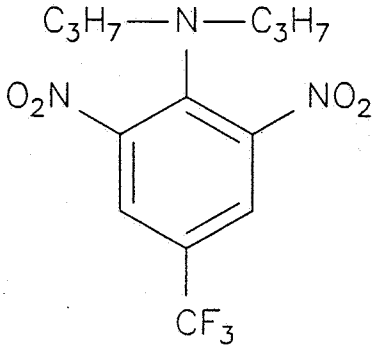
Task 3

BACKGROUND

The Trifluralin Guidance Document dated 4/87 required data depicting trifluralin residues in wet and dry milled products, including crude and refined oil, processed from corn grain bearing measurable weathered residues. This data gap was reiterated in the 10/91 Trifluralin Reregistration Standard Update. In response, DowElanco and the Trifluralin Data Development Consortium (1992; MRID 42403201) submitted data from a corn grain processing study. This submission is reviewed here to determine its adequacy in fulfilling residue chemistry data requirements. The Conclusions and Recommendations stated in this review pertain only to the magnitude of trifluralin residues in processed corn grain commodities.

The nature of the residue in plants and animals is adequately understood. The residue of concern in both plants and animals is trifluralin per se. Adequate analytical methods are available for enforcing trifluralin tolerances in plants. These methods are listed in PAM, Vol. II (Sec 180.207) as Methods II, III, and B.

Tolerances for residues of trifluralin, α,α,α-trifluoro-2,6-dinitro-*N,N*-dipropyl-*p*-toluidine, in or on raw agricultural commodities are currently expressed in terms of trifluralin per se (40 CFR §180.207 and §185.5900). As there are no Codex MRLs for residues of trifluralin, there is no question with respect to Codex/U.S. tolerance compatibility.



Trifluralin

CONCLUSIONS

1a. The subject corn grain processing study is adequate to ensure that residues of trifluralin do not concentrate in corn processed commodities pending the submission of adequate evidence concerning the phytotoxicity of trifluralin to corn plants when applied at rates greater than 5 lb ai/A.

1b. CBRS concludes that available storage stability data are adequate to support the subject corn processing residue data.

1c. No additional corn grain dust data are required. Data in the subject corn grain processing study indicate that residues of trifluralin do not concentrate in grain dust samples. Furthermore, CBRS expects that trifluralin residues are unlikely to occur on the grain surface since trifluralin is not applied late in the growing season.

2a. The registrant must amend trifluralin product labels to include a PHI, or latest allowable growth stage for treatments to corn since trifluralin applications may be made later in the growing season as evidenced by the subject study in which a single postemergence broadcast spray was applied to corn plants that were approximately 31 inches in height. It must be assured that the available data support the proposed PHI or growth stage.

2b. The amendment to trifluralin product labels to include a PHI or latest allowable growth stage for trifluralin treatments to corn may affect the registered use of trifluralin on sorghum since some labels combine both corn and sorghum and specify the same use directions. The registrant must amend trifluralin product labels to include a PHI or latest allowable growth stage for treatments to sorghum since trifluralin applications may be made later in the growing season. Again, assurances must be made that the available data support the proposed PHI or growth stage.

RECOMMENDATIONS

Note to SRRD: The "(N)" designation is inappropriate and should be deleted from all 40 CFR §180.207 entries.

DETAILED CONSIDERATIONS

Residue Analytical Methods

Trifluralin residues were determined using several related methods that utilize GC with electron capture detection (ECD). These methods are all modifications of Eli Lily Method AM-AA-CA-RO23-AA755, which was previously described in the Trifluralin Residue Chemistry Chapter dated 7/85, and is itself a modification of Method II in PAM, Vol II. (Sec. 180.207).

All sample matrices, excluding oils and soapstock, were analyzed using DowElanco Method GRM92.11. In this method, samples of grain, grits, meal, flour, dust, and presscake are extracted with hexane and cleaned up using a silica solid phase extraction (SPE) cartridge eluted with toluene. Residues in the toluene fractions are then analyzed by GC/ECD. Samples with high moisture contents (starch and gluten) are extracted with methanol and cleaned up using a reverse-phase (C18) SPE cartridge eluted with hexane. Residues are further purified using silica SPE cartridges eluted with toluene. Residues in the resulting toluene fraction are then analyzed by GC/ECD.

