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CHLORETHOXYPHOS (DPX-43898)

Task 1: Review and Evaluation of Individual Studies

January 14, 1993

Final Report

Contract No. 68D20057

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

Submitted by:
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CHLORETHOXYPHOS

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STUDY 1

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STUDY ID 41736821
Hawkins, D.R., B.C. Mayo, M.J. Redrup, and L.M. Haynes. 1989. The photodegradation of ^{14}C-DPX-43898 in water. Huntingdon Report No. HRC/DPT 174/8949; Du Pont Report No. AMR-931-87. Unpublished study performed by Huntingdon Research Centre Ltd., Huntingdon, Cambridgeshire, England, and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

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CONCLUSIONS:

Photodegradation in Water

1. Study MRID #41736821 is acceptable and completely satisfies the photodegradation in water (161-2) data requirement for chlorethoxyphos.

2. Chlorethoxyphos (tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos) [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester] photodegraded with a half-life of 27 days in sterile aqueous buffered solutions (pH 5) that were continuously irradiated with a UV-filtered xenon light source at approximately 25

C. The photolysis component alone for the degradation of chlorethoxyphos corrected for lamp intensity and a 12 hr. photoperiod was approx. 130 days. Chlorethoxyphos degraded with a half-life of 89 days when incubated in darkness under similar conditions. Degradates identified in the irradiated and dark control solutions were trichloroacetaldehyde and dichloroacetic acid.

METHODOLOGY:

Aliquots (20 mL) of sterile (autoclaved) 10 mM aqueous sodium acetate buffer solution adjusted to pH 5.0 were transferred to flat-topped borosilicate glass vessels (diameter 3 cm, height 8.5 cm) and treated at 1.0 ppm with tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; radiochemical purity 97%, specific activity 58.4 uCi/mg, New England Nuclear], dissolved in acetonitrile; the final concentration of the cosolvent (acetonitrile) was 1% by volume. The vessels were sealed, then placed in separate sockets of a water-cooled steel block. The steel block containing the vessels was placed in a photolysis apparatus, and the samples were continuously irradiated using a xenon arc lamp equipped with UV filters to eliminate radiation below 290 nm. It was reported that the intensity of the xenon lamp at sample distance (not specified) was 1.67 times greater than that of natural sunlight (midday in June at Huntingdon, England; 52°21'N, 0°15'W) at 290-400 nm (Figure 1); total radiant intensity received by the test solutions was not reported. The test solutions were maintained at 25 $\pm$ 1 C and continuously stirred using magnetic stirrers during irradiation. Additional aliquots of the sodium acetate buffer solution were placed in borosilicate glass tubes, treated at 1.0 ppm with [^{14}C]chlorethoxyphos as described above, sealed, and maintained at 25 $\pm$ 1 C in darkness in an incubator to serve as dark controls. Two vessels containing irradiated solutions and three tubes containing dark control solutions were collected at 0, 1, 2, 4, 7, and 15 days posttreatment.

Acetonitrile (5.0 mL) was added to each test solution, the sample was shaken for 2 minutes, then duplicate aliquots (200 uL) of each sample were analyzed for total radioactivity using LSC. Additional aliquots were analyzed by reverse-phase HPLC using a PRP-1 column eluted with a gradient mobile phase of acidified water (adjusted to pH 2.2 with phosphoric acid):acetonitrile; the column was equipped with UV (208 nm) detection. Eluate fractions were collected at 2-minute intervals and analyzed for total radioactivity by LSC. Radioactive peaks were identified by comparison to the retention times of radiolabeled reference standards of chlorethoxyphos, trichloroacetaldehyde, and dichloroacetic acid (Figure 2).

For degradate identification, additional vessels of sterile pH 5 buffer solution were treated at 1.0 ppm with [^{14}C]chlorethoxyphos and irradiated as described above. The test solutions were collected after 15 days of irradiation and sequentially partitioned twice with hexane and twice with diethyl ether; the aqueous phase was acidified

(pH 1) and partitioned twice with diethyl ether. The pH 1 diethyl ether phases were combined and partitioned into 0.1 N NaOH; the solution was acidified (pH 1) and partitioned twice with diethyl ether. All organic phases were analyzed for total radioactivity using LSC. Parent chlorethoxyphos was isolated in the hexane extracts; aliquots of the hexane extracts were analyzed by one-dimensional TLC on silica gel plates developed in either hexane:methylene chloride (7:3, v:v) or ethyl acetate:methanol:glacial acetic acid (180:50:3, v:v:v). Radioactive areas were visualized using a TLC linear analyzer and identified by comparison with a radiolabeled chlorethoxyphos reference standard that was cochromatographed with the samples. The degradate trichloroacetaldehyde was isolated in the pH 5 diethyl ether extracts. Aliquots of the ether extract were analyzed by HPLC using a Hypersil column eluted with an isocratic mobile phase of hexane:diethyl ether (90:10, v:v); the column was equipped with UV (208 nm) detection. Eluate fractions were collected and analyzed as previously described. An additional aliquot of the 15-day irradiated test solution was partitioned once with diethyl ether; aliquots of the organic phase were analyzed for trichloroacetaldehyde by gas-liquid chromatography (GLC) with electron-capture detection. The degradate dichloroacetic acid was isolated in the pH 1 diethyl ether extracts. Aliquots of the ether extract were analyzed by one-dimensional TLC on silica gel plates developed in ethyl acetate:methanol:glacial acetic acid (180:50:3, v:v:v); radioactive areas were detected and identified as described above.

DATA SUMMARY:

Tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl-O-(1,2,2,2-tetrachloroethyl) ester; radiochemical purity 97%], at 1 ppm, photodegraded with a registrant-calculated half-life of 27 days in sterile aqueous 10 mM sodium acetate buffer solutions (pH 5) containing 1% acetonitrile that were continuously irradiated with a UV-filtered xenon arc lamp at 25 $\pm$ 1 C for 15 days. The intensity of the lamp (total radiant intensity was not reported) was reported to be 1.67 times greater than that of midday sunlight in June at Huntingdon, England (52°21'N, 0°15'W; Figure 1). In contrast, [^{14}C]chlorethoxyphos degraded with a calculated half-life of 89 days when incubated in the dark under similar conditions. The major degradate identified in both the irradiated and dark control solutions was

trichloroacetaldehyde.

In the irradiated solutions, [^{14}C]chlorethoxyphos comprised 97-100% of the applied radioactivity immediately posttreatment and 66-79% at 15 days posttreatment (Table III in study text and Table IV in Appendix 3). At 15 days posttreatment, trichloroacetaldehyde comprised an average of 18% of the applied radioactivity;

dichloroacetic acid

comprised an average of 3%, and two unidentified [^{14}C]compounds Components A and D comprised averages of 1 and 2%, respectively. In the dark control solutions at 15 days posttreatment, chlorethoxyphos comprised 88-90% of the applied radioactivity, trichloroacetaldehyde comprised an average of 7%, dichloroacetic acid comprised an average of 2%, and Components A and D each comprised an average of 1% (Table IV in study text and Table IV in Appendix 3). During the study, material balances ranged from 92 to 102% of the applied in the irradiated solutions and 99 to 103% in the dark control solutions (Tables I and II).

COMMENTS:

1. The concentrations of the degradates, trichloroacetaldehyde and dichloroacetic acid, and the unidentified [^{14}C]compounds were presented as the average of two replicates that were collected at each sampling interval. The results from analysis of each test solution should have been presented to provide the maximum concentrations of the degradates and unidentified [^{14}C]compounds that were detected at each sampling interval, and to assess the variability between the replicates.

2. The study authors reported that the intensity of the light emitted by the xenon lamp was 1.67 times greater than that the intensity of natural sunlight at 290-400 nm; however, the measured intensities of the xenon light and natural sunlight at those wavelengths were not reported in a tabular form for comparison. In addition, the total irradiant intensity received by the test solutions was not reported. A complete wavelength distribution and total irradiant intensity of the xenon light source were not compared to natural sunlight; comparisons were only made at wavelengths of 290-400 nm for intensity and 290-500 nm for wavelength distribution.

3. The photolysis apparatus was not adequately described. The study authors reported that flat-topped, borosilicate glass vessels (diameter 3 cm, height 8.5 cm) containing the irradiated test solutions were placed in individual sockets of a water-cooled steel block in the photolysis apparatus. It was not reported how much of each vessel was exposed (other than the flat top) and how much of the vessel was encased in the steel block. A diagram of the photolysis apparatus was not provided. It was also unclear how the solutions were magnetically stirred within a steel block.

4. The study authors reported that the irradiated solutions were maintained at 25 $\pm$ 1 C using a water-cooled steel block and the dark controls were maintained at 25 $\pm$ 1 C in an incubator. The temperature of the irradiated solutions was measured using an indwelling thermocouple probe positioned in a test vessel containing untreated buffer solution that was located in a central socket of the steel block; the actual recorded temperatures that occurred during the study were not reported. In addition, the study authors reported in the Summary section of the study text that the dark control

solutions were incubated alongside the irradiated solutions; however, in the Experimental section the study authors reported that the dark control solutions were transferred to an incubator and the irradiated solutions were placed in the Suntest unit which contained the xenon lamp.

5. The study authors reported that the pH of the test solutions remained constant during the study; actual measured pH values were not reported.

6. An absorption spectrum of the pesticide in the test solution was not provided. An absorption spectrum of chlorethoxyphos in acetonitrile was provided in a photodegradation on soil study (Study 2, MRID 41736822) submitted concurrently with this study document; the spectrum indicates that chlorethoxyphos absorbs light primarily at wavelengths <290 nm (Figure 13; Study 2, MRID 41736822).

7. The solubility of chlorethoxyphos in water at 25 C was reported to be 2.1 ppm.

8. To calculate the degradation half-lives of chlorethoxyphos, the study authors used "robust iteratively re-weighted least squares" regression analysis instead of first-order linear regression (Page 62). The statistical estimation of the photodegradation half-lives of chlorethoxyphos calculated by the study authors (27 days in the irradiated solutions and 89 days in the dark controls) are of limited value because the calculations involve extrapolation considerably beyond the experimental time limits of the study. Data are often incapable of accurately predicting trends outside of their range because small differences are magnified and reactions which appear to be linear may, in fact, be curvilinear.

9. To assess whether the solutions remained sterile during the study, additional aliquots of the sterile pH 5 buffer solution were transferred to 26 tubes, treated at 1 ppm with tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos, and incubated at 25 $\pm$ 1 C in darkness. Eleven of the tubes were sampled immediately posttreatment and the remaining 15 tubes at 15 days posttreatment. Aliquots of the test solutions were used to prepare Tryptone Soya Agar plates that were incubated at 30 C for 5 days, then analyzed for bacterial or fungal growth; no bacterial or fungal growth was observed.

10. A study investigating the photodegradation of chlorethoxyphos labeled in the phosphoryl moiety of the compound in sterile aqueous buffered solution (pH 5) may be required.

11. The registrant reported that chlorethoxyphos (DPX-43898) is an organophosphorus insecticide used for control of corn root worms and other soil insects and is currently being developed for use on corn. The proposed use rate was reported as 0.3 oz ai/1000 feet of row (0.25 lb ai/A assuming 40-inch row spacing; MRID 41736820).

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DATA EVALUATION RECORD

STUDY 2

CHEM 129006	Chlorethoxyphos (DPX-43898)	§161-3

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41736822
Bramble, Jr., F.Q., and A.C. Barefoot. 1989. Photodegradation of ^{14}C-labeled DPX-43898 on soil (conducted in sunlight). Revision No. 1. Report No. AMR-795-87. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

DIRECT REVIEW TIME = 21

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CONCLUSIONS:

Photodegradation on Soil

1. Study MRID #41736822 is acceptable and completely satisfies the photodegradation on soil (161-3) data requirement for chlorethoxyphos.

2. Chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester] degraded with a half-life of 21 days on sandy loam soil incubated in natural sunlight at 22-28 C for 32 days. It appears degradation took place through processes other than direct phototransformation since in the dark control chlorethoxyphos degraded with a half-life of 26 days. The major

nonvolatile degradate identified in both the irradiated and dark control soil was trichloroacetaldehyde; tentatively identified were trichloroacetic acid and dichloroacetic acid. In the irradiated soil at 32 days posttreatment, $^{14}CO_2$ was the major degradate and totaled 27.2% of the applied radioactivity.

METHODOLOGY:

Samples (approximately 2 g) of sieved (2 mm), air-dried sandy loam soil (70.4% sand, 20.4% silt, 9.2% clay, 1.0% organic matter, pH 5.4, CEC 7.2 meq/100 g) were spread on the sidewall (area 9 cm^2) of eight culture flasks and treated at 0.4 lb ai/A (approximately 20 ppm) with tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; radiochemical purity 98.5%, specific activity 21.6 uCi/mg, Du Pont NEN Products] dissolved in hexane. The flasks of treated soil were sealed and incubated outdoors in natural sunlight in Wilmington, Delaware (39°40'N, 75°36'W), from September 11 through October 13, 1987. During the study, the radiant energy received by the irradiated soil totaled 110730 watt-hour/m^2 at wavelengths of 285-2800 nm (Table II); total radiant energy received by the irradiated soil was continuously monitored using a black and white pyranometer connected to an electronic integrator. The flasks were embedded in a conductive paste on a heat exchanger that maintained the soil temperature at 22-28 C (mean 24 C) using a circulating water bath (Figure 3). Ten additional samples of sandy loam soil were placed in culture tubes, treated with [^{14}C]chlorethoxyphos as described above, sealed, and maintained at 25 $\pm$ 1 C in darkness in an incubator to serve as dark controls; two of the treated soil samples were taken immediately for time 0 samples. Duplicate irradiated and dark control soil samples were collected at 7, 14, 21, and 32 days posttreatment. At each sampling interval prior to opening the sample containers, the air was drawn out of each container by vacuum, then sequentially through methanol cooled in a dry ice/acetone bath to trap volatiles and scintillation cocktail to trap evolved CO_2 (Figure 4).

