

DATA EVALUATION RECORD

STUDY 3

CHEM 081501 Choropicrin §163-2
 CAS No. 76-06-2
 FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 43798601

Skinner, W., and N. Jao. 1995. Laboratory volatility of [¹⁴C]chloropicrin. PTRL Report No.: 450W-1. PTRL Project No.: 450W. Unpublished study performed by PTRL West, Inc., Richmond, CA; and submitted by The Chloropicrin Manufacturers Task Force, c/o Niklor Chemical Company, Long Beach, CA.

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REVIEWED BY: J. M. Franti, B.S. Signature:
 TITLE: Scientist Date:
 M.K. Mahoney, M.S.
 Scientist

EDITED BY: C. A. Sutton, Ph.D. Signature:
 TITLE: Sr. Scientist/Asst. Project Manager Date:

APPROVED BY: P. H. Howard, Ph.D. Signature:
 TITLE: Project Manager Date:

ORG: Syracuse Research Corp.
 Arlington, VA 22202

TEL: 703/413-9369

APPROVED BY: Sid Abel
 TITLE: Environmental Scientist
 ORG: FMB/EFED/OPP
 TEL: 703/305-7346

SIGNATURE:



2012510

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CONCLUSIONS

Mobility - Laboratory Volatility

1. This study is scientifically valid and provides useful information on the volatility of chloropicrin from sandy loam soil (uncovered and tarp-covered).
2. This study meets Subdivision N Guidelines for the fulfillment of EPA data requirements on laboratory volatility.
3. Nonradiolabeled plus radiolabeled [¹⁴C]chloropicrin, at a nominal application rate of 300 ppm, volatilized from sandy loam soil (uncovered and tarp-covered) adjusted to 60% of 0.33 bar soil moisture content and incubated in darkness at 25.0 ± 1 °C for up to 8 days with an air flow (>90% relative humidity) rate of approximately 100 mL/min.

In the uncovered soil, the maximum volatility of the parent was 342 μg/cm²/hr and the maximum air concentration of the parent was 1.0 × 10⁴ μg × 10³/m³ (2-6 hour interval). Total [¹⁴C]volatiles accounted for 92.6% of the applied radioactivity at 8 days posttreatment (reviewer-calculated sum). Organic [¹⁴C]volatiles detected in the methanol, water vapor, and charcoal traps were 86.0%, 1.5%, and 0.08% of the applied radioactivity, respectively, at 8 days posttreatment; evolved ¹⁴CO₂ was 5.1% of the applied. The parent was initially (2 hours) present in the methanol trap at 0.8% of the applied radioactivity, increased to 16.3% by 6 hours and 65.4% by 24 hours, and was 81.5-85.5% at 46-116 hours posttreatment; data are reviewer-calculated means of cumulative volatilized parent. The degradate dichloronitromethane was detected in the methanol trap at 0.4% of the applied radioactivity. In the soil, extractable and nonextractable [¹⁴C]residues were 0.4% and 4.3% of the applied radioactivity, respectively, at 8 days posttreatment.

In the tarp-covered soil, the maximum volatility of the parent was 205 μg/cm²/hr and the maximum air concentration of the parent was 5918 μg × 10³/m³ (23-25 hour interval; interval in which the tarp was punctured). Total [¹⁴C]volatiles accounted for 89.3% of the applied radioactivity at 8 days posttreatment (reviewer-calculated sum). Organic [¹⁴C]volatiles detected in the methanol, water vapor, and charcoal traps were 79.9%, 1.2%, and 0.09% of the applied radioactivity, respectively, at 8 days posttreatment; evolved ¹⁴CO₂ was 8.2% of the applied. The parent was initially (4 hours) present in the methanol trap at 1.9% of the applied radioactivity, increased to 38.7% by 23 hours and 67.2% by 47 hours, and was 79.1% at 144 hours posttreatment; data are reviewer-calculated means of cumulative volatilized parent. The degradate dichloronitromethane was detected in the methanol trap at 0.7% of the applied radioactivity. In the soil, extractable and nonextractable [¹⁴C]residues were 1.1% and 6.1% of the applied radioactivity, respectively, at 8 days posttreatment.