Oil and soapstock samples were analyzed using a related method from ABC Laboratory. For this method, oil samples are extracted with hexane and residues in the hexane fraction are partitioned into acetonitrile. The acetonitrile fraction is then diluted with 5% NaCl and the residues are partitioned into hexane and concentrated. Residues are reconstituted in hexane and cleaned up using a Florisil column eluted with hexane. Residues in the Florisil-purified hexane fraction are dried, reconstituted in toluene, and then analyzed by GC/ECD. Soapstock samples are extracted with methanol and filtered. The methanol extracts are then diluted with 5% NaCl, and residues are partitioned into methylene chloride and concentrated. Residues are reconstituted in hexane and cleaned up using a Florisil column eluted with hexane. Residues in the Florisil-purified hexane fraction are dried, reconstituted in toluene, and then analyzed by GC/ECD.

For method validation, duplicate samples of each matrix were fortified with trifluralin at 0.01, 0.05, 0.1, and 0.2 ppm. Although the detection limit for each of the above methods was reported as 0.01 ppm, residues recovered from the 0.01 ppm fortifications were reported as <0.05 ppm. Therefore, for the purposes of this review, the detection limit for the above methods is considered to be 0.05 ppm for all corn matrices. Method recoveries from control samples fortified at 0.05-0.2 ppm are summarized in Table 1. The registrant reported that control and treated soapstock samples contained an endogenous interference peak at the retention time for trifluralin; however, additional analysis using GC/MS indicated that the endogenous peak was not trifluralin. Sample calculations and chromatograms were provided. These data indicate that the GC/ECD methods are adequate for collecting data on residues of trifluralin per se from corn grain and dust and from processed corn matrices.

Table 1. Recovery of trifluralin from corn grain and dust and processed corn commodities fortified with trifluralin at 0.05-0.2 ppm.

Matrix	Number of Samples	Percent Recovery
Whole grain	6	101 - 108
Meal	6	105 - 114
Grits	6	100 - 114
Flour	6	108 - 119
Dust	6	102 - 134[b]
Starch	6	94 - 121
Gluten	6	75 - 123
Presscake	12	102 - 123
Soapstock[a]	12	74 - 126
Crude Oil	12	98 - 168[b]
Refined Oil	12	91 - 138[b]

[a]Recoveries from soapstock were corrected for an interfering background peak. [b]Recoveries in excess of 120% occurred at the 0.05 ppm level of fortification.

Storage Stability Data

Whole corn grain was stored at ≤-10 °C for approximately 100 days prior to processing. After processing, corn grain and the processed fractions were stored at -20 °C for approximately 450 days prior to analysis. The total storage interval for whole grain samples was approximately 550 days.

Previously, the registrant submitted data (1989;MRID 41335901) pertaining to the storage stability of trifluralin residues in or on 24 crop matrices (including wheat forage, potatoes, garlic, green peas, plums, cantaloupes, grapes, green hops, sugarcane, turnip greens, peanut meats, cabbage, carrots, cottonseed, flax seed, soybeans, soybean oil, wheat straw, wheat grain, alfalfa hay, dry beans, corn grain, peanut hulls, and sunflower seed) which were reviewed in the 10/91 Trifluralin Update. The samples were stored frozen at -25°C to -15°C for periods ranging from 79 to 554 days and, without exception, showed little or no significant decline in trifluralin residue levels during the frozen storage periods. Specifically wheat forage, cottonseed, flax seed, soybeans, soybean oil, and wheat straw commodities were stored frozen at -25°C to -15°C for ca. 500 days, or more. Almost half of the commodities (cottonseed, flax seed, soybeans, soybean oil, wheat grain, wheat straw, dry beans, corn grain, peanut meats, peanut hulls, and sunflower seed) were initially stored at 20°C to 25°C for 7 days and then refrigerated for the next 53 days at ca. 4°C prior to being stored in their frozen state. Of these commodities, only flax seed, soybeans, and wheat grain showed any significant decline in trifluralin residues levels (as much as 50%) as a result of

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storage at the higher temperatures. In addition, data from a recently reviewed (CBRS. No. 9991) sorghum processing study (1992; MRID 42325001) indicate that trifluralin is stable at -20 °C for at least 114 days in sorghum grain, 58 days in starch, and 120 days in flour. Available data also indicate that residues are stable in soybeans for at least 908 days (CBTS 9845, N. Dodd, 8/20/92).

No data are available demonstrating the frozen storage stability of trifluralin in corn grain, grits, meal, flour, and starch for the storage intervals in the current study. However, due to the preponderance of data demonstrating the stability of trifluralin residues in/on a number of dissimilar commodities (including oilseed, non-oily grain, leafy vegetable, root, and fruit crop representatives) when stored frozen, CBRS concludes that the available storage stability data are adequate to support the subject corn processing data.

