

DATA EVALUATION RECORD

STUDY 5

PC No. 029001 1,3-Dichloropropene (In-Line) §163-3
CAS No. 542-75-6
DP Barcode D267814, D267853
FORMULATION-12-EMULSIFIABLE CONCENTRATE
STUDY ID 45112902

van Wesenbeeck, I., and A.M. Phillips. 2000. Field volatility of 1,3-dichloropropene and chloropicrin from surface drip irrigation application of In-Line to vegetable beds under polyethylene tarp. Laboratory Study ID. 990072. Unpublished study performed by Global Environmental Chemistry Laboratory - Indianapolis Lab, Dow AgroSciences, LLC, Indianapolis, IN; Paragon Research, Fresno, CA, and A&L Great Lakes Laboratories, Inc., Fort Wayne, IN. Study was submitted by Dow AgroSciences LLC, Indianapolis, IN.

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There have been changes to contractor's DER	
There have not been changes to contractor's DER	



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CONCLUSIONS

This study is considered scientifically valid. However, the study does not meet Subdivision N Guidelines for the fulfillment of EPA data requirements on field volatility because the test substance was not added to the soil at the highest recommended label rate for a single application.

ABSTRACT

Mobility - Field Volatility

The volatilization of 1,3-dichloropropene (*cis/trans*-1,3-dichloropropene; In-Line, 60.2% as 1,3-dichloropropene plus 33.0% as chloropicrin) under field conditions was studied following an application of 24.6 gallons/A (166.12 lb a.i./A) by surface drip irrigation to a fallow plot of sand soil in Douglas, Georgia. Volatilized 1,3-dichloropropene was collected near the center of the treated plot at 15, 33, 55, 90, and 150 cm above the bed top, and at 100 and 300 feet (1.5 m height) from the center of the treated plot on three sides of the plot. Volatilized 1,3-dichloropropene was also collected using active flux chambers that sat on the soil surface. Air samples were collected on a 5-hour/5-hour/14-hour schedule (7 am, noon, and 5 pm daily) for 14 days after application. Air concentrations ($\mu\text{g}/\text{m}^3$) were corrected for mean recovery efficiencies. Volatilization ($\mu\text{g}/\text{m}^2/\text{hour}$) was measured using the aerodynamic flux density (33- and 90-cm sampling heights) and active flux chamber (<8-cm sampling height) methods. Soil gas was sampled once per day to a depth of 36 inches, and was a maximum of 4668 mg/m³ 1 day after treatment. Samples were analyzed for *cis*- and *trans*-dichloropropene using GC/MS.

The concentration of 1,3-dichloropropene in the air decreased with increasing height, and concentrations measured off-site (100 and 300 m) were more dependent on direction than on distance. During Event 3 (the 14 hours following the end of treatment), 1,3-dichloropropene was 643.9 $\mu\text{g}/\text{m}^3$ at 15 cm, 518.1 $\mu\text{g}/\text{m}^3$ at 33 cm, 385.5 $\mu\text{g}/\text{m}^3$ at 55 cm, 314.7 $\mu\text{g}/\text{m}^3$ at 90 cm, and 211.3 $\mu\text{g}/\text{m}^3$ at 150 cm. At this same interval, concentrations on the North side of the plot were 8.0 and 2.6 $\mu\text{g}/\text{m}^3$ at 100 and 300 m, respectively, on the South side were 233.9 and 112.4 $\mu\text{g}/\text{m}^3$, and on the West side were 138.9 and 7.1 $\mu\text{g}/\text{m}^3$. Air concentrations tended to be higher during the 14-hour overnight interval. For example, at a height of 33 cm, 1,3-dichloropropene was 518.1 $\mu\text{g}/\text{m}^3$ after the first night (5-19 hours following treatment), 225.0 $\mu\text{g}/\text{m}^3$ after 19-24 hours, 234.4 $\mu\text{g}/\text{m}^3$ after 24-29 hours, and 582.9 $\mu\text{g}/\text{m}^3$ after the second night (29-43 hours). Overnight air concentrations in the center of the plot did not decrease to <100 $\mu\text{g}/\text{m}^3$ (33-cm height) until the sixth night after treatment (Event 18). The multi-directional 14-day average concentration of 1,3-dichloropropene (90 cm height) was 52.9 $\mu\text{g}/\text{m}^3$ on-site, 10.2 $\mu\text{g}/\text{m}^3$ at 100 feet from center, and 5.0 $\mu\text{g}/\text{m}^3$ at 300 feet from center.

