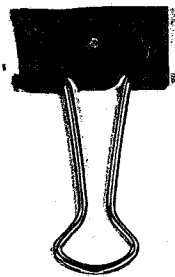


US EPA ARCHIVE DOCUMENT



DATA EVALUATION RECORD

STUDY 9

CHEM 128847 Difenconazole §163-1  
CAS No. 119446-68-3  
FORMULATION-00-ACTIVE INGREDIENT

STUDY ID 42245137

Pollock, R. J. 1989. Leaching characteristics of parent CGA-169374. Project No.: 983.  
Unpublished study performed by Analytical Development Corporation, Colorado Springs, CO;  
and submitted by CIBA-GEIGY Corporation, Greensboro, NC.

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REVIEWED BY: A. M. Glucksman, M.S.  
TITLE: Scientist

Signature: *C.A. Sutton for A.M. Glucksman*  
Date: *4/17/00*

EDITED BY: D. R. Hunt, B.A.  
TITLE: Scientist

Signature: *Don Hunt*  
Date: *4/14/2000*

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

Signature: *C.A. Sutton for P.H. Howard*  
Date: *4/17/00*

ORG: Syracuse Research Corporation  
Arlington, VA 22202

TEL: 703/413-9369

APPROVED BY: James A. Hetrick  
TITLE: Chemist  
ORG: ERB I/EFED/OPP  
TEL: 703/305-5237

SIGNATURE:

## CONCLUSIONS

### Metabolism - Leaching & Adsorption/Desorption

1. This study is not scientifically valid and does not provide useful information on the soil mobility (unaged column leaching) of difenoconazole in four soils. The experimental method was inadequate because the soil columns were leached with distilled water.
2. This study does not meet Subdivision N Guidelines for the fulfillment of EPA data requirements on soil mobility (column leaching) for the following reason:
  - (i) soil columns were leached with distilled water rather than the required 0.01-0.02 *N* CaCl<sub>2</sub> solution.
3. The soil mobility (column leaching) of radiolabeled [ $\Delta$ -<sup>14</sup>C]difenoconazole, at a nominal application rate of 10 ppm (equivalent to 0.2 lb/A), was studied in loam (New York), sandy loam, loamy sand, and loam (Texas) soil columns which were leached with distilled water over 45 days. However, the results were questionable because soils were leached with distilled water rather than 0.01-0.02 *N* CaCl<sub>2</sub> solution.

In the loam soil (from New York), based on LSC analysis, most of the [<sup>14</sup>C]residues retained in the soil column following leaching remained in the 0- to 1-inch depth (86%). Residues were also detected in each one-inch soil section from 1- to 12-inches (total residues <3.4%; reviewer-calculated). Only [<sup>14</sup>C]residues in the 0- to 1-inch depth were characterized. Based on the characterization data reported for the acetone extracts (containing 80.7% of the applied radioactivity), the parent was present at 75.2% of the applied radioactivity in the 0- to 1-inch depth. The minor degradates CGA-142856, CGA-71019, CGA-205375, and CGA-205374 were each present at  $\leq 2.3\%$  of the applied radioactivity in the 0- to 1-inch depth. Nonextractable [<sup>14</sup>C]residues accounted for 15.8% of the applied radioactivity. Following extraction with hexane, an additional 9.0% of the applied radioactivity was extracted from the soil; 87.8% of the recovered radioactivity was parent. Total [<sup>14</sup>C]residues in the leachate were 0.89% of the applied radioactivity (reviewer-calculated mean).

In the sandy loam soil, based on LSC analysis, most of the [<sup>14</sup>C]residues retained in the soil column following leaching remained in the 0- to 1-inch depth (94.1%). Residues were also detected in each one-inch soil section from 1- to 12-inches (total residues <3.9%; reviewer-calculated). Only [<sup>14</sup>C]residues in the 0- to 1-inch depth were characterized. Based on the characterization data reported for the acetone extracts (containing 73.3% of the applied radioactivity), the parent was present at 68.6% of the applied radioactivity in the 0- to 1-inch depth. The minor degradates CGA-142856, CGA-71019, CGA-205375, and CGA-205374 each accounted for  $\leq 1.5\%$  of the applied radioactivity in the 0- to 1-inch depth. Nonextractable [<sup>14</sup>C]residues accounted for 24.9%

of the applied radioactivity. Following extraction with hexane, an additional 14.0% of the applied radioactivity was extracted from the soil; 93.8% of the recovered radioactivity was parent. [<sup>14</sup>C]Residues were not detected in the leachate.

In the loamy sand soil, based on LSC analysis, most of the [<sup>14</sup>C]residues retained in the soil column following leaching remained in the 0- to 1-inch depth (86.7%). Residues were also detected in each one-inch soil section from 1- to 12-inches (total residues <5.9%; reviewer-calculated). Only [<sup>14</sup>C]residues in the 0- to 1-inch depth were characterized. Based on characterization data reported for the acetone extracts (containing 103% of the applied radioactivity), the parent was present at 93.1% of the applied radioactivity in the 0- to 1-inch depth. The minor degradates CGA-142856, CGA-71019, CGA-205375, and CGA-205374 each accounted for ≤3.1% of the applied radioactivity in the 0- to 1-inch depth. Nonextractable [<sup>14</sup>C]residues accounted for 6.5% of the applied radioactivity. Total [<sup>14</sup>C]residues in the leachate were 1.6% of the applied radioactivity (reviewer-calculated mean).