Magnitude of the Residue

Corn Processed Commodities. A tolerance of 0.05 (N) ppm has been established for residues of trifluralin per se in or on corn grain (excluding popcorn) 40 CFR §180.207. Trifluralin is currently registered for a single application to field corn as a broadcast or directed postemergence spray that is then incorporated into the soil. Trifluralin is applied relatively early in the growing season when plants are at least 8 inches in height. The maximum recommended use rate depends on soil type and is 0.5 lb ai/A for course textured soils, 0.75 lb ai/A for medium textured soils, and 1 lb ai/A for fine textured soils. No PHI is listed for corn grain.

DowElanco and the Trifluralin Data Development Consortium (1992; MRID 42403201) submitted data depicting trifluralin residues in or on corn grain and dust and in processed corn commodities. In a test conducted in IA, trifluralin (4 lb/gal EC) was applied at 5 lb ai/A (5x the maximum label rate) as a single postemergence broadcast spray to corn plants approximately 31 inches in height. The trifluralin was immediately incorporated into the soil, which was classified as a loam (medium textured soil). The registrant characterized the 5x rate as "the highest surviving rate plot," but did not specify or provide any evidence that rates greater than 5 lb ai/A produce adverse phytotoxic effects. Control and treated plots were harvested 86 days posttreatment and corn grain samples were immediately frozen. Prior to processing, grain samples were stored at approximately ≤-10 °C for approximately 100 days. Using commercially simulated practices, corn grain was dry-milled to yield grain dust, grits, meal, flour, presscake, soapstock, and crude and refined oil, and wet-milled to yield starch, gluten, presscake, soapstock, and crude and refined oil. After processing, samples of grain and each processed fraction were stored at -20 °C for approximately 450 days prior to analysis.

Duplicate control and treated samples of corn grain and dust and each processed commodity were analyzed for trifluralin residues using one of several related GC/ECD methods. Apparent residues of trifluralin in or on control samples of grain and dust and in each of the

processed commodities were nondetectable (<0.05 ppm). Trifluralin residues were nondetectable (<0.05 ppm) in or on grain harvested from corn treated postemergence with trifluralin at 5 lb ai/A (5x). Trifluralin residues were also nondetectable in all commodities processed from 5x treated grain. However, the exaggerated rate used in the subject study (5x) is less than the theoretical concentration factor for corn oil (25x according to CBRS memo entitled, Maximum Theoretical Concentration Factors, prepared by S. Hummel, dated 1/93).

References

Citations for the MRID documents referenced in this review are presented below. Submissions reviewed in this document are indicated by shaded type.

42325001 Rice, F.; Gresham, M.E. (1992) Magnitude of the Trifluralin Residues in Grain Sorghum Processed Commodities: Report No. 38640. Unpublished study prepared by ABC Laboratories Inc. 147 p.

42403201 Shackelford, D. (1992) Processing Study with Trifluralin on Corn Grain: Laboratory ID No. AAC9004. Unpublished study prepared by ABC Laboratories Inc. and DowElanco. 131 p.

TRIFLURALIN (0179/036101)
TENTATIVE RESIDUE CHEMISTRY DATA SUMMARY THROUGH 1/93[1]

REASSESSMENT OF U.S. TOLERANCES AND POTENTIAL FOR HARMONIZATION WITH CODEX[2]

Guideline Number and Topic[3]	Phase V data requirements satisfied?[4]	MRID(s)[5]
171-3 Directions for use	No	
171-4(a) Plant Metabolism	Yes	
171-4(b) Animal Metabolism	Yes	
171-4(c) Residue Analytical Methods - Plants	Yes	
171-4(d) Residue Analytical Methods - Animals	No	
171-4(e) Storage Stability	No	
171-4(k) Crop Field Trials		
171-4(k) Root and Tuber Vegetables Group[6]		
Carrots	Yes	
Potatoes [see 171-4(l)]	Yes	
Sugar beets [see 171-4(l)]	Yes	
Turnips	Yes	
171-4(k) Leaves of Root and Tuber Vegetables		
Turnip tops	Yes	
171-4(k) Bulb Vegetables Group		
Garlic	Yes	
Onions (dry bulb)	Yes[7]	
171-4(k) Leafy Vegetables (except Brassica)		
Celery	Yes	
Upland Cress	No	
171-4(k) Brassica Leafy Vegetables Group		
Broccoli	Yes	
Brussels sprouts	Yes	
Cabbage	Yes	
Cauliflower	Yes	
Collards	Yes	
Kale	Yes	
Mustard greens	Yes	
171-4(k) Legume Vegetables (succulent/dried)		
Adzuki Beans	Yes	
Beans (dried)	Yes	
Field Peas (Cowpeas, Black-eyed peas)	Yes	
Guar Beans [see 171-4(l)]	Yes	
Mung Beans	Yes	
Peas (succulent and dried)	Yes	
Snap Beans	Yes	
Soybeans [see 171-4(l)]	Yes[8 9 10 11 12]	
171-4(k) Foliage of Legume Vegetables		

