

DATA EVALUATION RECORD

STUDY 15

CHEM 036101

Trifluralin

\$164-1

FORMULATION--12--EMULSIFIABLE CONCENTRATE

STUDY ID 42309101

Decker, O.D. TRIFLURALIN DISSIPATION STUDY IN MANSFIELD, ILLINOIS AND SHELLMAN, GEORGIA. Sponsored and Submitted by Trifluralin Data Development Consortium; Performed by North American Environmental Chemistry Laboratory, DowElanco; Greenfield, IN under Laboratory Identification Number AAC8706; Study completed on 23 April 1992; Received by EPA 5 May 1992.

DIRECT REVIEW TIME - 1.5 days

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CONCLUSIONS:

Field Dissipation - Terrestrial

This field dissipation study is scientifically valid and can be used as supplemental data. However, it cannot be used to fulfill the data requirement (164-1) for the following reasons:

In order to validate the analytical data, the separatory GC data should be confirmed by another analytical methodology (preferably MS).

Degradates identified in laboratory data were not analyzed for in field samples, and the degradation pathway of trifluralin in the field could not be determined from this study (See Comments for details).

Emulsifiable concentrate trifluralin formulations were reported to dissipate with a half-life ranging from 29 to 35 days when applied to coarse (sandy loam soil at Shellman, GA site) and fine (silty clay loam soil at Mansfield, IL site) soils, respectively. However, granular trifluralin formulation was reported to dissipate with a half-life ranging from 15 to 86 days when applied to sandy loam soil in Shellman, GA. These half-lives were calculated from nonlinear dissipation curves. Furthermore, since trifluralin was not discernible in soil segments below the top 6 inches of soil, trifluralin did not demonstrate any leaching potential.

The mean recoveries immediate posttreatment for the emulsifiable concentrate at the Georgia and Illinois sites were 0.94 ppm (132% of applied) and

0.99 ppm (200% of applied) in the 0-6" soil depth, respectively. For the granular formulation the mean recoveries immediate posttreatment at the Georgia and Illinois sites were 0.85 ppm (113% of applied) and 0.67 ppm (134% of applied), respectively. By termination of study the mean recoveries for the emulsifiable concentrate were 0.04 ppm (Day 398 posttreatment) and 0.09 ppm (Day 193 posttreatment) for the Illinois and Georgia sites, respectively. For the granular formulation, at termination of study the mean recoveries were 0.04 ppm (Day 573 posttreatment) and 0.10 ppm (Day 549 posttreatment) for the Illinois and Georgia sites, respectively. There was an increase in the mean at Day 7 posttreatment at the Georgia site for the emulsifiable concentrate formulation (1.91 ppm which is  $\approx$ 400% of applied) and at the Illinois site for the granular formulation (1.01 ppm which is 135% of applied).

#### METHODOLOGY:

Trifluralin formulations, TREFLAN EC or TREFLAN MTF and TREFLAN TR10, were applied to separate bare soil plots on research farms at Mansfield, IL and Shellman, GA. Soil textural classification for the 0-6" soil depths were reported as silty clay loam for the Illinois site and sandy loam at the Georgia site (See Table III for details). Site descriptions (See Table I and Figures 1 & 2) and pesticide use histories (See Table II) were furnished, as well.

Prior to application the test sites were disced and harrowed. An application rate of 1.5 lb a.i./A was reported for the Mansfield site and 1.0 lb a.i./A for the Shellman site (See Table V). For liquid formulations, TREFLAN EC or TREFLAN MTF, the concentrate was mixed with water and applied to a series of 36-foot x 63.3-foot plots at Mansfield and 36-foot x 40-foot plots at the Shellman site (See Table VI). The granular formulation, TREFLAN TR10, was applied at the respective application rates (See Table VII). Immediately following application of test material, the chemical was disc incorporated. One untreated control plot at each site was prepared in the same manner (except no test material applied).