Soil samples were extracted three times with methanol:water (9:1, v:v) using ultrasonication; after each extraction, the extracts were separated from the soil by centrifugation. The extracts from each sample were combined, and aliquots (100 uL) were analyzed for total radioactivity using LSC. Additional aliquots (50 uL) were analyzed by HPLC on a Zorbax ODS column eluted with a gradient mobile phase of water and acetonitrile; fractions were collected at 0.5-minute intervals and analyzed for total radioactivity by LSC. Radioactive peaks were identified by comparison to the retention times of radiolabeled reference standards of chlorethoxyphos, trichloroacetaldehyde (chloral), dichloroacetic acid, and trichloroacetic acid. Aliquots of the soil extracts were also analyzed by one-dimensional TLC on silica gel plates developed in ethyl acetate:methanol:acetic acid (80:20:1, v:v:v). Radioactive areas were visualized using a TLC linear analyzer and identified by

comparison with radiolabeled reference standards that had been chromatographed with the samples. The extracted soils were air-dried, then analyzed for unextracted [^{14}C]residues using LSC following combustion.

To confirm the identification of the degradate trichloroacetaldehyde, an aliquot of the 32-day dark control soil extract was filtered and concentrated. The concentrate was applied to a C-18 column, then the column was washed with acidified water (pH 1); trichloroacetaldehyde was eluted from the column with ether. The eluate was concentrated and analyzed using GC/MS with electron ionization.

Aliquots of the trapping solutions were analyzed for total radioactivity using LSC.

DATA SUMMARY:

Tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; radiochemical purity 98.5%], at 0.4 lb ai/A (approximately 20 ppm), degraded with a registrant-calculated half-life of 20.9 days on sandy loam soil that was irradiated with sunlight outdoors (39°40'N, 75°36'W) in early fall for 32 days at 22-28 C (Table V and Figure 11). [^{14}C]Chlorethoxyphos degraded with a calculated half-life of 25.8 days on soil incubated in the dark. During the study, the radiant energy received by the irradiated soil totaled 110730 watt-hour/m^2 at wavelengths of 285-2800 nm (Table II). The major nonvolatile degradate identified in both the irradiated and dark control soil samples was

trichloroacetaldehyde (chloral).

In the irradiated soil, [^{14}C]chlorethoxyphos decreased from an average of 101.1% of the applied radioactivity immediately posttreatment to 58.1% after 14 days, 46.1% after 21 days, and 34.5% after 32 days. After 32 days of irradiation, trichloroacetaldehyde comprised 17.2% of the applied (maximum of 22.1% at 21 days) and polar [^{14}C]compounds comprised 6.7% (maximum of 7.2% at 21 days; Table IV). One-dimensional TLC analysis of the 32-day irradiated soil extract determined that the polar compounds consisted primarily of two compounds tentatively identified as

trichloroacetic acid and

dichloroacetic acid

(Figure 7). Also after 32 days of irradiation, unextracted [^{14}C]residues in the soil comprised an average of 3.3% of the applied, evolved $^{14}CO_2$ totaled 27.2%, and other [^{14}C]volatiles totaled 0.9% (Table III). During the study, material balances decreased from an average of 103.0 to 89.8% of the applied in the irradiated soil.

In the dark control soil at 32 days posttreatment, chlorethoxyphos comprised an average of 41.6% of the applied radioactivity, trichloroacetaldehyde comprised 43.3%, extractable polar [^{14}C]compounds comprised 11.1%, unextracted [^{14}C]residues comprised 2.2%, $^{14}CO_2$ totaled 0.5%, and other [^{14}C]volatiles totaled 0.1% (Table III). During the study, material balances ranged from 96.5 to 98.7% of the applied in the dark control soil.

COMMENTS:

1. The study authors concluded that chlorethoxyphos did not significantly photodegrade on soil as indicated by the degradation half-lives of 20.9 and 25.8 days in the irradiated and dark control soils, respectively. However, the nonvolatile degradate trichloroacetaldehyde did appear to photodegrade to CO_2 (Figure 16).

2. All results, including concentrations of parent chlorethoxyphos, the degradate trichloroacetaldehyde, the tentatively identified polar [^{14}C]compounds, evolved $^{14}CO_2$ and volatiles, unextractable [^{14}C]residues, and material balances, were presented by the study authors as the average of the two replicates that were collected at each sampling interval. The results from analysis of each soil sample should have been presented to provide the maximum concentrations of the parent compound, degradates, and tentatively identified [^{14}C]compounds that were detected at each sampling interval, and to assess the variability between the replicates.

3. Unidentified [^{14}C]compounds described as "Polars" in Table IV totaled 7.2 and 11.1% of the applied radioactivity in the irradiated and dark control soil extracts, respectively. The study authors reported that one-dimensional TLC analysis determined that the polar [^{14}C]compounds consisted primarily of two compounds tentatively identified as trichloroacetic acid and dichloroacetic acid, which were present in approximately equal concentrations and neither exceeded 6.2% of the applied radioactivity. A sample TLC radiochromatogram from analysis of the 32-day irradiated soil extract was provided (Figure 7).

4. The hours of sunlight per day during the study were not reported. Daily atmospheric cover is presented in Appendix II.

5. The study authors reported that the irradiated soil samples were maintained at 22-28 C (mean 24 C) using an heat exchanger cooled with a circulating water bath. The temperature of the irradiated soil samples was measured with a thermocouple embedded in a separate soil sample (Figure 3). The actual recorded temperatures that occurred during the study were not reported.

6. An absorption spectrum of chlorethoxyphos in acetonitrile is presented in Figure 13; the spectrum indicates that chlorethoxyphos absorbs light primarily at wavelengths <290 nm.

7. For HPLC analysis of the soil extracts, radioactive peaks were reconstructed following LSC analysis of fractions collected at 0.5-minute intervals, then identified by comparison to the retention times of [^{14}C]-labeled reference standards. It is preferred that either UV and/or in-line radioactivity detection be used in addition to fraction collection for HPLC analysis.

8. A study investigating the photodegradation of chlorethoxyphos labeled in the phosphoryl moiety of the compound on soil may be required.

9. The registrant reported that chlorethoxyphos (DPX-43898) is an organophosphorus insecticide used for control of corn root worms and other soil insects and is currently being developed for use on corn. The proposed use rate was reported as 0.3 oz ai/1000 feet of row (0.25 lb ai/A assuming 40-inch row spacing; MRID 41736820). The study authors reported that the treatment rate of 0.4 lb ai/A used in this study approximates the expected maximum field use rate.

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Pages 29 through 42 are not included.

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DATA EVALUATION RECORD

STUDY 3

CHEM 129006 | Chlorethoxyphos (DPX-43898) | §161-4

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41736823
Dykes, J. 1990. Photodegradation of DPX-43898 in air. ABC Report No. 38363; Du Pont Report No. AMR-1535-89. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Columbia, MO, and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

DIRECT REVIEW TIME = 25

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SIGNATURE:

CONCLUSIONS:

Photodegradation in Air

1. Study MRID #41736823 is unacceptable and does not satisfy the photodegradation in air (161-4) data requirement for chlorethoxyphos for the following reasons:

a) material balances were incomplete; up to 40.4% of the applied radioactivity in the irradiated air samples and 37.2% in the dark controls was not accounted for.

b) photodegradation of the test substance in the air could not be distinguished from photodegradation of the test substance that had

condensed on the sides of the photoreactor vessel or that had not volatilized.

2. Since the material balances were incomplete and the study was not designed to distinguish photodegradation in air from photodegradation on glass surfaces, the problems with this study cannot be resolved with the submission of additional data. A new photodegradation in air study should not be initiated until the technical issues with the photodegradation in air study are resolved. Thus, the data requirement will be placed in a reserved status.

METHODOLOGY:

Aliquots (100 uL) of tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; radiochemical purity >98%, specific activity 58.4 uCi/mg, Du Pont NEN Products], dissolved in hexane, were transferred to 72-L borosilicate glass vessels using a syringe; approximately 0.109 mg of [^{14}C]chlorethoxyphos was added to each vessel. The vessels had been equilibrated overnight with ambient air at 30 C prior to treatment. An additional aliquot (100 uL) of the [^{14}C]chlorethoxyphos test solution was diluted with methanol (250 mL) and analyzed for total radioactivity by LSC to determine the treatment rate. The opening of each vessel was sealed with a borosilicate glass plate, then placed in a photoreactor that shielded the main body of the vessel so that light would only enter through the 12.8-cm diameter opening (Figure 3). The photoreactor was placed in a photolysis apparatus, and the samples were continuously irradiated using a xenon arc lamp equipped with dual borosilicate filters to eliminate radiation below 290 nm. The measured intensity of the light source at the sample distance (83 cm) was 0.705996-35.35109 x 10^{19} photons/cm^2·day at 310-740 nm; it was reported that the intensity of the xenon lamp was approximately one-half that of natural sunlight at summer solstice (40°N) at 310-400 nm (Table I). The vessels were maintained at 30 $\pm$ 2 C during irradiation. Additional 72-L vessels were treated with [^{14}C]chlorethoxyphos as described above, sealed, and maintained at 30 $\pm$ 2 C in darkness in an environmental chamber to serve as dark controls; two treated vessels were analyzed immediately for time 0 samples. Duplicate irradiated and dark control vessels were collected at 7, 14, and 30 days posttreatment.

Upon sampling, each vessel was placed in a freezer (-22 C) overnight to condense any volatilized chlorethoxyphos and its degradates onto the walls of the vessel. Following condensation, methanol (250 mL) was added to each vessel which was then resealed, swirled, and allowed to settle (time interval not specified); the solution was swirled and allowed to settle three times. Aliquots of each solution were analyzed for total radioactivity using LSC. A subsample (5-10 mL) of the solution was stored (time interval unspecified) frozen (-22 C) until HPLC analysis. Aliquots (120 uL) were analyzed by reverse-phase HPLC using a PRP-1 column eluted with a gradient mobile

phase combining deionized water plus 0.1% phosphoric acid:acetonitrile; fractions were collected at 0.5-minute intervals and analyzed for total radioactivity by LSC. Radioactive peaks were identified by comparison to the retention time of a reference standard of chlorethoxyphos; it was not reported how the reference standard was detected.

DATA SUMMARY:

Tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; radiochemical purity >98%], at a nominal concentration of 1 ug/L, appeared to slowly photodegrade with a registrant-calculated half-life of 198 days in air that was continuously irradiated with a dual-filtered xenon arc lamp at 30 ± 2 C for 30 days (Table IV). The intensity of the lamp, 0.705996-35.35109 x 10^{19} photons/cm^2·day at 310-740 nm, was reported to be approximately one-half that of natural sunlight at summer solstice (40° N) at 310-400 nm. It was determined that 24 hours of irradiation with the xenon lamp was equivalent to 10.8 hours of natural sunlight; however, the study author estimated that only about 10% of the volume of the photoreactor vessel was exposed to irradiation. In contrast, [^{14}C]chlorethoxyphos decreased in air samples from the dark control from 98.1-98.9% of the recovered immediately posttreatment to 91.9-93.0% at 30 days (Table III). In the irradiated air samples at 30 days posttreatment, chlorethoxyphos was the only compound identified and comprised 86.7-88.0% of the recovered radioactivity; two unidentified [^{14}C]compounds A and B comprised 4.6-6.1 and 2.7-3.1%, respectively, and additional (number unspecified) unidentified [^{14}C]compounds described as "Other" totaled 4.1-4.7%. During the study, material balances decreased from 98.6-100.3% of the applied at day 0 posttreatment to 59.6-68.1% of the applied in the irradiated air samples and 62.8-78.6% in the dark control air samples at 30 days (Table II).

COMMENTS:

1. The material balances decreased throughout the study with 31.9-40.4% of the applied radioactivity unaccounted for in the irradiated air samples and 21.4-37.2% in the dark controls by 30 days posttreatment. The study author reported that attempts to extract additional [^{14}C]material from the walls of the photoreactor vessels using methanol, acetonitrile, ethyl acetate, and hexane were unsuccessful. The study author believed that parent chlorethoxyphos had adsorbed to both the glass photoreactor vessels and the Teflon gaskets used to seal the borosilicate glass plate to the photoreactor vessel. It was not adequately demonstrated that the loss of [^{14}C]material was due to adsorption of parent chlorethoxyphos to the glass photoreactor vessels and Teflon gaskets. In studies submitted concurrently with this study, there was no loss of [^{14}C]material in either the photodegradation in water study (Study 1, MRID 41736821), in which aqueous buffered (pH 5) solutions of chlorethoxyphos were incubated in borosilicate glass vessels and tubes, or in the aerobic soil

metabolism study (Study 4, MRID 41736824), where Teflon tubing was specifically used in places of the metabolism apparatus that would come in contact with chlorethoxyphos or volatile degradates.

2. The study was not a true photodegradation in air study because of the sampling procedures used. The air was not directly sampled; therefore, photodegradation of the test substance in the air could not be distinguished from photodegradation of the test substance that had condensed on the sides of the photoreactor vessel or that had not volatilized. Following irradiation or incubation in darkness, the flasks were placed in the freezer to condense any volatilized chlorethoxyphos and its degradates onto the walls of the photoreactor vessel, then the condensate was rinsed from the vessel using methanol and analyzed.

3. The study author reported that the photoapparatus (Figure 3) was a simplified version of the photoreactor proposed by Crosby (Crosby, D.G. and K.W. Moilanen. 1974. "Vapor-phase photodecomposition of aldrin and dieldrin" Archives of Environmental Contamination and Toxicology 2:62-74) and was chosen to minimize catalytic effects of surfaces. However, the study author estimated that only about 10% of the volume of the photoreactor vessel was exposed to irradiation; therefore, the rate constant calculated for the photodegradation of chlorethoxyphos in air may be underestimated and the half-life may be overestimated by an order of magnitude. The study author reported that the calculated rate constant and half-life apply only to photodegradation of chlorethoxyphos in the photoapparatus and cannot be used directly in modeling atmospheric photodegradation rates; however, the study author also estimated a theoretical half-life of <1 month in natural sunlight after compensation for the intensity of the xenon lamp and the proportion of the photoreactor vessel that was exposed to irradiation.