Using the aerodynamic flux method, it was estimated that the total mass of 1,3-dichloropropene lost from the soil was 29.2% of the applied. Volatilization was a maximum of 72.4 mg/m²hr at 0-5 hours ("Event 2", concurrent with treatment). Overnight flux concentrations were 56.1 mg/m²hr after 1 day (24-29 hours), and 28.6-

33.2 mg/m²hr after 2 and 3 days, varied from 4.7-15.9 mg/m²hr after 4-6 days, 3.7-5.1 mg/m²hr after 7-10 days, and 1.0-1.5 mg/m²hr after 11-13 days. Daytime (7 am-5 pm) flux concentrations were <10 mg/m²hr by 3 days.

Using the active flux chamber method, it was estimated that the total mass of 1,3-dichloropropene lost from the soil was 20.5% of the applied. 1,3-Dichloropropene flux concentrations were highest over the polyethylene tarp-covered beds, decreasing from 116.8-416.5 mg/m²hr at 5 hours following the start of application to 23.9-49.2 mg/m²hr at 24 hours, and were generally <10 mg/m²hr after 3 days, <5 mg/m²hr after 5 days, and <1 mg/m²hr after 9 days. Flux concentrations over an uncovered bed decreased from 53.1 mg/m²hr at 5 hours following the start of application to 28.2 mg/m²hr at 1 day, 12.8 mg/m²hr after 2 days, 6.4 mg/m²hr after 5 days, and ≤1.6 mg/m²hr after 7 days. Flux concentrations over the furrows were <1 mg/m²hr at all sampling intervals, with the exception of 6.4 mg/m²hr in one of two chambers at 6 days posttreatment.

MATERIALS AND METHODS

1,3-Dichloropropene¹ (*cis*-/*trans*-1,3-dichloropropene; In-Line, 60.2% as 1,3-dichloropropene plus 33.0% as chloropicrin; Formulation No. EF1499, TSN 101998, Lot No. GHD-3698/23A, Dow AgroSciences LLC, p. 25) was applied at 24.6 gallons/A (166.12 lb a.i./A, p. 32) by surface drip irrigation to a fallow plot (4.33 treated acres, slope 1-5%, depth to water table not reported) of sand soil (0- to 15-cm depth, 86.2% sand, 7.7% silt, 3.6% clay, 0.5% organic matter, pH 6.9, CEC 1.8 meq/100 g; Table 1, p. 60) located in Douglas, Georgia, on December 6, 1999 (pp. 26-28, 31-33). The fields were laid out in 32-inch beds on 60-inch centers; a permanent drip tape located on the soil surface at the center of the beds was covered with 1.25 mil black polyethylene (p. 27). The drip tape contained emitters at 12-inch spacings (p. 31). 1,3-Dichloropropene was injected into the charged line from approximately 12:22 pm until 5:03 pm, then the irrigation system was flushed with water (p. 31). Water samples (4/interval) were collected from the ends of the drip lines 100 and 200 minutes after the start of irrigation; the average concentration of 1,3-dichloropropene in the irrigation water was 777.8 ± 11.5 µg/mL (pp. 31-32; Table 3, p. 62).

To collect volatilized 1,3-dichloropropene using the aerodynamic flux method, air was continuously drawn (flow rate approximately 1.5 L/minute) through an air sampler (activated charcoal adsorption tube) using a battery-operated vacuum pump (p. 33). Five air samplers were placed vertically at 15, 33, 55, 90, and 150 cm above ground level on a pole (flux mast) located at the center of the treated plot (sampler locations A-E, respectively; p. 28; Figures 1-2, pp. 88-89). Six off-site poles, each equipped with one air sampler positioned 150 cm above ground level, were placed along north-south and east-west transects through the treated plot center at distances of approximately 20 feet

¹ Vapor pressure (25°C) reported as 34.3 mm Hg (*cis*) and 23.0 mm Hg (*trans*, p. 23). Aqueous solubility (25°C) reported as 2180 mg/L (*cis*) and 2320 mg/L (*trans*).