Soil samples for the liquid application test sites were collected prior to treatment, immediately after application (time 0), and at 7, 28, 68, 112, 175, and 398 days posttreatment at the Mansfield site and at 7, 34, 69, 118, 193, and 370 posttreatment at the Shellman site. For the granular application test sites, soil samples were collected immediately after application (time 0), and at 7, 28, 68, 112, 175, 398, and 573 days posttreatment at the Mansfield site and 7, 34, 69, 118, 370, and 549 days posttreatment at the Shellman site. Sampling was terminated basically when residue levels declined to <10% of initial application rate. Control samples were collected for only single sample assays. These soil samples were collected at 0-6, 6-12, and 12-18 inch soil depths for pre- and post-sampling intervals up to and including Day 68 at the Mansfield site and Day 69 at the Shellman site. Samples collected after these days included 18-24 and 24-30 inch soil depths. However, the last sample interval of the granular treated plot at the Mansfield site included a 30-36 inch soil depth segment. See Figures 6, 7, 8, 9, and 10 for details on sampling technique.

The top six inch soil sample was collected using a 4.25 inch diameter core cutter. These 0-6" soil cores inverted and three 0.75 inch diameter cores (0-6" segments) were removed from the 4.25 inch diameter cores. The 0.75 inch diameter cores were transferred to a brown glass jar and weighed. After the soil sample was weighed 200 mL of solvent (99:1 acetonitrile: water) was added to each jar, sealed, and shaken for dispersion prior to shipment to the laboratory for analysis. The 6-18 inch soil depth sample was taken with a 1.25 inch sample probe and the sample divided into two soil segments (6-12 inch and 12-18 inch). The 18-30 or 18-36 inch sample was taken with a 0.875 sample probe and segmented into 6 inch soil seg-

ments. All soil segments were handled identical to the 0-6 inch soil segments.

Normal transportation time ranged from 1-10 days to the Department of Analytical Chemistry, Lilly Research Laboratories, Greenfield, IN for analysis. Sample extracts were stored at 4°C until analyzed. In order to determine sample integrity during sample transportation, preparation, storage, and analysis, soil samples were spiked at 0.02, 0.094, and 2.02 ppm in the field and shipped with test samples to the laboratory (See Tables IX and X).

After soil samples were extracted with acetonitrile:water (99:1/v,v), an aliquot of the extract was diluted with deionized water to a maximum acetonitrile content of 50%. The diluted sample was then purified by C18 solid phase extraction and analyzed for trifluralin by gas chromatography.

Air temperature, soil temperature, precipitation, irrigation volumes, and pan evaporation were monitored daily. For the duration of the field dissipation studies, temperatures in Mansfield varied from the norms by an average of +0.25°F and in Shellman by an average of -1.03°F, and rainfall varied from the norm by an average of +0.29 inch at the Mansfield location and -0.35 inches at the Shellman location. Soil temperatures and pan evaporation data were obtained from near by weather stations to the individual test sites.

#### DATA SUMMARY:

Emulsifiable concentrate trifluralin formulations were reported to dissipate with a half-life ranging from 29 to 35 days when applied to coarse (sandy loam soil at Shellman, GA site) and fine (silty clay loam soil at Mansfield, IL site) soils, respectively. However, granular trifluralin formulation was reported to dissipate with a half-life ranging from 15 to 86 days when applied to sandy loam soil in Shellman, GA. These half-lives were calculated from nonlinear dissipation curves. Furthermore, since trifluralin was not discernible in soil segments below the top 6 inches of soil, trifluralin did not demonstrate any leaching potential.

The mean recoveries immediately posttreatment for the emulsifiable concentrate at the Georgia and Illinois sites were 0.94 ppm (132% of applied) and 0.99 ppm (200% of applied) in the 0-6" soil depth, respectively. For the granular formulation the mean recoveries immediately posttreatment at the Georgia and Illinois sites were 0.85 ppm (113% of applied) and 0.67 ppm (134% of applied). By termination of study the mean recoveries for the emulsifiable concentrate were 0.04 ppm (Day 398 posttreatment) and 0.09 ppm (Day 193 posttreatment) for the Illinois and Georgia sites, respectively. For the granular formulation the mean recoveries were 0.04 ppm (Day 573 posttreatment) and 0.10 ppm (Day 549 posttreatment) for the Illinois and Georgia sites, respectively. There was an increase in the mean at Day 7 posttreatment at the Georgia site for the emulsifiable concentrate formulation (1.91 ppm which is ≈400% of applied) and at the Illinois site for the granular formulation (1.01 ppm which is 135% of applied).