4. The study author incorrectly interpreted the absorption spectrum of chlorethoxyphos that is provided in Figure 1. The study author concluded that chlorethoxyphos would not photodegrade via direct phototransformation because the absorption spectrum of chlorethoxyphos in acetonitrile showed that chlorethoxyphos did not absorb light at wavelengths >220 nm (page 16). However, the handwritten memo at the bottom of the chromatogram instructs that the top wavelength scale (210-800 nm) be used to interpret the results, not the bottom wavelength scale (190-500 nm). Using the top scale, the spectrum indicates that chlorethoxyphos can absorb light up to approximately 320 nm, but absorbs light primarily at wavelengths <290 nm. This absorption spectrum of chlorethoxyphos was also provided as Figure 13 in the photodegradation on soil study (Study 2, MRID 41736822).

5. This study was not a single continuous experiment, but rather a series of small experiments that were combined. For each sampling interval (except day 0 posttreatment), four 72-L photoreactor vessels

were treated with [^{14}C]chlorethoxyphos, then two were irradiated and two were incubated in darkness.

6. The study author reported that the irradiated vessels and dark controls were maintained at 30 ± 2 C; however, the actual recorded temperatures that occurred during the study were not reported.

7. For HPLC analysis of the solutions, radioactive peaks were reconstructed following LSC analysis of fractions collected at 0.5-minute intervals, then identified by comparison to the retention times of reference standard chlorethoxyphos. It is preferred that either UV and/or in-line radioactivity detection be used in addition to fraction collection for HPLC analysis. It was unclear whether a labeled or unlabeled reference standard of chlorethoxyphos was used to establish the retention time.

8. The vapor pressure of chlorethoxyphos was reported to be 10^{-5} torr. (unspecified conditions).

9. The study author reported that the unknown A was "similar chromatographically" (page 19) to trichloroacetaldehyde (chloral hydrate) observed in the aqueous photodegradation study (Study 1; MRID 41736821); however, no characterization of this degradate was included.

9. The statistical estimation of the photodegradation half-life of chlorethoxyphos calculated by the study author (198 days) is of limited value because the calculations involve extrapolation considerably beyond the experimental time limits of the study. Data are often incapable of accurately predicting trends outside of their range because small differences are magnified and reactions which appear to be linear may, in fact, be curvilinear.

10. A study investigating the photodegradation of chlorethoxyphos labeled in the phosphoryl moiety of the compound in air may be required.

11. The registrant reported that chlorethoxyphos (DPX-43898) is an organophosphorus insecticide used for control of corn root worms and other soil insects and is currently being developed for use on corn. The proposed use rate was reported as 0.3 oz ai/1000 feet of row (0.25 lb ai/A assuming 40-inch row spacing; MRID 41736820).

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Pages 49 through 60 are not included.

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____ Description of the product manufacturing process.

____ Description of quality control procedures.

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____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

X FIFRA registration data.

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DATA EVALUATION RECORD

STUDY 4

CHEM 129006 | Chlorethoxyphos (DPX-43898) | §162-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41736824
Woodward, M.D., and C.M. Pukalski. 1990a. Aerobic metabolism of ^{14}C-DPX-43898 in soil. Report No. AMR-1457-89. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

DIRECT REVIEW TIME = 43

REVIEWED BY: L. Binari TITLE: Staff Scientist

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SIGNATURE: 

CONCLUSIONS:

Aerobic Soil Metabolism

1. Study MRID #41736824 may be used as ancillary data to the previously reviewed acceptable aerobic soil metabolism study. The aerobic soil metabolism (162-1) data requirement has been satisfied by a previously submitted and reviewed study MRID #40883705 (EFGWB #90-0067, 2/21/90).

a) The study authors reported that the organic [^{14}C]volatiles, which totaled 15.0% of the applied radioactivity (0.075 ppm) at 120 days posttreatment, were shown to consist of "mostly" parent chlorethoxyphos; however, it was not described how the ethylene

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glycol trapping solutions were analyzed for chlorethoxyphos, and quantitative results were not provided.

2. Chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester] degraded with a half-life of approximately 23 days in clay soil that was incubated in the dark at 25 C and 75% of field moisture capacity. Only chlorethoxyphos was identified in the soil; carbon dioxide was the major degradate and totaled 63.0% of the applied at 120 days posttreatment.

METHODOLOGY:

Samples of moist, sieved (2 mm) clay soil (20% sand, 35% silt, 44% clay, 4.2% organic matter, pH 6.2, CEC 33.6 meq/100 g) were weighed (58.65 g) into flasks, treated at 0.5 ppm with tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; radiochemical purity >99%, specific activity 58.4 uCi/mg, Du Pont NEN Products] dissolved in acetone, and moistened to 75% of field capacity. The flasks of treated soil were attached to a dynamic air-flow system; air (2-6 mL/minute) was continuously drawn through the flasks, then sequentially through an ethylene glycol solution to trap organic volatiles and an ethanolamine solution to trap evolved CO_2 (Figure 4). The treated soil was incubated in darkness at approximately 25 C and a relative humidity of approximately 50%. To maintain the soil moisture content, the flasks were periodically weighed and water was added, as needed, to adjust the flasks to the original weight. Duplicate flasks of soil and the corresponding trapping solutions were collected at 0, 2, 5, 7, 14, 19, 25, 30, 49, 61, 81, 100, and 120 days posttreatment. Soil samples were stored frozen for up to 2 weeks prior to extraction.

Soil samples were extracted twice with acetone:water (9:1, v:v), then once with acetone using a wrist-action shaker for 1 hour per extraction. The extracts were combined, concentrated, and centrifuged. The aqueous acetone supernatant was decanted, and the remaining particulate material was dissolved in acetone. Aliquots of the supernatant and the acetone-soluble material were analyzed for total radioactivity using LSC. Additional aliquots of each solution were analyzed by reverse-phase HPLC using a PRP-1 column eluted with a gradient mobile phase of acidified water (adjusted to pH 2.2 with phosphoric acid):acetonitrile; fractions were collected at 0.5-minute intervals and analyzed for total radioactivity by LSC. Radioactive peaks were identified by comparison to the retention time of a reference standard of chlorethoxyphos; it was not reported how the reference standard was detected.

To confirm identification of parent chlorethoxyphos, an aliquot of the 19-day soil aqueous acetone supernatant was partitioned with hexane. The hexane phase was concentrated and analyzed by one-dimensional TLC on silica gel plates developed with hexane:ethyl acetate (9:1, v:v). Radioactive areas were visualized using a TLC

linear analyzer and by autoradiography, quantified using the linear analyzer, and identified by comparison with a labeled reference standard of chlorethoxyphos that was cochromatographed with the samples. An additional aliquot of the concentrated hexane phase was analyzed using GC/MS with selected ion monitoring.

Unextracted [^{14}C]residues remaining in the extracted soil were quantified using LSC following combustion. The extracted 120-day soil samples were further extracted with hexane, ethyl acetate, chloroform, acetone, methanol, acetonitrile, water, and 0.1 N hydrochloric acid; however, $\leq$1.7% of the applied radioactivity was removed from the soil by any of these solvents (Table V). In a further attempt to characterize the unextracted [^{14}C]residues, the 120-day extracted soil samples were reextracted with 0.5 M NaOH, and the extract was precipitated with acid, differentiating the [^{14}C]residues into humin, humic acid, and fulvic acid fractions.

Aliquots of the trapping solutions were analyzed for total radioactivity using LSC.

DATA SUMMARY:

Tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; radiochemical purity >99%], at 0.5 ppm, degraded with a registrant-calculated half-life of approximately 23 days in clay soil that was incubated in the dark at approximately 25 C and 75% of field moisture capacity for 120 days. Chlorethoxyphos decreased from an average of 98.4% of the applied radioactivity at 0 days posttreatment to 48.4% at 14 days, 35.5% at 30 days, 13.0% at 61 days, and 6.6% at 120 days (Table IV). Unidentified [^{14}C]compounds described as "others" were detected at an average maximum of 3.6% of the applied (0.018 ppm) at 2 days posttreatment; it was reported that the "others" consisted of four or five compounds, each at <2% (0.01 ppm).

At 120 days posttreatment, evolved $^{14}CO_2$ totaled an average of 63.0% of the applied, and organic [^{14}C]volatiles (reported as consisting of "mostly" parent chlorethoxyphos) totaled 15.0% (Table III). Unextracted [^{14}C]residues totaled a maximum average of 20.9% of the applied radioactivity at 100 days posttreatment, and were 17.2% at 120 days. Of the unextracted [^{14}C]residues remaining in a 120-day soil sample after the final extraction, 6.0% of the applied was characterized as humin, 4.5% as humic acid, and 9.1% as fulvic acid (Figure 15). During the study, average material balances ranged from 93.2 to 106.1% of the applied.

COMMENTS:

1. The study authors reported that the organic [^{14}C]volatiles, which totaled 15.0% of the applied radioactivity (0.075 ppm) at 120 days

posttreatment, were shown to consist of "mostly" parent chlorethoxyphos; however, it was not described how the ethylene glycol trapping solutions were analyzed for chlorethoxyphos, and quantitative results were not provided. Subdivision N guidelines state that all degradates present at >0.01 ppm (approximately 2% of the applied) mast be identified. The registrant must provide the actual concentrations of parent chlorethoxyphos that were detected as organic volatiles and specify the concentrations of other volatilized [^{14}C]compounds that were present; the study authors implied that these data were generated during the study.

2. All results, including concentrations of parent chlorethoxyphos, unidentified [^{14}C]compounds, evolved $^{14}CO_2$ and volatiles, unextractable [^{14}C]residues, and material balances, were presented as the average of the two replicates that were collected at each sampling interval. The results from analysis of each soil sample should have been presented to provide the maximum concentrations of the parent compound, degradates, and unidentified [^{14}C]compounds that were detected at each sampling interval, and to assess the variability between the replicates.

3. One-dimensional TLC analysis of the 19-day soil extract determined that the polar compound consisted primarily of chlorethoxyphos (Figure 11).

4. The study authors classified the test soil as a La Hogue clay loam; however, using the USDA soil classification system, the percentages of sand, silt, and clay classified the test soil as a clay soil, and the soil was referred to as such in this review (Table II). The percentages of sand, silt, and clay totaled 99%, rather than 100%; however, this did not affect the outcome of the soil classification. The study authors also reported that the test soil was chosen because it is typical of the Midwest corn belt, and the higher organic content (4.2%) would reduce loss of chlorethoxyphos through vaporization.

5. The registrant reported that chlorethoxyphos (DPX-43898) is an organophosphorus insecticide used for control of corn root worms and other soil insects and is currently being developed for use on corn. The proposed use rate was reported as 0.3 oz ai/1000 feet of row (0.25 lb ai/A assuming 40-inch row spacing; MRID 41736820). The study authors reported that the treatment rate of 0.5 ppm used in this study was twice the proposed use rate of chlorethoxyphos (0.25 lb ai/A).

6. In an anaerobic soil metabolism experiment (Study 5, MRID 41736825) submitted concurrently with this study, chlorethoxyphos degraded with half-lives of 41-47 days in clay soil that was incubated anaerobically (flooding plus nitrogen atmosphere) in the dark at 25 C for 62 days following 19 days of aerobic incubation. Only chlorethoxyphos was identified in the soil and floodwater; carbon

dioxide was the major degradate and totaled 36.5% of the applied at the termination of the experiment.

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Pages 67 through 78 are not included.

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DATA EVALUATION RECORD

STUDY 5

CHEM 129006 Chlorethoxyphos (DPX-43898) §162-2

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41736825
Woodward, M.D. and C.M. Pukalski. 1990b. Anaerobic metabolism of ^{14}C-DPX-43898 in soil. Report No. AMR-1456-89. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

DIRECT REVIEW TIME = 29

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APPROVED BY: K. Poff
TITLE: Chemist
ORG: EFGWB/EFED/OPP

SIGNATURE: Kurt Poff

CONCLUSIONS:

1. Study MRID #41736825 may be used as supplemental data until the additional data to upgrade study MRID #41736825 is submitted and reviewed.

2. Chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester] degraded with half-lives of 41-47 days in clay soil that was incubated anaerobically (flooding plus nitrogen atmosphere) in the dark at 25 C for 62 days following 19 days of aerobic incubation. Only chlorethoxyphos was identified in the soil and floodwater; carbon dioxide was the major degradate and totaled 36.5% of the applied at the termination of the experiment.

METHODOLOGY:

Samples of moist, sieved (2 mm) clay soil (20% sand, 35% silt, 44% clay, 4.2% organic matter, pH 6.2, CEC 33.6 meq/100 g) were weighed (58.65 g) into flasks, treated at 0.5 ppm with tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; radiochemical purity >99%, specific activity 58.4 uCi/mg, Du Pont NEN Products] dissolved in acetone, and moistened to 75% of field capacity. The flasks of treated soil were attached to a dynamic air-flow system; air was continuously drawn (2-6 mL/minute) through the flasks, then sequentially through an ethylene glycol solution to trap organic volatiles and an ethanolamine solution to trap evolved CO_2 (Figure 4). The treated soil was incubated in darkness at 23-26 C for 19 days. To maintain the soil moisture content, the flasks were periodically weighed and water was added, as needed, to adjust the flasks to the original weight. At 19 days posttreatment, anaerobic conditions were established in half of the remaining flasks by flooding the soil with distilled water (100 mL) and purging the flasks with humidified nitrogen for 1 day. The anaerobic soil flasks were sealed and incubated at approximately 25 C in darkness. Duplicate aerobic soil flasks and the corresponding trapping solutions were collected at 0, 19, 49, and 81 days posttreatment. Anaerobic soil flasks were collected at 30 and 62 days after anaerobic conditions were established (49 and 81 days posttreatment); volatiles were collected at the same sampling intervals by flushing air through the flask, then sequentially through ethanolamine trapping solution, a combustion furnace (for volatiles gases such as methane), and a second ethanolamine trap. Aerobic soil samples were stored frozen for up to 2 weeks prior to extraction; it was not specified how long anaerobic soil:water samples were stored prior to extraction.