(sampler location I), 100 feet (sampler locations F, H), 150 feet (sampler location M), and 300 feet (sampler locations J, K) from the edge of the treated plot. No samplers were placed along the east side of the plot due to the presence of the irrigation pond. Pretreatment air samples were collected on-site during a 6-hour interval on the day before treatment ("Event 0", p. 34). Air sampling was re-initiated at noon, shortly before treatment began (12:22 pm); "Event 2", from noon until 5 pm, spanned the entire application period. On-site charcoal adsorption tubes were collected and replaced at approximately 7 am, 12 noon, and 5 pm daily ("Events") for 14 days; off-site charcoal adsorption tubes were changed to a 24-hour cycle after 7 days (p. 34).

To collect volatilized 1,3-dichloropropene using the active flux chamber method, five metal flux chambers (41 x 41 cm, 8 cm height) were placed in the plot (pp. 29-31; Figures 5-6, pp. 92, 93). Two chambers were placed over the center of the beds on top of the tarp, two over the center of the furrows on the soil, and one over the center of a bed from which the tarp had been removed. The base of the chambers was pressed into the soil (2.5-cm depth) to form an air-tight seal. Air was drawn into the chamber and through (1.5 L/minute) charcoal sampling tubes. Sampling intervals were the same as those used in the aerodynamic flux portion of the experiment.

Upon collection, the ends of the charcoal adsorption tubes were sealed, and the tubes were transported in a portable cooler to frozen storage (p. 33). The air flow rate of the samplers was determined and re-adjusted, if needed, using an electronic flow meter each time a charcoal adsorption tube was installed and replaced.

Soil moisture (15-cm intervals to a depth of 60 cm), soil temperatures (15-cm intervals to a depth of 75 cm), air temperatures (1.5-m height), relative humidity (33- and 90-cm heights), barometric pressure (1.5-m height), precipitation, total solar radiation, and wind velocity and direction (15-, 33-, 55-, and 90-cm height) were measured at the site (pp. 28, 35-36; Table 2, p. 61, Figures 17-18, pp. 104-105). Soil gas samples were collected daily using soil gas probes on the surface of the soil beneath the tarp, directly above the tarp, at depths of 15 and 60 cm below the center of the bed, and at depths 15 and 30 cm below the center of the furrow (p. 36).

The charcoal samplers were analyzed according to Dow AgroSciences Method C0785. Charcoal samples from the front and back half of each adsorption tube were removed, then extracted with hexane:methanol:ethyl acetate (98:1:1, v:v:v) by mechanical shaking for 20 minutes (p. 37). Extracts were separated from charcoal by centrifugation and aliquots were analyzed for *cis*- and *trans*-1,3-dichloropropene by GC/MS; GC conditions were not described. Limits of detection and quantitation were 0.03 and 0.10 $\mu\text{g}/\text{tube}$, respectively. Recovery efficiencies from charcoal adsorption tubes fortified with *cis*- and *trans*-1,3-dichloropropene at 0.10-5000 $\mu\text{g}/\text{tube}$ averaged $87 \pm 5\%$ (72-100%, $n = 86$) of the applied for *cis*-1,3-dichloropropene and $79 \pm 4\%$ (68-88%, $n = 86$) for *trans*-1,3-dichloropropene (Table 5, pp. 144-146). Results were corrected for mean recovery efficiencies (p. 130). Air concentrations ($\mu\text{g}/\text{m}^3$) of 1,3-dichloropropene were calculated as the sum of the concentrations of *cis*- and *trans*-1,3-dichloropropene detected in an

adsorption tube divided by the volume of air pulled through that tube. Volatilization ($\mu\text{g}/\text{m}^2/\text{hour}$) of 1,3-dichloropropene was calculated using various methods (pp. 40-43).

Water samples were analyzed using Dow AgroSciences Method GRM 94.11. 1,3-Dichloropropene was purged from the water by sparging with helium, then captured on a Tenax trap. The trap was heated and backflushed with helium, and the 1,3-dichloropropene was desorbed and transferred to a GC with MS detection. Recovery efficiencies from water fortified with *cis*- and *trans*-1,3-dichloropropene at 0.05-1,000,000 ng/mL averaged $109 \pm 13\%$ of the applied for *cis*-1,3-dichloropropene and $110 \pm 13\%$ for *trans*-1,3-dichloropropene (Table 7, p. 149)