Control samples were reported to be below the limit of detection or above the limit of detection and below the limit of quantitation. However, duplicate samples for those above the limit of detection were not consistent which indicated either field or laboratory contamination.

#### COMMENTS:

1. Degradates (a,a,a-trifluoro-2,6-dinitro-N-propyl-p-toluidine, a,a,a-trifluoro-5-nitro-4-propyl-toluene-3,4-diamine, 2-ethyl-7-nitro-1-propyl-5-(trifluoromethyl) benzimidazole-3-oxide, 2-ethyl-7-nitro-1-propyl-5-(trifluoromethyl)benzimidazole, 2-ethyl-7-nitro-5-(trifluoromethyl)benzimidazole; a,a,a-trifluoro-2,6-dinitro-p-cresol, and 2,2'-azoxybis(a,a,a-tri-fluoro-6-nitro-N-propyl-p-toluidine) were identified at maximum concentrations of 0.092-0.006 ppm (4.6%, 2.1%, 0.3%, 1.0%, 2.6%, 2.7% and 3.0%, respectively, of applied radioactivity) in aerobic metabolism study (MRID 41240501). Furthermore, the major degradates identified in anaerobic metabolism were a,a,a-trifluoro-5-nitro-N4, N4-dipropyl-toluene-3,4-diamine (which reached a maximum concentration of 5.4% (0.104 ppm) and 13.2% (0.264 ppm) of the applied radioactivity in the sandy loam soil and clay loam soil, respectively, at Day 60 postflooding, and 11.6% in the loam soil at Day 30 postflooding), 7-amino-2-ethyl-1-propyl-5-(trifluoromethyl) benzimidazole (which reached 7.3% in the sandy loam soil and 8.3% in the loam and clay loam soils at Day 60 postflooding), and a,a,a-trifluoro-N4,N4-dipropyltoluene-3,4,5-triamine (which reached 0.3% in the sandy loam soil, 4.1% in the loam soil, and 2.6% in the clay loam soil). Other degradates identified were a,a,a-tri-fluoro-2,6-dinitro-N-propyl-p-toluidine, a,a,a-trifluoro-5-nitro-N4-propyl-toluene-3,4-diamine, 2-ethyl-7-nitro-1-propyl-5-(trifluoromethyl)benzimidazole, and 2,2'-azoxybis(a,a,a-trifluoro-6-nitro-N-propyl-p-toluidine), which were each present at concentrations  $\leq 2.1\%$  (0.042 ppm) of the initial radioactivity. Degradates, 2-ethyl-7-nitro-1-propyl-5-(trifluoromethyl)benzimidazole-3-oxide and 7-amino-2-ethyl-5-(trifluoromethyl)benzimidazole, were each present at  $\leq 1.0\%$  of the initial radioactivity.

2. An application rate of 1.0 or 1.5 lb ai/A was reported for the study. Application rates range from 0.28 to 8.0 lb ai/A (normally used at 1/2 to 1 lb ai/A) on all sites except for prepaving applications for which the rates range from 12 to 16 lb ai/A.

3. EFGWB prefers that residues in samples be separated by chromatographic methods (such as TLC, HPLC, and GC) solvent systems of different polarity, and that specific compounds isolated by chromatography be identified using a confirmatory method such as MS in addition to comparison to the  $R_f$  of reference standards.

In this study aliquots of the extracts were analyzed by GC.

4. For the test sites the pesticide history was furnished for the previous three years prior to the study. At the Mansfield, IL site Co serve (active ingredient-trifluralin/oryzalin) was applied in 1984. Pretreatment samples from both sites were collected and indicated no trifluralin residues were discernible.

5. The data reported for both sites was variable.

The mean recoveries immediate posttreatment for the emulsifiable concentrate at the Georgia and Illinois sites were 0.94 ppm (132% of applied) and 0.99 ppm (200%) in the 0-6" soil depth, respectively. For the granular formulation the mean recoveries immediate post-treatment at the Georgia and Illinois sites were 0.85 ppm (113% of applied) and 0.67 ppm (134% of applied). In addition, there was an increase in the mean at Day 7 posttreatment at the Georgia site for the emulsifiable concentrate formulation (1.91 ppm which is  $\approx 400\%$  of

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