Water fractions were separated from the soil using a pipette, centrifuged, and aliquots were analyzed for total radioactivity using LSC. Soil samples were extracted twice with acetone:water (9:1, v:v) followed by once with acetone using a wrist-action shaker for 1 hour per extraction. The extracts were combined, concentrated, and centrifuged. The aqueous acetone supernatant was decanted, and the remaining particulate material was dissolved in acetone. Aliquots of the supernatant and acetone-soluble material were analyzed for total radioactivity using LSC. Additional aliquots of each solution were analyzed by reverse-phase HPLC using a PRP-1 column eluted with a gradient mobile phase of acidified water (adjusted to pH 2.2 with phosphoric acid):acetonitrile; fractions were collected at 0.5-minute intervals and analyzed for total radioactivity by LSC. Radioactive peaks were identified by comparison to the retention time of a reference standard of chlorethoxyphos; it was not reported how the reference standard was detected.

To confirm identification of parent chlorethoxyphos, an aliquot of the 62-day anaerobic (81 days posttreatment) soil aqueous acetone supernatant was partitioned with hexane. The hexane phase was concentrated and analyzed by one-dimensional TLC on silica gel plates developed with hexane:ethyl acetate (9:1, v:v). Radioactive areas were visualized using a TLC linear analyzer and by autoradiography, quantified using the linear analyzer, and identified by comparison with a labeled reference standard of chlorethoxyphos that was cochromatographed with the samples. An additional aliquot of the concentrated hexane phase was analyzed using GC/MS with selected ion monitoring.

Unextracted [^{14}C]residues remaining in the extracted soil were quantified using LSC following combustion. The extracted 81-day aerobic soil and 62-day anaerobic (81 days posttreatment) soil samples were further extracted with hexane, ethyl acetate, chloroform, acetone, methanol, acetonitrile, water, and 0.1 N hydrochloric acid; however, $\leq$1.9% of the applied radioactivity was removed from the soil by any of these solvents (Table V). In a further attempt to characterize the unextracted [^{14}C]residues, the extracted 62-day anaerobic soil samples were reextracted with 0.5 M NaOH, and the extract was precipitated with acid, differentiating the [^{14}C]residues into humin, humic acid, and fulvic acid fractions.

Aliquots of the trapping solutions were analyzed for total radioactivity using LSC.

A supplemental experiment was performed to account for low material balances that occurred during the anaerobic phase of the initial experiment described above (refer to Appendix II). Samples of clay soil were weighed into flasks, treated at a nominal concentration of 0.5 ppm with tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos, and moistened to 75% of field capacity as described above. The flasks of treated soil were attached to a dynamic air-flow system, but the air flow was increased to 30 mL/minute; organic volatiles and evolved CO_2 were trapped as described above. The treated soil was incubated in darkness at approximately 25 C for 19 days, then anaerobic conditions were established in the remaining flasks by flooding the soil with water and purging the flasks with humidified nitrogen. The anaerobic soil flasks were sealed and incubated at approximately 25 C in darkness. Duplicate flasks were collected at 0 and 19 days posttreatment and at 30 and 62 days after anaerobic conditions were established (49 and 81 days posttreatment). Volatiles were collected from the anaerobic flasks as described above. Water fractions were separated and analyzed as described above. Soil fractions were extracted with acetone:water, then the aqueous acetone extract was chilled on dry ice and filtered. The flask collecting the filtrate was also chilled on dry ice and a dry ice:acetone trap was used to trap any volatiles released during filtration; [^{14}C]volatiles were not detected in the trap. Soil extracts were analyzed using LSC and

HPLC as described above; unextractable [^{14}C]residues remaining in the extracted soil were quantified using LSC following combustion.

DATA SUMMARY:

Tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; radiochemical purity >99%], at 0.5 ppm, degraded with a registrant-calculated half-life of approximately 47 days in clay soil that was incubated anaerobically (flooding plus nitrogen atmosphere) at approximately 25 C in the dark for 62 days following 19 days of aerobic incubation (Figure 14). Chlorethoxyphos decreased from 40.2-49.5% of the applied radioactivity at 19 days posttreatment just prior to flooding to 26.7-52.1% after 30 days of anaerobic incubation (49 days posttreatment) and 16.1-19.1% after 62 days (81 days posttreatment; Figure 14). Unidentified [^{14}C]compounds described as "others" were detected at 1.4% of the applied (0.007 ppm) radioactivity after 62 days of anaerobic incubation; it was reported that the "others" consisted of at least four compounds, each at <1% of the applied (0.005 ppm; Table IV).

After 62 days of anaerobic incubation (81 days posttreatment), $^{14}CO_2$ totaled an average of 36.5% of the applied radioactivity, organic [^{14}C]volatiles (reported as consisting of "mostly" parent chlorethoxyphos) totaled an average of 4.8%, and unextracted [^{14}C]residues averaged a maximum of 25.2% (Table III). Of the unextractable [^{14}C]residues in the 62-day anaerobic soil sample, 8.4% of the applied was characterized as humin, 6.3% as humic acid, and 9.4% as fulvic acid (Figure 15). The majority of the [^{14}C]residues remained associated with the soil following flooding; only 0.5% of the applied radioactivity was associated with the water fraction after 30 and 62 days of anaerobic incubation (49 and 81 days posttreatment; Table III). During the study, average material balances ranged from 83.4 to 106.1% of the applied.

In a supplemental experiment that was performed to account for low material balances that occurred during the anaerobic phase of the initial experiment described above, tetrachloroethoxy-labeled [2-^{14}C]chlorethoxyphos, at 0.5 ppm, degraded with a registrant-calculated half-life of approximately 41 days in clay soil that was incubated anaerobically (flooding plus nitrogen atmosphere) at approximately 25 C in the dark for 62 days following 19 days of aerobic incubation (Figure B1 in Appendix II). During the aerobic phase of the supplemental experiment, air flow through the flasks was increased to 30 mL/minute compared to 2-6 mL/minute that was used in the initial experiment. As a result of the increased air flow, [^{14}C]volatiles totaled an average of 45.5% of the applied radioactivity by 19 days posttreatment; the study authors reported that the [^{14}C]volatiles consisted of parent chlorethoxyphos, but supporting data were not provided (Table B1 in Appendix II).

Relative chlorethoxyphos (volatilized plus nonvolatilized) decreased from 59.8-63.4% of the applied radioactivity at 19 days posttreatment just prior to flooding to 37.2-38.0% after 30 days of anaerobic incubation (49 days posttreatment) and 20.3-22.5% after 62 days (81 - days posttreatment; Table B3 in Appendix II). The study authors reported that no degradates were detected at >1% of the applied radioactivity (0.005 ppm) in the soil extracts; the number of degradates detected was not reported. At 81 days posttreatment, [^{14}C]volatiles (reported as consisting of parent chlorethoxyphos) totaled an average of 58.3% of the applied radioactivity, evolved $^{14}CO_2$ totaled 13.4% (maximum of 16.7% at 49 days posttreatment) of the applied, and unextracted [^{14}C]residues totaled 12.0% of the applied. Average material balances gradually decreased from 100.0% of the applied at day 0 to 93.2% at 81 days posttreatment.

COMMENTS:

1. Material balances for the initial experiment were incomplete; averages of 16.6 and 14.0% of the applied radioactivity were unaccounted for after 30 and 62 days of anaerobic incubation, respectively (49 and 81 days posttreatment, respectively). The study authors believed that the low material balances were due to incomplete trapping of evolved $^{14}CO_2$. In an attempt to determine the cause of the low material balances, a supplemental experiment was conducted in which air flow through the flasks during the aerobic phase of the experiment was increased to 30 mL/minute compared to 2-6 mL/minute that was used in the initial experiment. In the supplemental experiment, [^{14}C]volatiles totaled an average of 45.5% of the applied radioactivity by 19 days posttreatment, apparently the result of the increased air flow; in contrast, [^{14}C]volatiles totaled only 13.2% of the applied by 19 days in the initial experiment. The study authors reported that the [^{14}C]volatiles consisted of parent chlorethoxyphos; however, supporting data were not provided. In the supplemental experiment, average material balances did slowly decrease, but only to 93.2% of the applied after 62 days of anaerobic incubation (81 days posttreatment).

   Taken together, the initial and supplemental experiments indicate that chlorethoxyphos in soil degrades to CO_2 via trichloroacetaldehyde (not detected) under aerobic and anaerobic conditions. The registrant must provide the actual concentrations of parent chlorethoxyphos that were detected as organic volatiles, and specify the concentrations of any other volatilized [^{14}C]compounds that were present; it was implied that these data were generated during the study, but the data were not provided for review.

2. For the supplemental experiment, the actual concentrations of parent chlorethoxyphos detected in the soil extracts and the concentrations of volatilized chlorethoxyphos should be separately reported; only

the relative percent of parent chlorethoxyphos (volatilized plus nonvolatilized) was reported (Table B3 in Appendix II).

3. For the initial experiment, unidentified [^{14}C]compounds described as "Others" in Table IV totaled 1.5% of the applied radioactivity (0.0075 ppm). The study authors reported that the unidentified compounds consisted of at least four degradates, none at >1% of the applied (0.005 ppm). For the supplemental experiment, the study authors reported that no degradates were detected at >1% of the applied radioactivity in the soil extracts; the number of degradates detected was not reported.

4. The majority of the results, including unidentified [^{14}C]compounds, evolved $^{14}CO_2$ and volatiles, unextractable [^{14}C]residues, and material balances, were presented as the average of the two replicates that were collected at each sampling interval. The results from analysis of each soil or soil:water sample should have been presented to provide the maximum concentrations of the degradates and unidentified [^{14}C]compounds that were detected at each sampling interval, and to assess the variability between the replicates. Individual concentrations of parent chlorethoxyphos were obtained from the linear regression figures (Figure 14 in the main study text and Figure B1 in Appendix II).

5. For the initial experiment, the results from the aerobic soil flasks collected at 0, 19, 49, and 81 days posttreatment are the same as those reported in the aerobic soil metabolism study (Study 4, MRID 41736824) submitted concurrently with this study.

6. The study authors classified the test soil as a La Hogue clay loam; however, using the USDA soil classification system, the percentages of sand, silt, and clay classified the test soil as a clay, and the soil was referred to as such in this review (Table II). The percentages of sand, silt, and clay totaled 99%, rather than 100%; however, this did not affect the outcome of the soil classification. The study authors also reported that the test soil was chosen because it is typical of the Midwest corn belt, and the higher organic content (4.2%) would reduce loss of chlorethoxyphos through vaporization.

7. A study investigating the metabolism of chlorethoxyphos labeled in the phosphoryl moiety of the compound in anaerobic soil may be required.

8. The registrant reported that chlorethoxyphos (DPX-43898) is an organophosphorus insecticide used for control of corn root worms and other soil insects and is currently being developed for use on corn. The proposed use rate was reported as 0.3 oz ai/1000 feet of row (0.25 lb ai/A assuming 40-inch row spacing; MRID 41736820).

9. In an aerobic soil metabolism experiment (Study 4, MRID 41736824), chlorethoxyphos degraded with a registrant-calculated half-life of approximately 23 days in clay soil that was incubated in the dark at 25 C and 75% of field moisture capacity. Only chlorethoxyphos was identified in the soil; carbon dioxide was the major degradate and totaled 63.0% of the applied at 120 days posttreatment.

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Pages 87 through 104 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

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Reference 2 demonstrates that chloral hydrate can be easily leached from any of the four test soils indicating the compounds high mobility potential. Trichloroacetic acid, the major degradate of chloral hydrate in soils, is used as a standard for the highest mobility class in soils (MRID 41736820). The Freundlich adsorption constant k, for trichloracetic acid extrapolated from the information in Reference 1 and is <1 (0.1 to 0.3).

DATA SUMMARY:

See attached data tables and summaries.

DISCUSSION:

1. Studies MRID #41290618, #40883709 were previously reviewed as mentioned above. At that time, the registrant was requested to submit data on the mobility of the degradate trichloroacetaldehyde, which exists in the hydrate form (chloral hydrate) in moist soil. In response, the registrant submitted two published German studies (publication dates of 1968 and 1974) concerning the mobility of chloral hydrate and trichloroacetic acid; partial English translations were also provided (Attachments I and II). The partial English translations were too brief to allow an adequate review of the studies. It is apparent, however, that neither study is consistent with Subdivision N guidelines. The registrant summarized from Attachment I that chloral hydrate is highly mobile in soil with a Freundlich constant of <1.

2. The registrant notes that in the aerobic soil metabolism study (Study 4, MRID 41736824) submitted concurrently with this document, no degradates were detected at >2% of the applied radioactivity (0.01 ppm) in soil extracts. The registrant also submitted published Chinese (1982, Attachment III) and German (1969, Attachment IV) studies to support the rapid degradation of chloral hydrate in soil; partial English translations were provided.

   In a previously reviewed and acceptable aerobic soil metabolism study (MRID #40883706 and #41290617 EFGWB#'s 90-0067, 2/21/90; 90-186, 1/31/89) Chloral hydrate accounted for a maximum of 21.9% of applied at day 5 and decreased to less than 5.1% at day 7, which gives an indication of the rapid degradation of chloral hydrate in soil.

3. In a terrestrial field dissipation study (Study 8, MRID 41736829) submitted concurrently with this document, the major degradate detected in the soil was trichloroacetic acid (TCA); TCA was detected in the 0- to 3-inch soil depth at up to 0.05 ppm at the North Carolina site, 0.07 ppm at the Illinois site, 0.23 ppm at the Iowa site, and 0.35 ppm at the California site. The registrant reported that trichloroacetic acid has a Freundlich constant of <1 (Attachment I, MRID 41736826) and is used as a standard for the highest mobility class in soils (MRID 41736820).