RESULTS/DISCUSSION

The volatilization of 1,3-dichloropropene (*cis/trans*-1,3-dichloropropene; In-Line, 60.2% as 1,3-dichloropropene plus 33.0% as chloropicrin) under field conditions was studied following an application of 24.6 gallons/A (166.12 lb a.i./A) by surface drip irrigation to a fallow plot of sand soil in Douglas, Georgia. Volatilized 1,3-dichloropropene was collected near the center of the treated plot at 15, 33, 55, 90, and 150 cm above the bed top, and at 100 and 300 feet (1.5 m height) from the center of the treated plot on three sides of the plot. Air samples were collected on a 5-hour/5-hour/14-hour schedule (7 am, noon, and 5 pm daily) for 14 days after application. Air concentrations ($\mu\text{g}/\text{m}^3$) were corrected for mean recovery efficiencies. Volatilization ($\mu\text{g}/\text{m}^2/\text{hour}$) was measured using the aerodynamic flux density (33- and 90-cm sampling heights) and active flux chamber (<8-cm sampling height) methods. Soil gas was sampled once per day to a depth of 36 inches, and was a maximum of 4668 mg/m³ 1 day after treatment (Figure 14, p.101). Samples were analyzed for *cis*- and *trans*-dichloropropene using GC/MS.

The concentration of 1,3-dichloropropene in the air decreased with increasing height, and concentrations measured off-site (100 and 300 m) were more dependent on direction than on distance (Table 4, pp. 63-72). During Event 3 (the 14 hours following the end of treatment), 1,3-dichloropropene was $643.9 \mu\text{g}/\text{m}^3$ at 15 cm, $518.1 \mu\text{g}/\text{m}^3$ at 33 cm, $385.5 \mu\text{g}/\text{m}^3$ at 55 cm, $314.7 \mu\text{g}/\text{m}^3$ at 90 cm, and $211.3 \mu\text{g}/\text{m}^3$ at 150 cm. At this same interval, concentrations on the North side of the plot were 8.0 and $2.6 \mu\text{g}/\text{m}^3$ at 100 and 300 m, respectively, on the South side were 233.9 and $112.4 \mu\text{g}/\text{m}^3$, and on the West side were 138.9 and $7.1 \mu\text{g}/\text{m}^3$. Air concentrations tended to be higher during the 14-hour overnight interval. For example, at a height of 33 cm, 1,3-dichloropropene was $518.1 \mu\text{g}/\text{m}^3$ after the first night (5-19 hours following treatment), $225.0 \mu\text{g}/\text{m}^3$ after 19-24 hours, $234.4 \mu\text{g}/\text{m}^3$ after 24-29 hours, and $582.9 \mu\text{g}/\text{m}^3$ after the second night (29-43 hours). Overnight air concentrations in the center of the plot did not decrease to $<100 \mu\text{g}/\text{m}^3$ (33-cm height) until the sixth night after treatment (Event 18). The multi-directional 14-day average concentration of 1,3-dichloropropene (90 cm height) was $52.9 \mu\text{g}/\text{m}^3$ on-site, $10.2 \mu\text{g}/\text{m}^3$ at 100 feet from center, and $5.0 \mu\text{g}/\text{m}^3$ at 300 feet from center (Table 9, p. 87).

Using the aerodynamic flux method, it was estimated that the total mass of 1,3-dichloropropene lost from the soil was 29.2% of the applied (95.4 kg; p. 46). Volatilization was a maximum of 72.4 mg/m²hr at 0-5 hours ("Event 2", concurrent with treatment; Table 6, p. 80). Overnight flux concentrations were 56.1 mg/m²hr after 1 day (24-29 hours), and 28.6-33.2 mg/m²hr after 2 and 3 days, varied from 4.7-15.9 mg/m²hr after 4-6 days, 3.7-5.1 mg/m²hr after 7-10 days, and 1.0-1.5 mg/m²hr after 11-13 days. Daytime (7 am-5 pm) flux concentrations were <10 mg/m²hr by 3 days.