METHODOLOGY

Samples (5020 g) of sieved (2 mm) sandy loam soil (Watsonville, CA; 53.2% sand, 27.2% silt, 19.6% clay, 1.27% organic matter, pH 7.2, CEC 12.5 meq/100 g; Table II, p. 34) were weighed into glass columns (15 cm i.d.; 30 cm in height) equipped with gas inlet/outlet ports, adjusted to 60% of 0.33 bar soil moisture content, and pre-incubated at 25°C for 4-5 days prior to treatment (pp. 16, 18). At six inches below the soil surface, each column was treated with nonradiolabeled plus radiolabeled [¹⁴C]chloropicrin (trichloronitromethane; radiochemical purity 99.0%, specific activity 7.58 mCi/mmol; p. 15; Figure 1, p. 48), dissolved in acetonitrile, at a nominal application rate of 300 ppm (p. 17). The columns were covered and incubated in a 25 ± 1°C water bath at an average air flow rate of approximately 100 mL/min for 8 days (p. 12). The temperature, flow rate, and air humidity were measured daily (p. 17); data were reported in Tables IV and V (pp. 36-37). To trap [¹⁴C]volatiles, moist, filtered air was pumped across the top of the column and into the following series of successive traps: water vapor, three methanol, activated charcoal, and two 10% KOH traps (Figure 3, p. 52). Additional sample columns were prepared as previously described except that the soil surface was covered with a tarp for 48 hours following treatment (p. 19); the tarp was punctured (20 times with a 16-gauge needle) at 24 hours posttreatment. The volatile traps were removed from the uncovered soil for analysis at 2, 6, 24, 46, 74, 116, and 188 hours posttreatment; and from the tarp-covered soil at 4, 23, 25, 28, 47, 52, 72, 116, 144, and 188 hours posttreatment. The soil was removed from each column for analysis at 8 days posttreatment.

At 8 days posttreatment, triplicate subsamples of each soil were extracted with acetonitrile and centrifuged (p. 19). The soil samples were further extracted with acetone followed by acetonitrile. The extracts from the uncovered soil samples were analyzed separately for total radioactivity by LSC. The extracts from the tarp-covered soil extracts were combined and analyzed by LSC. The combined soil extracts (both soils) were analyzed by HPLC (Supelco C18 column) using a mobile phase gradient of water:acetonitrile (100:0 to 0:100, v:v) with UV (220 nm) and radioactive flow detection (p. 21); the limit of detection was twice background (0.008 ppm; p. 23). Eluent fractions were analyzed by LSC (p. 22). Samples were co-chromatographed with nonradiolabeled reference standards of the parent and the potential degradate nitromethane. Triplicate subsamples of post-extracted soil were analyzed by LSC following combustion (p. 20); the limit of detection was twice background (0.1 ppm; p. 23).

At each sampling interval, triplicate aliquots of the methanol and KOH trapping solutions were analyzed for total radioactivity by LSC (p. 19). The ¹⁴CO₂ in KOH traps was precipitated with BaCl₂ and the supernatant was analyzed by LSC (p. 22); results indicated that >99.6% of the applied radioactivity in the traps was ¹⁴CO₂ (p. 27). The water vapor traps were periodically defrosted by rinsing with methanol, and triplicate aliquots of the rinsate were analyzed by LSC. [¹⁴C]Residues in the methanol (those

containing >0.1% of the applied radioactivity; p. 27) and water vapor traps were analyzed by HPLC as previously described (p. 27). To confirm the identity of the degradate dichloronitromethane, samples of the methanol trap solution were further analyzed by GC (Stabilwax column) with mass selective detection in the electron impact mode (pp. 20, 27). The charcoal traps were extracted with acetone:water (1:1, v:v), and triplicate aliquots of the extracts were analyzed by LSC. The remaining post-extracted charcoal pellets were analyzed by LSC following combustion.

To determine viability, soil samples were serially diluted, plated on selective media, and examined for colony forming units (p. 16); results indicated that the soils were viable (Table III, p. 35).

DATA SUMMARY

Nonradiolabeled plus radiolabeled [^{14}C]chloropicrin (radiochemical purity 99.0%), at a nominal application rate of 300 ppm, volatilized from sandy loam soil (uncovered and tarp-covered) adjusted to 60% of 0.33 bar soil moisture content and incubated in darkness at $25 \pm 1^\circ\text{C}$ for up to 8 days with an air flow (>90% relative humidity) rate of approximately 100 mL/min (pp. 26, 27; Figures 10-11, pp. 68-69).