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Pages 107 through 140 are not included.

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____ Description of quality control procedures.

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DATA EVALUATION RECORD

STUDY 7

CHEM 129006 Chlorethoxyphos (DPX-43898) §163-2

FORMULATION--04--GRANULAR (G)

STUDY ID 41736827
Barefoot, A.C., and M. Troup-Mayforth. 1990. Laboratory volatility of DPX-43898. Report No. AMR-1172-88. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

DIRECT REVIEW TIME = 23

REVIEWED BY: L. Binari TITLE: Staff Scientist

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SIGNATURE: K. Poff

CONCLUSIONS:

Laboratory Volatility

1. Study MRID #41736827 is acceptable and completely satisfies the laboratory volatility 163-2 data requirement for chlorethoxyphos.

2. The mass flux of chlorethoxyphos which was calculated from the total amount of compound which evolved per hour using the area of the soil surface was measured at 45.6 cm^2. The highest concentration, which was 281 ug/m^3, was measured on Day 1 of the experiment where chlorethoxyphos was incorporated 1/4 inch into the sandy loam soil. By day 16 the concentration decreased to 23.1 ug/m^3. When incorporated 1 inch into the soil chlorethoxyphos was measured at 183 ug/m^3 on Day 1 and decreased to 36.6 ug/m^3 by Day 13. Three other

soils were tested at 1/4 inch and 1 inch incorporation of chlorethoxyphos 10G at approximately 4X. The volatilization was dependent upon the %OM and the depth of incorporation of chlorethoxyphos into the soil.

METHODOLOGY:

Portions (200 g) of sandy loam (72% sand, 19% silt, 9% clay, 1.0% organic matter, pH 6.4, CEC 6.7 meq/100 g) and silt loam (29.2% sand, 52.0% silt, 18.8% clay, 5.6% organic matter, pH 6.9, CEC 26.7 meq/100 g) soil were placed into flasks, moistened to 70% of holding capacity, and treated at approximately 15 ppm (1 lb ai/A) with chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; Fortress, 10% G, du Pont]; three flasks were prepared for each soil type. Following treatment, the pesticide was incorporated into the soil to a depth of 0.25 inches. Three additional flasks of sandy loam soil were prepared and treated as described above, but the pesticide was incorporated to a depth of 1 inch. The flasks of treated soil were attached to a nitrogen-flow system; humidified nitrogen was continuously drawn (1 L/minute) through the flasks, then sequentially through two ethylene glycol trapping solutions (Figure 2). The treated soil was maintained in an incubator at 25 C. Trapping solutions were collected and replaced at 1- to 3-day intervals during the study (Table III).

An aliquot (20 mL) of each ethylene glycol trapping solution was partitioned twice with hexane. Hexane extracts were combined and an aliquot (3 uL) was analyzed for chlorethoxyphos using GC with electron-capture detection; the limit of detection was not reported.

DATA SUMMARY:

Chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; Fortress, 10% G], at approximately 15 ppm (1 lb ai/A), volatilized with observed half-lives of approximately 7 days in sandy loam soil and 10-12 days in silt loam soil when incorporated to a depth of 0.25 inches and incubated at 25 C, 70% of moisture holding capacity, and under a flow of humidified nitrogen at 1 L/minute; when incorporated to a depth of 1 inch in sandy loam soil, chlorethoxyphos volatilized with an observed half-life of >13 days (final sampling interval; Table IV and Figure 4). Volatilization decreased with increasing soil organic matter content and depth of incorporation.

In sandy loam soil, the volatilization of chlorethoxyphos incorporated to 0.25 inches decreased from an average of 0.369 ug/cm^2/hr at 1 day posttreatment to 0.126 ug/cm^2/hr at 7 days and was 0.030 ug/cm^2/hr at 16 days (final sampling interval; Table III). The air concentration of chlorethoxyphos decreased from an average of 281 ug/m^3 at 1 day posttreatment to 181-209 ug/m^3 at 2-4 days, 95.7 ug/m^3 at 7 days, and was 23.1 ug/m^3 at 16 days. By 16 days posttreatment,

an average of 71.6% of the applied chlorethoxyphos had been volatilized (Table IV).

In silt loam soil, the volatilization of chlorethoxyphos incorporated to 0.25 inches increased from 0.192 ug/cm^2/hr at 1 day posttreatment to 0.278 ug/cm^2/hr at 2 days, then decreased to 0.057 ug/cm^2/hr by 6 days, increased to 0.141 ug/cm^2/hr by 12 days, and decreased to 0.029 ug/cm^2/hr by 19 days (final sampling interval). The air concentration of chlorethoxyphos increased from 146 ug/m^3 at 1 day posttreatment to 212 ug/m^3 at 2 days, then decreased to 43.6 ug/m^3 at 6 days, increased to 107 ug/m^3 at 12 days, and decreased to 22.3 ug/m^3 at 19 days. By 19 days posttreatment, an average of 64.8% of the applied chlorethoxyphos had been volatilized.

In sandy loam soil, the volatilization of chlorethoxyphos incorporated to 1 inch decreased from 0.241 ug/cm^2/hr at 1 day posttreatment to 0.119 ug/cm^2/hr at 4 days, was 0.039 ug/cm^2/hr at 7 days, and 0.051 ug/cm^2/hr at 10 and 13 days (final sampling interval). Air concentration of chlorethoxyphos decreased from 183 ug/m^3 at 1 day posttreatment to 90.5 ug/m^3 at 4 days and was 29.5-38.8 ug/m^3 at 7-13 days. By 13 days posttreatment, an average of 37.0% of the applied chlorethoxyphos had been volatilized.

COMMENTS:

1. The material balances were incomplete; 28.4-63.0% of the applied chlorethoxyphos was unaccounted for at the final sampling interval. The study authors failed to analyzed the soil for nonvolatilized chlorethoxyphos.

2. The efficiency of the trapping solution was not reported.

3. All results, including air concentrations of chlorethoxyphos, volatilization (mass flux) values, and material balances, were presented as the average of three replicates that were collected at each sampling interval. The results from analysis of each replicate should have been presented to provide the maximum concentrations of the parent compound that were detected at each sampling interval, and to assess the variability between the replicates.

4. In the experiment in which chlorethoxyphos was incorporated to a depth of 1 inch in sandy loam soil, the experiment was terminated before the pattern of decline of the test substance was established; only 37.0% of the applied chlorethoxyphos had volatilized by the final sampling interval at 13 days posttreatment.

5. The vapor pressure of chlorethoxyphos was reported as 1.7 x 10^{-3} mm Hg at 25 C (MRID 41736820).

6. The registrant reported that chlorethoxyphos (DPX-43898) is an organophosphorus insecticide used for control of corn root worms and other soil insects and is currently being developed for use on corn.

The proposed use rate was reported as 0.3 oz ai/1000 feet of row (0.25 lb ai/A assuming 40-inch row spacing; MRID 41736820). The study authors reported that the treatment rate of 1 lb ai/A used in this study was four times the proposed use rate of chlorethoxyphos (0.25 lb ai/A).

Fortress

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Pages 145 through 152 are not included.

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____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

X FIFRA registration data.

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DATA EVALUATION RECORD

STUDY 8

CHEM 129006 Chlorethoxyphos (DPX-43898) §164-1

FORMULATION--04--GRANULAR (G)

STUDY ID 41736829
Slates, R.V., and C.D. Crowe. 1990. Field soil dissipation of Fortress soil insecticide. Du Pont Report No. AMR-1143-88. Unpublished study performed by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE, and Minnesota Valley Testing Laboratories, Inc., New Ulm, MN; and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

STUDY ID 41290603
Babicki, W.A., Jr., G.F. Barber, and R.V. Slates. 1988. Residue method for determination of Fortress insecticide active ingredient DPX-43898 and its oxon analogue IN-34158 in soil by electron-capture gas chromatography. Report No. AMR-1194-88. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

STUDY ID 41290605
Barber, G.F. 1989b. Analytical method for the determination of residues of trichloroacetic acid in crops and soils. Report No. AMR-1253-88. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

DIRECT REVIEW TIME = 40

REVIEWED BY: L. Binari TITLE: Staff Scientist

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CONCLUSIONS:

Terrestrial Field Dissipation

1. Portions of study MRID #41736829 may be upgradable to acceptable with the submission of additional information but does not currently satisfy the terrestrial field dissipation 164-1 data requirement for chlorethoxyphos for the following reasons:

2. The portions of this study conducted in Iowa and California are scientifically sound and may be made upgradable by rectifying the following deficiencies:

   field test procedures were not completely described,

   field test data were incomplete, and

   field maintenance practices were not adequately reported.

   The portion of this study conducted in North Carolina is scientifically sound and may be made upgradable by rectifying the following deficiencies:

   soil samples collected during the initial 2 weeks posttreatment (five sampling intervals) may have been misidentified; therefore, conclusions based on the results of those soil samples are tentative.

   The portion of this study conducted in Illinois is unacceptable and may not be upgradable for the following reason:

   the data were too variable to accurately assess the dissipation of chlorethoxyphos in the soil.

3. In order for the portions of this study conducted in Iowa and California to be upgraded to acceptable, the registrant must provide complete descriptions of the field test procedures and field maintenance practices; submit additional field test data, such as depth to water table, slope of field, etc.; and specify which soil residue data were used to calculate the dissipation rates and substantiate the methods used for the calculations (see Comment 1). In order for the portion of this study conducted in North Carolina to be upgraded to acceptable, the registrant must provide documentation to illustrate how samples collected at 5 sampling intervals were misidentified.

4. Chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester] dissipated with observed half-lives of 7-14 days in sandy loam soil in California and 14-35 days in clay loam soil in Iowa after chlorethoxyphos (Fortress, 10% granular) was applied at 0.6 oz ai/1000 row-feet (0.5 lb ai/A) as a preemergence soil-incorporated band over-row application to plots of field corn. Chlorethoxyphos was detected in the 18- to 24-inch soil depth at the

Iowa site, contamination is suspected. At the California site chlorethoxyphos did not leach below the 0- to 3-inch depth. In California rainfall totaled 0.01 inches at 7 days posttreatment, 0.03 inches at 14 and 30 days, and 7.22 inches at the end of the study; irrigation added an additional 15.5-31 inches by the end of the study (irrigation water was not accurately measured). In Iowa rainfall totaled 1.50 inches at 14 days posttreatment, 3.55 inches at 35 days, and 31.76 inches at the end of the study. The degradate trichloroacetic acid was detected at both sites, but the oxon analog of chlorethoxyphos, [IN-34158; phosphoric acid, diethyl (1,2,2,2-tetrachloroethyl) ester], was detected at the Iowa (and Illinois) site.

METHODOLOGY:

Chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; Fortress, 10% G, du Pont] was applied as a 7-inch band over-row preemergence treatment at 0.6 oz ai/1000 row-feet (0.5 lb ai/A) to plots of field corn in four states:

to sandy loam soil (plot size 4 rows x 80 feet; row width was not reported) in Fayetteville, North Carolina, on June 23, 1988;

to silty clay loam soil (plot size 135 feet, 30-inch row width, number of rows not reported) in Rochelle, Illinois, on May 13, 1988;

to clay loam soil (plot size 4 rows per 10 feet x 75 feet) in Humboldt, Iowa, on June 3, 1988; and

to sandy loam soil (plot size 30 x 66 feet, 40-inch row width) in Madera, California, on June 7, 1988.

Soil characteristics are presented in Appendix A. The pesticide was applied at the time the corn was planted and lightly incorporated into the 0- to 3-inch soil depth. At each site, an untreated plot (plot sizes were not reported) was maintained as a control; the distance between the treated and untreated plots was not reported. Soil cores (1-inch diameter, 0- to 18- or 0- to 36-inch depth) were collected from the treated and control plots prior to treatment, immediately posttreatment, and periodically up to 162 days posttreatment at the North Carolina site, up to 365 days at the Iowa site, and up to 532-540 days at the Illinois and California sites. Soil cores were divided into 0- to 3-, 3- to 6-, 6- to 12-, 12- to 18-, 18- to 24-, 24- to 30-, and 30- to 36-inch segments; the soil segments were composited according to depth and sampling interval. Soil samples were stored frozen approximately 6-20 months prior to extraction; soil extracts were stored for up to 64 days prior to analysis.

Composite soil samples were homogenized using a Hobart chopper and dry ice. To isolate parent chlorethoxyphos and its oxon analog [IN-34158; phosphoric acid, diethyl (1,2,2,2-tetrachloroethyl) ester], soil subsamples were extracted with hexane:acetone (1:1, v:v) using a mechanical tumbler for 2 hours. The samples were centrifuged, and the extracts were decanted and partitioned twice with deionized water; the aqueous phases were discarded. Aliquots of the remaining hexane solutions were filtered, then analyzed for chlorethoxyphos and IN-34158 using GC with electron-capture detection.

To isolate the degradate trichloroacetic acid (TCA), additional soil subsamples (50 g) were extracted with methanol:water (95:5, v:v) using sonication for 20-30 minutes, then rinsed twice with methanol:water by vigorous shaking for 30 seconds; after each extraction or rinse, the samples were centrifuged and the supernatants decanted. The extracts and rinses were combined, and aliquots of the combined solution were placed in a silanized tube, treated with 10% aqueous sodium acetate solution, and concentrated. The concentrated extract was partitioned twice with hexane; the hexane phases were discarded. The remaining aqueous phase was transferred to another silanized tube using methanol; the water was removed from the sample by dilution with methanol followed by evaporation under nitrogen, the procedure was repeated six times. The resulting concentrate was derivatized to the methyl ester in the presence of methanol and sulfuric acid at 60 C for 2 hours. The derivatized sample was cooled to room temperature, then diluted with hexane and 10% aqueous sodium chloride solution. The hexane solution containing TCA methyl ester was eluted through a silica gel clean-up column with hexane. An aliquot of the eluate was analyzed for TCA methyl ester using GC with electron-capture detection.