Using the active flux chamber method, it was estimated that the total mass of 1,3-dichloropropene lost from the soil was 20.5% of the applied (p. 46). 1,3-Dichloropropene flux concentrations were highest over the polyethylene tarp-covered beds (FC #1 and #2), decreasing from 116.8-416.5 mg/m²hr at 5 hours following the start of application to 23.9-49.2 mg/m²hr at 24 hours, and were generally <10 mg/m²hr after 3 days, <5 mg/m²hr after 5 days, and <1 mg/m²hr after 9 days (Table 8, pp. 82-86). Flux concentrations over the uncovered bed decreased from 53.1 mg/m²hr at 5 hours following the start of application to 28.2 mg/m²hr at 1 day, 12.8 mg/m²hr after 2 days, 6.4 mg/m²hr after 5 days, and ≤1.6 mg/m²hr after 7 days. Flux concentrations over the furrows (FC #3 and 4) were <1 mg/m²hr at all sampling intervals, with the exception of 6.4 mg/m²hr in one of two chambers at 6 days posttreatment.

During the study, air temperatures (33 cm) ranged from 2.6 to 24.3°C and daily average wind velocity (33 cm) ranged from 0.1 to 3.1 m/second (Table 6, pp. 80-81).

DEFICIENCIES/DEVIATIONS

1. The air concentration data reported in Tables 4 and 6 are not in agreement, although they apparently include some of the same information (air concentrations at 33 and 90 cm, in μg/m³). The study author does not provide any information to suggest why data for the same sampling height/Event differ. There is not a consistent pattern to the differences; the concentrations reported in Table 6, which were used to calculate flux, are both higher and lower than values in Table 4.
2. The study authors reported that the aerodynamic flux method used in this study is a modified form of the Thornthwaite-Holzman equation (Majewski, M.S., M.M. McChesney, J.N. Seiber. A Field Comparison of Two Methods for Measuring DCPA Soil Evaporation Rates, *Environmental Toxicology and Chemistry*, 1991, 10: 301-311.) which is based upon the log-law of the wind speed profile. Aerodynamic flux is a gradient method requiring accurate vertical gradient measurements of horizontal wind speed, air temperature, and pesticide concentrations. The modified equation compensates for conditions which cause the atmosphere to be stable or unstable.
3. The study authors stated that the flux method was preferable to the aerodynamic method for determining mass flux because it would allow analysis of small areas. This permitted a comparison of mass loss from varying small-scale physical components in the field, in this case the raised beds and furrows.

4. Charcoal adsorption tubes were stored frozen for up to 70 days prior to analysis (Table 1, pp. 135-140). The study authors stated that 1,3-dichloropropene has been shown to be stable when stored frozen on charcoal for up to 364 days (p. 115). The storage stability study was referenced, but was not available to review.
5. The depth to the water table not reported.
6. Immediate posttreatment soil samples were not collected to confirm the application rate. The application rate was determined by weighing the test substance container prior to and after application (pp. 32-33). A density of 11.2 lb/gallon was assumed to convert weight (1194.0 lbs) of the In-Line used to volume (106.7 gallons) used. 1,3-Dichloropropene comprised 60.2% of the In-Line. The treated plot measured 4.33 acres. The study authors reported that collection of immediate posttreatment soil samples to confirm the application rate was impractical because of the method of application and the lateral and vertical movement of 1,3-dichloropropene in the soil (p. 16).
7. The depths at which soil temperatures were measured are not consistent within the text. The data tables indicate that soil temperature was measured in 15-cm increments. Also, although reportedly measured during the study, percent relative humidity values were not reported.
8. Stability of 1,3-dichloropropene on the charcoal adsorption tubes during shipment was investigated (p. 39). Activated charcoal adsorption tubes were fortified with *cis*- and *trans*-1,3-dichloropropene at 10, 100 and 2,000 $\mu\text{g}/\text{tube}$. The fortified tubes were shipped to the field site, where some were exposed to ambient air, and all were returned to the analytical laboratory with a shipment of field samples. Travel spike sample recoveries ranged from 97-143% (mean 110-112%), and field spike sample recoveries ranged from 99-120% (mean 105-108%; p. 141).
9. GC conditions were not described. Two pages of the analytical methods are missing from the document; Appendix A pagination jumps from page 9 to page 12. These pages should appear between Dow AgroSciences pages 115 and 116. The lack of these pages was not critical to the interpretation of the study, since the method is summarized within the report text.

ATTACHMENT 1
Data Critical to the Study Interpretation

**THE FOLLOWING ATTACHMENT IS NOT AVAILABLE ELECTRONICALLY
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Page is not included in this copy.

Pages 9 through 30 are not included in this copy.

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