In the uncovered soil, the maximum volatility of the parent compound was $342 \mu\text{g}/\text{cm}^2/\text{hr}$ and the maximum air concentration of the parent was $1.0 \times 10^4 \mu\text{g} \times 10^3/\text{m}^3$ (2-6 hour interval; Table XIII, p. 45). Total [^{14}C]volatiles accounted for 92.6% of the applied radioactivity at 8 days posttreatment (reviewer-calculated sum; Table VI, p. 38). Organic [^{14}C]volatiles detected in the methanol, water vapor, and charcoal traps were 86.0%, 1.5%, and 0.08% of the applied radioactivity, respectively, at 8 days posttreatment; evolved $^{14}\text{CO}_2$ was 5.1% of the applied. The parent compound was initially (2 hours) present in the methanol trap at 0.8% of the applied radioactivity, increased to 16.3% by 6 hours and 65.4% by 24 hours, and was 81.5-85.5% of the applied at 46-116 hours posttreatment (Table XI, p. 43); data are reviewer-calculated means of cumulative volatilized parent. The degradate dichloronitromethane was detected in the methanol trap at 0.4% of the applied radioactivity. In the soil, extractable and nonextractable [^{14}C]residues were 0.4% and 4.3% of the applied radioactivity, respectively, at 8 days posttreatment.

In the tarp-covered soil, the maximum volatility of the parent compound was $205 \mu\text{g}/\text{cm}^2/\text{hr}$ and the maximum air concentration of the parent was $5918 \mu\text{g} \times 10^3/\text{m}^3$ (23-25 hour interval; interval in which the tarp was punctured; Table XII, p. 44). Total [^{14}C]volatiles accounted for 89.3% of the applied radioactivity at 8 days posttreatment (reviewer-calculated sum; Table VI, 38). Organic [^{14}C]volatiles detected in the methanol, water vapor, and charcoal traps were 79.9%, 1.2%, and 0.09% of the applied radioactivity, respectively, at 8 days posttreatment; evolved $^{14}\text{CO}_2$ was 8.2% of the

applied. The parent compound was initially (4 hours) present in the methanol trap at 1.9% of the applied radioactivity, increased to 38.7% by 23 hours and 67.2% by 47 hours, and was 79.1% of the applied at 144 hours posttreatment (Table X, p. 42); data are reviewer-calculated means of cumulative volatilized parent. The degradate dichloronitromethane was detected in the methanol trap at 0.7% of the applied radioactivity. In the soil, extractable and nonextractable [^{14}C]residues were 1.1% and 6.1% of the applied radioactivity, respectively, at 8 days posttreatment.

Material balances (based on LSC analysis of individual replicates) were 96.6-98.1% of the applied radioactivity for the uncovered soil, and were 96.0-97.0% of the applied for the tarp-covered soil (Table VI, p. 38).

COMMENTS

1. Trapping efficiencies were not reported for the charcoal, methanol, and KOH traps. The reviewer noted that the material balances were within the reasonable range of 90-110% (Table VI, p. 38).
2. Application rates were verified by injecting an aliquot of the dosing solution into a flask containing acetonitrile and analyzing the solution by LSC (p. 18); the final application rate (302 and 303 ppm for uncovered and tarp-covered soils, respectively) was calculated using known weights of both the applied pesticide and the amount of treated soil. Time 0 analysis of the treated soil was not conducted.
3. The limits of detection were reported, but the limits of quantitation were not. Both limits of detection and quantitation should be reported to allow the reviewer to evaluate the adequacy of the method used for the determination of the test compound and its degradates.
4. The study authors stated that the parent compound was applied at the maximum proposed application rate (via subsurface injection) of 500 lb a.i./A (300 ppm; p. 17).
5. The study authors stated that the soil in the test vessels compacted during the first day of the study, and, therefore, required an addition of 6% more soil in order to maintain the column depth of 25.4 cm (pp. 16-17). The density of the soil in the vessels was 1.12 g/cm³.
6. The vapor pressure of the parent was reported to be 18.9 mm Hg at 20.6°C (p. 15).

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