The quantitation limit for all three compounds was 0.01 ppm. Recovery efficiencies from soil samples fortified with chlorethoxyphos at 0.01-4.0 ppm ranged from 55 to 122% (mean 93%) of the applied, IN-34158 at 0.01-4.0 ppm ranged from 64 to 117% (mean 87%), and TCA at 0.01-0.10 ppm ranged from 70 to 109% (mean 86%). Results were not corrected for soil moisture content; it was not specified if results were corrected for recovery efficiency.

DATA SUMMARY:

Chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester] dissipated with observed half-lives of 7-48 days from the upper 3 inches of plots planted to corn located in North Carolina, Illinois, Iowa, and California following a preemergence soil-incorporated band over-row application of chlorethoxyphos (Fortress, 10% G) at 0.6 oz ai/1000 row-feet (0.5 lb ai/A). Chlorethoxyphos leached into the 3- to 6-inch soil depth at the Illinois (silty clay loam) site, into the 12- to 18-inch depth at the North Carolina (sandy loam) site, and into the 18- to 24-inch depth at the Iowa (clay loam) site; chlorethoxyphos did not leach

below the 0- to 3-inch soil depth at the California (sandy loam) site. The degradate

trichloroacetic acid (TCA),

derivatized as TCA methyl ester, was detected in soil from all four sites. Downward movement of TCA into the 3- to 6-inch soil depth occurred at the North Carolina, Illinois, and Iowa sites; TCA did not leach below the 0- to 3-inch depth at the California site. The oxon analog of chlorethoxyphos,

phosphoric acid, diethyl (1,2,2,2-tetrachloroethyl) ester (IN-34158),

was detected only in soil from the Illinois and Iowa sites; IN-34158 did not leach below the 0- to 3-inch soil depth at those sites.

In North Carolina, chlorethoxyphos dissipated with an observed half-life of 14-48 days from the upper 3 inches of a plot (4 rows x 80 feet; row width not reported) of sandy loam soil planted to corn that was treated on June 23, 1988 (Table I). In the 0- to 3-inch soil depth, chlorethoxyphos increased from 0.43 ppm at day 0 posttreatment to 1.41 ppm at 14 days, then decreased to 0.26 ppm at 48 days, 0.08 ppm at 61 days, and 0.02 ppm at 162 days (final sampling interval). The study authors inverted the 0- to 14-day results for the half-life calculations (see Comment 3). Chlorethoxyphos was detected three times in the 3- to 6-inch soil depth at 0.05 ppm at 0 days posttreatment, 0.04-0.05 ppm at 1 day, and 0.02 ppm at 76 days; twice in the 6- to 12-inch depth at 0.05 ppm at day 0 and 0.02 ppm at 1 day; once in the 12- to 18-inch depth at 0.04 ppm at day 0. Chlorethoxyphos was <0.01 ppm (quantitation limit) at all other sampling intervals and depths (up to 36 inches; Table Ia). In the 0- to 3-inch soil depth, the degradate TCA increased from 0.02 ppm at day 0 posttreatment to 0.05 ppm at 4 days, was 0.03-0.04 ppm at 12-14 days, and was <0.01 ppm (quantitation limit) at all other sampling intervals. At lower soil depths, TCA was detected only once in the 3- to 6-inch depth, at 0.01 ppm at 12 days posttreatment, and was <0.01 ppm at all other sampling intervals and depths. The degradate IN-34158 was <0.01 ppm (quantitation limit) at all sampling intervals and soil depths. Rainfall totaled 1.10 inches at 14 days posttreatment, 7.76 inches at 48 days, and 25.86 inches at the end of the study (6/23/88 to 12/2/88). The average monthly air temperatures ranged from 28 to 91 F; soil temperatures were not reported.

In Illinois, chlorethoxyphos dissipated with an observed half-life of approximately 14 days from the upper 3 inches of a plot (135 feet, 30-inch row width, number of rows not reported) of silty clay loam soil planted to corn that was treated on May 13, 1988 (Table II). In the 0- to 3-inch soil depth, chlorethoxyphos decreased from 3.16 ppm at 0 days posttreatment to 1.18 ppm at 3 days and 0.86 ppm at 7 days, increased to 1.55 ppm at 14 days, then decreased to 0.13 ppm at 27 days, 0.04 ppm at 111 days, and <0.01 ppm at 161 and 329 days;

chlorethoxyphos was 0.02 ppm at 364 days (final sampling interval; Table IIa). Chlorethoxyphos was detected three times in the 3- to 6-inch soil depth, at 0.01 ppm at 0 and 111 days posttreatment and at 0.03 ppm at 1 day, and was <0.01 ppm at all other sampling intervals and depths. In the 0- to 3-inch soil depth, TCA was 0.02-0.04 ppm at 0-7 days posttreatment, 0.07 ppm at 14 days, 0.02 ppm at 27 days, 0.05 ppm at 61 days, and <0.01 ppm at 111 and 161 days. At lower soil depths, TCA was detected only once in the 3- to 6-inch depth, at 0.02 ppm at 14 days posttreatment, and was <0.01 ppm at all other sampling intervals and depths. In the 0- to 3-inch soil depth, IN-34158 was 0.03 ppm at 0 days posttreatment, 0.01 ppm at 1 and 3 days, and <0.01 ppm at all other sampling intervals; IN-34158 was not detected in soil depths below 3 inches. Rainfall plus irrigation totaled 1.2 inches at 14 and 27 days and was 31.3-32.8 inches (irrigation water was not accurately measured) by the termination of the study. The average monthly air temperatures ranged from 9 to 90 F; soil temperatures were not reported.

In Iowa, chlorethoxyphos dissipated with an observed half-life of 14-35 days from the upper 3 inches of a plot (4 rows per 10 feet x 75 feet) of clay loam soil planted to corn that was treated on June 3, 1988 (Table III). In the 0- to 3-inch soil depth, chlorethoxyphos was 1.17 ppm at 0 days posttreatment, 0.86-1.12 ppm at 1-7 days, 0.77 ppm at 14 days, 0.05-0.06 ppm at 35-122 days, and <0.01 ppm at 276 and 365 days (final sampling interval; Table IIIa). Chlorethoxyphos was detected twice in the 3- to 6-inch soil depth, at 0.02 ppm at 35 and 61 days posttreatment; once in the 6- to 12-inch depth, at 0.01-0.02 ppm at 35 days; once in the 12- to 18-inch depth, at 0.03-0.04 ppm at 35 days; and once in the 18- to 24-inch depth, at 0.01 ppm at 35 days; chlorethoxyphos was <0.01 ppm at all other sampling intervals and depths. In the 0- to 3-inch soil depth, TCA increased from 0.05 ppm at 0-1 days posttreatment to 0.23 ppm at 7 days, then decreased to 0.19 ppm at 14 days, 0.03 ppm at 35 days, 0.02 ppm at 61 days, and <0.01 ppm at 122 days. In the lower soil depths, TCA was detected only once in the 3- to 6-inch depth, at 0.05 ppm at 14 days posttreatment, and was <0.01 ppm at all other sampling intervals and depths. IN-34158 was detected only once in the 0- to 3-inch soil depth, at 0.01 ppm at 3 days posttreatment, and was <0.01 ppm at all other sampling intervals and depths. Rainfall totaled 1.50 inches at 14 days posttreatment, 3.55 inches at 35 days, and 31.76 inches at the end of the study. The average monthly air temperatures ranged from 6 to 87 F; soil temperatures were not reported.

In California, chlorethoxyphos dissipated with an observed half-life of 7-14 days from the upper 3 inches of a plot (30 x 66 feet, 40-inch row width) of sandy loam soil planted to corn that was treated on June 7, 1988 (Table IV). In the 0- to 3-inch soil depth, chlorethoxyphos was 1.11-1.73 ppm at 0-3 days (maximum at 3 days) posttreatment, then decreased to 0.32 ppm at 14 days, 0.03 ppm at 30 days, <0.01-0.02 ppm at 59-273 days, and <0.01 ppm at 364 days (final sampling interval; Table IVa). Chlorethoxyphos was not detected (<0.01 ppm) in soil depths below 3 inches at any sampling interval.

In the 0- to 3-inch soil depth, TCA increased from 0.03 ppm at 0 days posttreatment to 0.35 ppm at 14 days, then decreased to 0.14 ppm at 30 days, 0.02 ppm at 59 days, and <0.01 ppm at 121 days. TCA was not detected in soil depths below 3 inches at any sampling interval. IN-34158 was not detected at any sampling interval. Rainfall totaled 0.01 inches at 7 days posttreatment, 0.03 inches at 14 and 30 days, and 7.22 inches at the end of the study; irrigation added an additional 15.5-31 inches by the end of the study (irrigation water was not accurately measured). Average monthly air temperatures ranged from 31 to 99 F; soil temperatures were not reported.

COMMENTS:

1. The study authors reported that dissipation of chlorethoxyphos did not follow first-order kinetics at any of the test sites; therefore, initial (50% dissipation) and secondary (90% dissipation) half-lives were calculated. The study authors calculated dissipation times using the ln(ppm chlorethoxyphos) versus the square root of the application to sampling time. It was not reported which sampling intervals were used to calculate the initial (50%) half-lives and which were used to calculate the secondary (90% dissipation) half-lives; plots showing all sampling intervals at which chlorethoxyphos was detected were provided (Figure A-D). In addition, the study authors did not adequately explain why the square root of the sampling interval was used to calculate half-lives, and the Dynamac reviewer could not confirm any of the dissipation times calculated by the study authors; therefore, only observed half-lives were reported in this review.

   The following dissipation times were reported by the study authors and all pertain to dissipation from the 0- to 3-inch soil depth (page 17). At the North Carolina site, chlorethoxyphos dissipated with an initial (50%) half-life of 3.7 days and a secondary (90%) half-life of 41 days; the study authors inverted the 0- to 14-day results for the half-life calculations (see Comment 3). At the Illinois site, chlorethoxyphos dissipated with an initial half-life of 2.6 days and a secondary half-life of 28 days. At the Iowa site, chlorethoxyphos dissipated with an initial half-life of 3.7 days and a secondary half-life of 41 days. At the California site, chlorethoxyphos dissipated with an initial half-life of 0.9 days and a secondary half-life of 9.6 days. The registrant must specify which data were used and substantiate the methods used to calculate the dissipation rates.

2. Results reported in Tables I-IV in the study text were incomplete. The tables did not present results from all sampling intervals that chlorethoxyphos was detected, and results from soil depths below 6 inches were not presented; therefore, results were frequently cited from the Analytical Information presented in Appendix I (renamed as Tables Ia-IVa, respectively). In addition, the 176-day sampling

interval reported in Table III should have been reported as the 276-day sampling interval.

3. At the North Carolina site, chlorethoxyphos detected in the 0- to 3-inch soil depth increased from 0.43 ppm at day 0 posttreatment to 0.69 ppm at 1 day, 1.23 ppm at 4 days, 1.21 ppm at 12 days, and 1.41 ppm at 14 days, then decreased to 0.26 ppm at 48 days, 0.08 ppm at 61 days, and was 0.02 ppm at 162 days. The study authors reported that the increase in chlorethoxyphos during the initial 2 weeks posttreatment appeared illogical and concluded that the samples must have been identified in reverse order when barcodes were assigned. In the Sampling Information it was reported that ID barcodes were assigned at the time of sample collection (pages 39, 56, 74, and 91); therefore, the registrant must provide some sort of documentation to illustrate how samples collected at five sampling intervals were misidentified.

   In addition, chlorethoxyphos was detected down to the 12- to 18-inch soil depth at day 0 posttreatment and to the 6- to 12-inch depth at 1 day, then was not detected below the 3- to 6-inch depth for the remainder of the study. The study authors contend that detection of chlorethoxyphos at the lower soil depths (below 6 inches) was due to contamination during sampling. According to the Sampling Information in Appendix I, the 0- to 18-inch soil cores were shipped intact (no field processing); therefore, if the 0- and 1-day soil samples are actually 0- and 1-day samples, then contamination is a possibility. However, if the 0- and 1-day soil samples are actually 14- and 12-day samples, since the study authors believe that the 0- to 14-day samples were reversed, then chlorethoxyphos in the lower soil depths is most likely due to leaching; and the study authors failed to define the extent of leaching since soil samples were not collected below 18 inches at those sampling intervals.

4. At the Illinois site, the data were too variable to accurately establish the half-life of dissipation of chlorethoxyphos in the soil; in the 0- to 3-inch soil depth, chlorethoxyphos decreased from 3.16 ppm at day 0 posttreatment to 1.18 ppm at 3 days and 0.86 ppm at 7 days, increased to 1.55 ppm at 14 days, then decreased to 0.13 ppm at 27 days, 0.04 ppm at 111 days, <0.01 ppm at 161 and 329 days, and was 0.02 ppm at 364 days.