TRIFLURALIN (0179/036101)
TENTATIVE RESIDUE CHEMISTRY DATA SUMMARY THROUGH 1/93[1]
REASSESSMENT OF U.S. TOLERANCES AND POTENTIAL FOR HARMONIZATION WITH CODEX[2]

Guideline Number and Topic[3]	Phase V data requirements satisfied?[4]	MRID(s)[5]
Bean vines and hay	Yes	
Pea vines and straw	Yes	
Soybean forage, hay and straw	Yes	
171-4(k) Fruiting Vegetables Group		
Peppers	Yes	
Tomatoes [see 171-4(l)]	Yes	
171-4(k) Cucurbit Vegetables Group		
Cantaloupes	Yes	
Cucumbers	Yes	
Summer Squash	No[13]	42354502
Watermelons	Yes	
171-4(k) Citrus Fruits Group		
Grapefruit	Yes	
Lemons	Yes	
Oranges [see 171-4(l)]	Yes	
Tangeloes	Yes	
Tangerines	Yes	
171-4(k) Stone Fruits Group		
Apricots	Yes	
Nectarines	Yes	
Peaches	Yes	
Plums (fresh prunes) [see 171-4(l)]	Yes	
171-4(k) Small Fruits and Berries Group		
Grapes [see 171-4(l)]	Yes	
171-4(k) Tree Nuts Group		
Almonds	Yes	
Pecans	Yes	
Walnuts	Yes	
171-4(k) Cereal Grains Group		
Barley [see 171-4(l)]	Yes[14]	
Corn (field) [see 171-4(l)]	No[15]	
Sorghum [see 171-4(l)]	Yes[16]	
Wheat [see 171-4(l)]	Yes	
171-4(k) Forage, Fodder, and Straw of Cereal Grains		
Barley forage, hay, and straw	Yes	
Corn forage, fodder, and silage	No	
Sorghum forage, fodder, silage, and hay	No	
Wheat forage, hay, and straw	No	
171-4(k) Non-grass Animal Feeds		
Alfalfa forage and hay [see 171-4(l)]	No[17]	

TRIFLURALIN (0179/036101)
TENTATIVE RESIDUE CHEMISTRY DATA SUMMARY THROUGH 1/93[1]

REASSESSMENT OF U.S. TOLERANCES AND POTENTIAL FOR HARMONIZATION WITH CODEX[2]

Guideline Number and Topic[3]	Phase V data requirements satisfied?[4]	MRID(s)[5]
171-4(k) Miscellaneous Commodities		
Asparagus	Yes	
Cottonseed [see 171-4(l)]	No	
Flax [see 171-4(l)]	No	
Hops [see 171-4(l)]	Yes	
Mustard seed	No	
Peanuts [see 171-4(l)]	No	
Peppermint [see 171-4(l)]	No	
Rape seed	Yes[18]	
Safflower seed	Yes	
Spearmint [see 171-4(l)]	No	
Sugarcane [see 171-4(l)]	No[19 20]	
Sunflower seed and forage [see 171-4(l)]	No	
171-4(l) Processed Food/Feed		
Corn, Field	No[21]	42403201
Cotton	No[22]	42354501
Oranges	No	
Peanuts	No	
Peppermint	No	
Potato	No[23]	
Sorghum, grain	Yes[24]	42325001
Soybeans	No	
Spearmint	No	
Sugar beets	No[25]	
Sugarcane	No	
Sunflower	No	
Wheat	No	
171-4(j) Meat/Milk/Poultry/Eggs	No	
171-4(f) Potable Water	N/A	
171-4(g) Fish	N/A	
171-4(h) Irrigated Crops	N/A	
171-4(i) Food Handling Establishments	N/A	
171-5 Reduction of Residues	N/A	

[1]Registration Standard issued 7/3/85. Reregistration Standard Update issued 10/29/91.

[2]There are no Codex MRLs for residues of trifluralin. Therefore, the question of compatibility between Codex and U.S. tolerances is rendered moot.

[3]N/A = Guideline requirement not applicable.