5. Field test procedures were not completely described, although sufficient information was provided to permit review of the study. Information that was not provided in this study document included the following:

   a) For all sites, soil CEC values were not reported. The soil (0- to 6-inch depth) from the treated plot at the Iowa site was classified as a loam; however, using the USDA soil classification system, the percentages of sand, silt, and clay classified the test soil as a clay loam and the soil was referred to as such in this review (Appendix A).

b) For all sites, the test sites were not adequately characterized; plot sizes of the untreated control plots, distance between the treated and untreated plots, depth to the water table, and slope of the field were not reported. In addition, treated plot sizes were not adequately described. The treated plot size at North Carolina was reported as 4 rows x 80 feet, but the width of each row was not reported. The treated plot size at Illinois was reported as 135 feet, 30-inch row width; however, it was unclear if that meant the plot was one 30-inch row, 135 feet long. A figure illustrating the layout of the treated plot in relationship to the untreated plot was not provided.

c) The study authors reported in the Basic Study Information table (page 13) that soil cores were collected up to 540 days posttreatment at the Iowa site; however, it was reported in the Sampling Information that the final sampling interval occurred at 365 days posttreatment (page 74).

d) Sampling procedures were not accurately described. The study authors reported that 24-40 soil cores were generally collected from each plot at each sampling interval. In Appendix I, it was reported that 6 soil cores per row (4 row total) were collected at the North Carolina site and 10 cores per row (number of rows was not specified) were collected at the Illinois site, but the number of cores per row were not reported for the Iowa and California sites.

e) Field maintenance procedures were not completely described. The study authors reported that the field corn planted the day of treatment was grown to maturity; however, it was not reported when or if the corn was harvested and whether or not any soil cultivation occurred at harvest.

f) It was not specified if results were corrected for recovery efficiency. If the results were not corrected for recovery efficiency, then concentrations of chlorethoxyphos, TCA, and IN-34158 may have been underestimated; recovery efficiencies from soil samples fortified with chlorethoxyphos at 0.01-4.0 ppm ranged from 55 to 122% (mean 93%) of the applied, TCA at 0.01-0.10 ppm ranged from 70 to 109% (mean 86%), and IN-34158 at 0.01-4.0 ppm ranged from 64 to 117% (mean 87%).

6. Freezer storage stability studies for chlorethoxyphos and IN-34158 (Study 12, MRID 42559238) and TCA (Study 13, MRID 42559239) were submitted in the same data package as this study and found to be scientifically sound. In the stability studies, chlorethoxyphos was stable in clay loam soil stored frozen (-20 C) for up to 31 months, and TCA was stable in clay loam soil stored frozen (-20 C) for up to 23 months. IN-34158 decreased by approximately 35% after 35-42 days of storage and 46% after 3 months, then did not degrade further during 6-31 months of storage. Further experiments determined that

IN-34158 degraded rapidly during the period between fortification of the soil samples and when the samples were actually frozen; however, the compound did not appear to degrade further during frozen storage. Although IN-34158 was not stable prior to frozen storage, IN-34158 appeared to be a minor soil degradate in this study; it was detected only at the Illinois and Iowa test sites at ≤0.03 ppm. In this study, soil samples were stored frozen approximately 6-20 months prior to extraction; soil extracts were stored up to 64 days prior to analysis. In general, extracts from the 0- to 1-month soil samples were analyzed within 1 week of extraction, but extracts from the later sampling intervals were stored up to 2 months prior to analysis. The registrant may be required to provide information showing that chlorethoxyphos and its degradates TCA and IN-34158 are stable in soil extracts stored for up to 2 months.

7. For all sites, minimum and maximum air temperatures were reported as monthly means, rather than reporting daily minimum and maximum air temperatures; the soil temperatures were not reported. The plots at the Illinois and California sites were irrigated, but the amount was not accurately measured; in the Weather and Irrigation Information, it was reported that approximately 2-2.5 inches of irrigation water was applied to the Illinois plot on three occasions (6/15, 7/6, and 7/20/88; page 57) and 15.5-31 inches was applied during 28 irrigation events (6/7/88-6/6/89) at the California site (page 94). For the California site, meteorological data were not collected at the test site and the distance from the site to the NOAA weather station (unnamed) was not reported.

8. The North Carolina site had not been treated with any pesticides prior to this study; previous use of the test site was not reported. In addition to chlorethoxyphos, the North Carolina site also received two applications of atrazine (4/22 and 6/23/88), one application of butylate (4/22/88), and one application of alachlor (6/23/88).

   Prior to this study, the Illinois site was treated with DPX-V9360 in 1986; previous use of the site was not reported. In addition to chlorethoxyphos, the Illinois site also received one application each of atrazine (5/13/88), alachlor (5/13/88), and glyphosate (6/7/88).

   Prior to this study, the Iowa site was treated with alachlor in 1987 and 1986; previous use of the site was not reported. In addition to chlorethoxyphos, the Iowa site also received one application of alachlor (5/15/88).

   The California site had not been treated with any pesticides prior to this study; previous use of the site was not reported. In addition to chlorethoxyphos, the California site also received three applications of glyphosate (5/4, 8/22, and 11/2/88).

9. The registrant reported that chlorethoxyphos (DPX-43898) is an organophosphorus insecticide used for control of corn root worms and other soil insects and is currently being developed for use on corn.

The proposed use rate was reported as 0.3 oz ai/1000 feet of row (0.25 lb ai/A assuming 40-inch row spacing; MRID 41736820). The study authors reported that the treatment rate of 0.6 oz ai/1000 feet of row used in this study was twice the proposed use rate; the maximum application rate was not reported.

Fortress

Page_____ is not included in this copy.

Pages 164 through 221 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

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The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

DATA EVALUATION RECORD

STUDY 9

CHEM 129006 Chlorethoxyphos (DPX-43898) §164-1

FORMULATION--04--GRANULAR (G)

STUDY ID 41736828
Woodward, M.D. 1990c. Field soil dissipation of Fortress soil insecticide. Supplement No. 1. Report No. AMR-831-87. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

DIRECT REVIEW TIME = 11

REVIEWED BY: L. Binari TITLE: Staff Scientist

EDITED BY: W. Martin TITLE: Asst. Task Leader
K. Ferguson Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

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APPROVED BY: K. Poff
TITLE: Chemist
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SIGNATURE: Kenneth Poff

CONCLUSIONS:

Terrestrial Field Dissipation:

1. Study MRID #41736828 may be used as ancillary information but does not satisfy the terrestrial field dissipation 164-1 data requirement for chlorethoxyphos for the following reason:

a) Study MRID # 41736828 was previously submitted and reviewed under MRID #41290619 (EFGWB #90-0067; 2/21/90). At that time the study was judged to be supplemental but did not satisfy the 164-1 data requirement primarily because the fate of the potential degradates were not addressed in the field. The registrant will not be able to address this concern due to the soil samples being destroyed in a fire.

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2. Chlorethoxyphos was applied as a granular (Fortress 10G) to a Iowa loam soil (pH= 6.8, OM= 3.3%) and a Illinois clay loam soil (pH= 6.2, OM= 4.9%) at rates equivalent to 0.5 to 1.0 lbs ai/acre (2-4 times the maximum label rate) and incorporated to a depth of 3 inches. Almost all of the chlorethoyxphos remained in the top 3 inches of soil at both the Iowa and Illinois sites (although rainfall at both sites during the study were somewhat lower than the historical mean, the almost complete lack of mobility of chlorethoxyphos suggest that supplemental irrigation would have little if any effect). The study authors calculated an initial dissipation half-life of 2.0 days for chlorethoxyphos in the top 3 inches of the Iowa loam soil and reported that 90% had dissipated in 17 days. For chlorethoxyphos in the top 3 inches of the Illinois clay loam soil, the study authors calculated an initial dissipation half-life of 2.6 days and reported that 90% had dissipated in 26 days.

DISCUSSION:

This study was previously submitted and reviewed (review date was not reported) under MRID 41290619. At that time, EPA judged the study acceptable for supplemental information, but requested that soil samples be analyzed for the degradates trichloroacetaldehyde and trichloroacetic acid. In addition, EPA also requested that for future terrestrial field dissipation studies, three composite soil samples should be prepared and analyzed, rather than compositing all of the soil segments into a single sample for each depth and sampling interval.

In response, the registrant contends that the soil samples should have been analyzed for trichloroacetic acid (TCA), but not trichloroacetaldehyde (which exists as the hydrate, chloral hydrate, in moist soil). The registrant submitted published Chinese (1983, Attachment I) and German (1969, Attachment II) studies which found chloral hydrate degraded rapidly in soil with half-lives of $\leq$3.3 days (Table I), and the major degradates were trichloroacetic acid and evolved CO_2; partial English translations of these studies were provided, but were too brief to allow an adequate review of the data. Additionally, in an aerobic soil metabolism study (Study 4, MRID 41736824) submitted concurrently with this response document, no nonvolatile degradates were detected at >2% of the applied (0.01 ppm) during the 120-day study; at 120 days posttreatment, evolved $^{14}CO_2$ was the major degradate totaling 63.0% of the applied radioactivity. The registrant notes that, based on the criteria of the EPA ("Standard Evaluation Procedure for Terrestrial Field Dissipation Studies") and the results from the current aerobic soil metabolism study, no degradates of chlorethoxyphos are present in sufficient quantity to require analyses in the terrestrial field dissipation studies. Trichloroacetic acid, but not chloral hydrate, was detected in previously submitted adsorption/desorption (MRID 41290618), confined rotational crop (MRID 41290620), and plant metabolism (MRID 41290601) studies. Therefore, in a terrestrial field dissipation study (Study

8, MRID 41736829) that was submitted concurrently with this study document, soil samples were analyzed for chlorethoxyphos, trichloroacetic acid, plus the oxon analog of chlorethoxyphos, [IN-34158; phosphoric acid, diethyl (1,2,2,2-tetrachloroethyl) ester], because of its toxicity.

The soil samples from this study could not be analyzed for trichloroacetic acid because the samples were destroyed by fire.

The current terrestrial field dissipation study (Study 8, MRID 41736829) was conducted at four sites (North Carolina, Illinois, Iowa, and California) where chlorethoxyphos (Fortress, 10% G) was applied at 0.6 oz ai/1000 feet of row (0.5 lb ai/A) as a preemergence soil-incorporated band over-row application to plots of field corn in May or June of 1988. In the 0- to 3-inch soil depth, trichloroacetic acid was detected at maximums of 0.05 ppm at North Carolina, 0.07 ppm at Illinois, 0.23 ppm at Iowa, and 0.35 ppm at California (Tables III-VI). Downward movement of TCA into the 3- to 6-inch soil depth occurred at the North Carolina, Illinois, and Iowa sites; TCA did not leach below 3 inches at the California site.

The soil segments from the current terrestrial field dissipation study were also composited into a single soil sample according to depth and sampling interval. The registrant reported that the terrestrial field dissipation SEP sampling procedure, requiring three composite soil samples for each depth and sampling interval, was not available at the time the study was conducted, and that the results from the various test sites can be considered as multiple samples from the same site.

Fortress

Page_____ is not included in this copy.

Pages 225 through 254 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

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The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

DATA EVALUATION RECORD

STUDY 10

CHEM 129006 Chlorethoxyphos (DPX-43898)

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 42559238
Babicki, W.A., Jr., and R.V. Slates. 1991. Freezer storage stability of Fortress insecticide active ingredient DPX-43898 and its oxon analogue IN-34158 in soil. Supplement No. 1. Report No. AMR-1244-88. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

STUDY ID 41290609
Babicki, W.A., Jr., and R.V. Slates. 1989. Freezer storage stability of Fortress insecticide active ingredient DPX-43898 and its oxon analogue IN-34158 in soil. Report No. AMR-1244-88. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

STUDY ID 41290603
Babicki, W.A., Jr., G.F. Barber, and R.V. Slates. 1988. Residue method for determination of Fortress insecticide active ingredient DPX-43898 and its oxon analogue IN-34158 in soil by electron-capture gas chromatography. Report No. AMR-1194-88. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

DIRECT REVIEW TIME = 13

REVIEWED BY: L. Binari TITLE: Staff Scientist

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ORG: EFGWB/EFED/OPP

SIGNATURE: Kurt Poff

A study investigating the storage stability of chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester] and its oxon analog IN-34158 was previously submitted under MRID 41290609; a review date was not reported. At that time, the study authors indicated that the study was an interim report containing data for 1 year of a 3-year study, and additional data would be reported as available. This study presents the results from the original study plus results from additional storage intervals for up to 31 months posttreatment.

CONCLUSIONS:

Ancillary Study - Freezer Storage Stability

1. Freezer storage stability studies are not specifically required by Subdivision N guidelines.

2. Chlorethoxyphos [DPX-43898, phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester] was stable in soil stored frozen (-20 C) for 31 months. Phosphoric acid, diethyl (1,2,2,2-tetrachloroethyl) ester (IN-34158), the oxon analog of chlorethoxyphos, was found to degrade rapidly during the period between fortification of the soil samples and when the samples were actually frozen, but did not appear to degrade further during 31 months of frozen storage.

3. This study is scientifically sound. Based on the information provided by this study, soil samples containing chlorethoxyphos and IN-34158 may be stored frozen for up to 31 months prior to analysis.

4. If samples are stored frozen for longer than 31 months prior to analysis, additional storage stability information may be required.

METHODOLOGY:

Samples (50 g) of clay loam soil (20.4% sand, 44.4% silt, 35.2% clay, 4.2% organic matter, pH 6.2, CEC 33.6 meq/100 g) were placed in glass jars and treated at 0.10 ppm with either chlorethoxyphos [DPX-43898, phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; purity 98.4%, source not specified] or its oxon analog phosphoric acid, diethyl (1,2,2,2-tetrachloroethyl) ester (IN-34158; purity 92%, source not specified) dissolved in hexane:acetone (95:5, v:v). The jars were capped, and four treated soil samples for each test substance were taken immediately for time 0 samples. The remaining soil samples were stored frozen at -20 C. Duplicate soil samples for each test substance were collected for analysis at approximately 1, 3, 6, 12, 18, 24, and 31 months posttreatment.

Soil samples were extracted with hexane:acetone (1:1, v:v) using a mechanical tumbler for 2 hours. The samples were centrifuged, and the extracts were decanted. The extracts were partitioned twice with

deionized water; the aqueous phases were discarded. An aliquot of the remaining hexane phase was filtered (0.5 um), then analyzed for chlorethoxyphos and IN-34158 using GC with electron-capture detection; the limit of quantitation for both compounds was 0.01 ppm. Recovery efficiencies from soil samples freshly fortified at 0.1 ppm ranged from 86 to 115% (mean 102%) of the applied for chlorethoxyphos and 86 to 110% (mean 98%) for IN-34158 (Tables I and II). It was not reported if results were corrected for recovery efficiency and/or soil moisture content.