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[4]Applies to List B only; List A chemicals were not subject to Phase IV of FIFRA '88.

[5]MRIDs that were reviewed in the current submission are designated in shaded type.

[6]CBRS 7827 by P. Deschamp dated 4/25/91. Registrant proposed conducting field trials in CA and FL to determine residues in/on radishes and processing studies on potatoes and sugar beets to support tolerance for members of the Root and Tuber Vegetables Group (excluding carrots).

[7]CBRS 8771 by N. Dodd dated 12/30/91. Registrant submitted a Section 3 label amendment for trifluralin on onions. No new residue data were submitted.

[8]CBRS 8832 by W. Chin dated 12/6/91. Registrant requested an amended use. No new residue data were submitted.

[9]CBRS 8560 by N. Dodd dated 3/10/92. Registrant requested an EUP for XRM-5313 on soybeans. No new residue data were submitted.

[10]CBRS 9506 by N. Dodd dated 6/12/92. Registrant requested a label amendment. No new residue data were submitted.

[11]CBRS 9845 by N. Dodd dated 8/20/92. Registrant submitted amendment to its petition for temporary tolerances in/on soybeans.

[12]CBRS 8400 and 8646 by N. Dodd dated 3/27/92. Registrant proposed temporary tolerances for residues of trifluralin in/on field corn fodder, corn forage, corn grain and soybeans. Storage stability data were submitted which demonstrated that residues of DE-498 are stable in/on soybeans for up to 411 days when stored frozen (MRID 419317). Storage stability data submitted on field corn forage, fodder, and grain (MRID 419317-21) were deemed inadequate for evaluation. Field corn magnitude studies (MRID unspecified) were conducted at 16 locations in 13 states but CBTS deemed them inadequate. No corn processing data were submitted. Soybean field trials were conducted (MRID 419521-06, 419317-19, and 419317-20) but CBTS deemed them inadequate. No soybean processing data were submitted.

[13]CBRS 10143 by B. Cropp-Kohlligian dated 9/28/92. The field trial data for summer squash are adequate to support a cucurbits vegetable group tolerance.

[14]CBRS 9453 by N. Dodd dated 7/2/92. CBTS review of supplemental label for trifluralin on barley. No new residue data submitted.

[15]CBRS 5966 by W. Anthony dated 1/29/90. Label amendment request. No new residue data submitted.

[16]CBRS 5966 by W. Anthony dated 1/29/90. Label amendment requested. No new residue data submitted.

[17]CBRS 9501 by N. Dodd dated 8/6/92. Registrant requested a label amendment. No new residue data were submitted.

[18]CBRS 8833 by W. Chin dated 11/22/91. Registrant requested a label amendment. No new residue data were submitted.

[19]CBRS 7063 by W. Anthony. Label amendment requested. No new residue data submitted.

[20]CBRS 8074 by W. Anthony dated 7/22/91. Label amendment requested. Registrant cited residue data (MRID No. 413067-01) which was submitted and reviewed in the Trifluralin Reregistration Standard Update (10/29/91). No additional data submitted.

[21]CBRS 10338 by B. Cropp-Kohlligian dated 2/8/93. Corn processing study is deemed adequate pending the submission of evidence of phytotoxicity at rates >5 lb ai/A. A PHI or latest growth stage permitted to be treated is required.

[22]CBRS 10143 by B. Cropp-Kohlligian dated 9/28/92. The submitted cottonseed processing study is not adequate. The registrant must submit data depicting residues in cottonseed commodities processed from cottonseeds bearing measureable trifluralin residues or conduct field trial studies at exaggerated application rates equivalent to the maximum theoretical concentration factor (8x).

[23]CBRS 7827 by P. Deschamp dated 4/25/91. Registrant proposed conducting field trials in CA and FL to determine residues in/on radishes and processing studies on potatoes and sugar beets to support tolerance for members of the Root and Tuber Vegetables Group (excluding carrots).

[24]CBRS 9991 by B.Cropp-Kohlligian dated 9/28/92. Registrant submitted adequate grain sorghum processing data. Trifluralin residues are not likely to concentrate in flour or starch. Food Additive Tolerances are not required. No additional grain sorghum data are required.

[25]CBRS 7827 by P. Deschamp dated 4/25/91. Registrant proposed conducting field trials in CA and FL to determine residues in/on radishes and processing studies on potatoes and sugar beets to support tolerance for members of the Root and Tuber Vegetables Group (excluding carrots).

cc: BLCKohlligian; Trifluralin Reregistration Standard File; Lois Rossi, SRRD.