DATA SUMMARY:

Chlorethoxyphos was stable in clay loam soil that was treated with chlorethoxyphos [DPX-43898; phosphorothioic acid, O,O-diethyl O-(1,2,2,2-tetrachloroethyl) ester; purity 98.4%] at 0.1 ppm and stored frozen (-20 C) for 31 months. At 31 months posttreatment, parent chlorethoxyphos comprised 89-97% of the applied (Table I). During the study, recovery of chlorethoxyphos from stored soil samples ranged from 87 to 110% (mean 99%) of the applied.

The oxon analog of chlorethoxyphos, phosphoric acid, diethyl (1,2,2,2-tetrachloroethyl) ester (IN-34158), was found to degrade rapidly during the period between fortification of the soil samples with IN-34158 (purity 92%) at 0.1 ppm and when the soil was actually frozen; however, IN-34158 did not appear to degrade further during frozen storage (see Comment 1). IN-34158 decreased from 107-113% of the applied at day 0 posttreatment to 63-67% in soil samples stored 35-42 days, then ranged from 43 to 60% in soil stored 3-31 months (Table II).

COMMENTS:

1. The study authors believed that the 45% decrease in IN-34158 that appeared to occur during frozen storage was due to microbial degradation, and that the degradation would not have occurred if the soil was frozen "immediately". The study authors reported that the sealed jars containing the soil samples fortified with IN-34158 were placed in boxes in the freezer, and theorized that the samples may not have frozen quickly enough, allowing for microbial degradation. To investigate this, two additional experiments were performed. In the first experiment, six additional clay loam soil samples (50 g) were moistened to 75% of field capacity, treated at 0.10 ppm with IN-34158, and incubated in darkness at room temperature; duplicate samples were taken for analysis at 0, 1, and 7 days posttreatment. IN-34158 decreased to 30-40% of the applied at 1 day posttreatment and was not detected at 7 days; recovery efficiencies from soil samples freshly fortified at 0.1 ppm ranged from 90 to 110% of the applied (Table III).

In the second experiment, two additional soil samples were moistened to 75% of field capacity, then stored frozen (-20 C) for 3 days. After 3 days of frozen storage, the soil samples were treated at 0.10 ppm with IN-34158 and returned to frozen storage. At 8 days posttreatment, IN-34158 comprised 100% of the applied (Table IV).

2. The same clay loam soil was used in both the chlorethoxyphos and trichloroacetic acid (TCA) freezer storage stability studies; therefore, the soil CEC value was obtained from the TCA storage stability study (Study 13, MRID 41290612).

3. The registrant reported that chlorethoxyphos (DPX-43898) is an organophosphorus insecticide used for control of corn root worms and other soil insects and is currently being developed for use on corn. The proposed use rate was reported as 0.3 oz ai/1000 feet of row (0.25 lb ai/A assuming 40-inch row spacing; MRID 41736820).

Fortress

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Pages 260 through 265 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

X FIFRA registration data.

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DATA EVALUATION RECORD

STUDY 11

CHEM 129006 Chlorethoxyphos
(DPX-43898)

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 42559239
Slates, R.V. 1991. Freezer storage stability of trichloroacetic acid in soil. Supplement No. 1. Report No. AMR-1342-88. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

STUDY ID 41290612
Barber, G.F. 1989c. Freezer storage stability of trichloroacetic acid in soil. Report No. AMR-1342-88. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

STUDY ID 41290605
Barber, G.F. 1989b. Analytical method for the determination of residues of trichloroacetic acid in crops and soil. Report No. AMR-1253-88. Unpublished study performed and submitted by E.I. du Pont de Nemours and Company, Inc., Wilmington, DE.

DIRECT REVIEW TIME = 14

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A study investigating the storage stability of trichloroacetic acid (TCA) was previously submitted under MRID 41290612; a review date was not reported. At that time, the study author indicated that the

study was an interim report containing data for 4 months of a 3-year study, and additional data would be reported as available. This study presents the results from the original study plus results from additional storage intervals of up to approximately 23 months posttreatment.

CONCLUSIONS:

Ancillary Study - Freezer Storage Stability

1. Freezer storage stability studies are not specifically required by Subdivision N guidelines.

2. Trichloroacetic acid was stable in clay loam soil stored frozen (-20 C) for up to 23 months.

3. This study is scientifically sound. Based on the information provided by this study, soil samples containing trichloroacetic acid may be stored frozen up to 23 months prior to analysis.

4. If samples are stored frozen for longer than 23 months prior to analysis, additional storage stability information may be required.

METHODOLOGY:

Samples (50 g) of clay loam soil (20.4% sand, 44.4% silt, 35.2% clay, 4.2% organic matter, pH 6.2, CEC 33.6 meq/100 g) were weighed into polyethylene centrifuge bottles and treated at 0.10 ppm with trichloroacetic acid (TCA; purity 99%, source not specified) dissolved in water. The bottles were capped, and the treated soil samples were stored frozen at -20 C. Duplicate soil samples were collected for analysis at 0, 30, 111, 197, 279, 384, 568, and 691 days posttreatment.

Soil samples (50 g) were extracted with methanol:water (95:5, v:v) using a sonication bath for 20-30 minutes, then rinsed twice with methanol:water by vigorous shaking for 30 seconds. Between each extraction or rinse, the samples were centrifuged and the supernatant decanted; the extracts and rinses were combined. An aliquot (40 mL) of the combined solution was placed in a silanized tube, treated with 10% aqueous sodium acetate solution, and concentrated. The concentrated extract was partitioned twice with hexane; hexane phases were discarded. The remaining aqueous phase was transferred to another silanized tube using methanol; water was removed from the sample by dilution with methanol followed by evaporation under nitrogen, the procedure was repeated six times. The resulting concentrate was derivatized to the methyl ester in the presence of methanol and sulfuric acid at 60 C for 2 hours. The derivatized sample was cooled to room temperature, then diluted with hexane and 10% aqueous sodium chloride solution. The organic (hexane) phase

containing TCA methyl ester was applied to a silica gel clean-up column and eluted with hexane. An aliquot of the eluate was analyzed for TCA methyl ester using GC with electron-capture detection; the limit of quantitation was 0.01 ppm. Recovery efficiencies from soil samples freshly fortified (treatment rate unspecified) with TCA ranged from 75 to 90% (mean 82%) of the applied (Table I). It was not reported if results were corrected for recovery efficiency and/or soil moisture content.

DATA SUMMARY:

Trichloroacetic acid was stable in clay loam soil that was treated with trichloroacetic acid (TCA, purity 99%) at 0.1 ppm and stored frozen (-20 C) for up to 23 months. At 23 months (691 days) posttreatment, parent TCA comprised 81-84% of the applied (Table I). During the study, recovery of TCA from stored soil samples ranged from 73 to 92% (mean 81%) of the applied.

COMMENTS:

Trichloroacetic acid was determined to be a primary soil degradate of chlorethoxyphos in terrestrial field dissipation studies (Study 8, MRID 41736829). This study was submitted to contribute towards the fulfillment of data requirements for chlorethoxyphos.

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Fortress

Page_____ is not included in this copy.

Pages 270 through 272 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

X FIFRA registration data.

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The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY
CHLORETHOXYPHOS
Last Update on December 14, 1993
[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

LOGOUT	Reviewer:	Section Head:	Date: JAN 1 0 1994

Common Name:CHLORETHOXYPHOS
Smiles Code:
PC Code # :129006 CAS #:54593-83-8 Caswell #:

Chem. Name :phosphorothiotic acid, O,O-diethyl O-(1,2,2,2-tetrachloro ethyl) ester
Action Type:Soil insecticide;controls corn rootworm and other soil pests

Trade Names:Fortress
(Formul'tn):Granular
Physical State: liquid

Use :Terrestrial Food uses
Patterns :proposed use rate is 0.3oz ai/1000 ft row = 0.25 lb/ai/acre
(% Usage) :
:

Empirical Form: $C_6H_{11}Cl_4O_3PS$
Molecular Wgt.: 336.00 Vapor Pressure: 1.70E -3 Torr
Melting Point : °C Boiling Point: 105 .8torr°C
Log Kow : 4.59 pKa: @ °C
Henry's : 3.50E -4 Atm. M3/Mol (Measured) 3.58E -4 (calc'd)

Solubility in ...			Comments
Water	2.10E	ppm @25.0 °C	
Acetone	E	ppm @ °C	
Acetonitrile	E	ppm @ °C	
Benzene	E	ppm @ °C	
Chloroform	E	ppm @ °C	
Ethanol	E	ppm @ °C	
Methanol	E	ppm @ °C	
Toluene	E	ppm @ °C	
Xylene	E	ppm @ °C	
	E	ppm @ °C	
	E	ppm @ °C	

Hydrolysis (161-1)
[A] pH 5.0:72 days,chloral hydrate major degradate.
[A] pH 7.0:59 days,chloral hydrate major degradate.
[A] pH 9.0:4.3 days,dichloroacetic acid major degradate.
[ ] pH :
[ ] pH :
[ ] pH :

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Photolysis (161-2, -3, -4)
[A] Water:27 days (uncorrected for light intensitiy or continuous rad.
[ ] :@ pH 5 Xenon light, chloral hydrate and DCA major deg.
[ ] :Dark control half-life was 89 days, corresponding to hydro.
[ ] :

[A] Soil :21 days nat.sun.chloral hydrate,polars,CO2 degradates.
[ ] Air :

Aerobic Soil Metabolism (162-1)
[A] 20 day half-life in a static system. Major degradates were CO2
[ ] chloral hydrate.
[ ] In a flow through system, half-life was 7 days. Volatilization
[ ] contributed most to dissipation. Some chloral hydrate and CO2
[ ] was detected.
[ ]
[ ]

Anaerobic Soil Metabolism (162-2)
[S] Half-life was 41-47 days in clay soil. Only chlorethoxyphos
[ ] was identified in the soil and floodwater. CO2 was the major
[ ] degradate totalling 36.5% at 62 days incubation.
[ ]
[ ]
[ ]
[ ]

Anaerobic Aquatic Metabolism (162-3)
[ ]
[ ]
[ ]
[ ]
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[ ]
[ ]

Aerobic Aquatic Metabolism (162-4)
[ ]
[ ]
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[ ]
[ ]

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Soil Partition Coefficient (Kd) (163-1)

[A]	Soil	OM%	Kdads	Kddes
[ ]	Hanford SdLm	1.0	40	7.5
[ ]	North Car.LmSd	2.0	53	8.2
[ ]	Fargo StLm	4.3	150	5.3
[ ]	LaHogue Lm	5.0	200	5.2

[ ] Parent binds strongly to soil.

Soil Rf Factors (163-1)
[A] Adsorption desorption literature indicate that chloral hydrate
[ ] and trichloroacetic acid have Kd values of 0.1 to 0.3.
[ ] Soil column leaching studies indicate that chloral hydrate
[ ] is easily leached from four test soils indicating high mobility
[ ] of degradates.
[ ]

Laboratory Volatility (163-2)
[ ]
[ ]

Field Volatility (163-3)
[ ]
[ ]

Terrestrial Field Dissipation (164-1)
[A] Half-life of 2.0 days in the top 3 inches in Iowa
[A] Half-life of 2.6 days in Illinois
[A] Half-life of 14-35 days in Illinois
[A] Half-life of 14-48 days in North Carolina in top 3 inches.
[A] Half-life of 7-14 days in California.
[ ] Parent was detected in the 12 to 18 inch depth in N.C.
[ ] Parent was detected in the 18 to 24 inch depth in Iowa
[ ] Parent did not leach below 3 inches in CA
[ ] The oxon analog was detected in Illinois and Iowa, the major
[ ] degradate detected in all sites was trichloroacetic acid.

Aquatic Dissipation (164-2)
[ ]
[ ]
[ ]
[ ]
[ ]
[ ]

Forestry Dissipation (164-3)
[ ]
[ ]

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Long-Term Soil Dissipation (164-5)
[ ]
[ ]

Accumulation in Rotational Crops, Confined (165-1)
[ ]
[ ]

Accumulation in Rotational Crops, Field (165-2)
[ ]
[ ]

Accumulation in Irrigated Crops (165-3)
[ ]
[ ]

Bioaccumulation in Fish (165-4)
[A] Channel Cat. exposed to 0.0047 mg/L parent showed BCF's 1100 mus.
[ ] 4000 vis., 2100 whole fish; 2wks depuration gave 31, 86,46 resp.

Bioaccumulation in Non-Target Organisms (165-5)
[ ]
[ ]

Ground Water Monitoring, Prospective (166-1)
[ ]
[ ]
[ ]
[ ]

Ground Water Monitoring, Small Scale Retrospective (166-2)
[ ]
[ ]
[ ]
[ ]

Ground Water Monitoring, Large Scale Retrospective (166-3)
[ ]
[ ]
[ ]
[ ]

Ground Water Monitoring, Miscellaneous Data (158.75)
[ ]
[ ]
[ ]

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Field Runoff (167-1)
[ ]
[ ]
[ ]
[ ]

Surface Water Monitoring (167-2)
[ ]
[ ]
[ ]
[ ]

Spray Drift, Droplet Spectrum (201-1)
[ ]
[ ]
[ ]
[ ]

Spray Drift, Field Evaluation (202-1)
[ ]
[ ]
[ ]
[ ]

Degradation Products

Dichloroacetic acid abiotic hydrolysis pH 7-9
Chloral hydrate abiotic hydrolysis pH 5-7
CO2 aerobic/anaerobic soil
Trichloroacetic acid aerobic soil and plant;found in terr.field.
Oxon analog; phosphoric acid, diethyl (1,2,2,2-tetrachlorehtyoxyph.
was detected in the field at Iowa and Illinois.
Based upon literature review, DETP diethylthiophosphate and DEP
diethylphosphate are the major degradates of the phosphoryl moiety.

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Comments

TCA was detected in the terrestrial field dissipation studies and is considered to be highly mobile. It is unclear if TCA can be metabolized microbially.

References: EFGWB#93-0258-0259
Writer : KLP