In the loam soil (from Texas), based on LSC analysis, most of the [<sup>14</sup>C]residues retained in the soil column following leaching remained in the 0- to 1-inch depth (98.9%). Residues were also detected in each one-inch soil section from 1- to 12-inches (total residues <2.9%; reviewer-calculated). Only [<sup>14</sup>C]residues in the 0- to 1-inch depth were characterized. Based on the characterization data reported for the acetone extracts (containing 87.0% of the applied radioactivity), the parent was present at 78.9% of the applied radioactivity in the 0- to 1-inch depth. The minor degradates CGA-142856, CGA-71019, CGA-205375, and CGA-205374 each accounted for ≤2.0% of the applied radioactivity in the 0- to 1-inch depth. Nonextractable [<sup>14</sup>C]residues accounted for 9.5% of the applied radioactivity. Total [<sup>14</sup>C]residues in the leachate were 0.42% of the applied radioactivity (reviewer-calculated mean).

## METHODOLOGY

To determine pesticide mobility, PVC columns (3-in i.d.) were packed to a depth of 12 inches with untreated, sieved (2 mm) loam (from New York), sandy loam (from Minnesota), loamy sand (from North Carolina), or loam (from Texas) soil (p. 8; Table I, p. 15); the Texas loam soil was air dried prior to sieving. Three columns were utilized for each soil. To support the bottom of the column, each column was fitted with a window screen (5-in i.d.) that was covered on the inside with glass fiber filter paper. To equilibrate the soil columns, the soils were saturated with potable water (from the bottom to the top of the columns; p. 10). Soil subsamples (35 g) were treated with radiolabeled [ $\Delta$ -<sup>14</sup>C]difenoconazole {CGA-169374; 1-(2-[4-chlorophenoxy)-2-chlorophenyl-(4-methyl-1,3-dioxalan-2-yl)-methyl]-1H-1,2,4-triazole; radiochemical purity 98%, specific activity 19.2  $\mu$ Ci/mg; p. 8}, dissolved in acetone, at a nominal application rate of 10 ppm (equivalent to 0.2 lb/A; pp. 8, 9). The top of each column was treated with a subsample

(10 g; dry weight) of the treated soil; the treated layer was covered with a subsample (10 g) of untreated soil. The soil surfaces were covered with glass fiber filter papers. The columns were leached three times per week with 58 mL (0.5 acre-inches) and twice per week with 116 mL (1 acre-inch) of distilled water over a period of 45 days (see Comment #1); the leachate was collected at each leaching interval. Following leaching, the columns were divided into twelve one-inch sections, placed in plastic bags and mixed by kneading; samples were extracted and analyzed immediately (pp. 10, 11).

Following leaching, duplicate aliquots of leachate collected from each soil column were analyzed for total radioactivity by LSC (p. 10). [<sup>14</sup>C]Residues in the leachate were not characterized.

Duplicate soil subsamples from each soil section were analyzed for total radioactivity by LSC following combustion (p. 11); data were corrected for combustion efficiency (approximately 95%; Appendix, p. 38). The 0- to 1-inch soil section from the three columns were combined for the New York loam, sandy loam, and loamy sand soils (Appendix, p. 35); the 0- to 1-inch soil section from two of three columns were combined for the Texas loam soil. Subsamples of the combined 0- to 1-inch soil sections were oven dried and triplicate subsamples were analyzed by LSC following combustion. Duplicate soil samples were extracted three times by stirring and shaking with acetone followed by a single extraction with methanol (Appendix, p. 36). The samples were centrifuged and the supernatants were filtered (Whatman #1 paper), combined, and concentrated by rotary evaporation and/or by evaporation under nitrogen. The extract was transferred with acetone to a flask, diluted with acetone (extract:acetone, 1:2, v:v), and analyzed by LSC (Appendix, p. 38). Subsamples of the post-extracted soil were analyzed by LSC following combustion. Samples containing greater than 10% of the applied radioactivity in the post-extracted soil were refluxed with methanol:water (9:1, v:v; Appendix, p. 36). The extract was vacuum-filtered (Whatman #1 paper) and concentrated by rotary evaporation. The extract was extracted five times with hexane; the organic layers were combined and concentrated by rotary evaporation. The concentrated organic extract was transferred with hexane into a flask and duplicate aliquots were analyzed by LSC (Appendix, p. 37). The post-extracted/refluxed soil was analyzed by LSC following combustion. Samples containing greater than 10% of the radioactivity in the post-extracted/refluxed soil were refluxed with water:0.1 M dimethylformamide in oxalic acid (35:350, v:v). The extract was vacuum-filtered (Whatman #1 paper), collected in a flask, transferred to a separatory funnel, and partitioned three times with ethyl acetate; duplicate aliquots of the extract were analyzed by LSC. The remaining soil was analyzed by LSC following combustion.

To identify the parent compound and degradates, soil extracts were analyzed by one-dimensional TLC using silica gel plates developed with chloroform:acetic acid (9:1, v:v; Appendix, p. 37). Samples were co-chromatographed with nonradiolabeled reference standards of the parent and degradates CGA-71019, CGA-142856, CGA-205374, and

CGA-205375, which were visualized with UV light (wavelength not specified). Areas of radioactivity were quantified using x-ray film; radiolabeled areas were scraped from the plates, extracted by sonication with methanol, and analyzed by LSC. To confirm the identity of the parent and degradates, soil extracts were further analyzed by two-dimensional TLC using silica gel plates developed with methylene chloride:methanol (9:1, v:v) followed by ether:hexane (4:1, v:v) OR ether:acetic acid (99:1, v:v) followed by toluene:ethyl acetate (1:1, v:v). Samples were co-chromatographed with nonradiolabeled reference standards of the parent and degradates, which were visualized by UV light (wavelength not specified). The TLC plates were exposed to x-ray film.

The filter paper (top and bottom) and the screen from the column were extracted with acetone and analyzed by LSC following combustion (p. 11); total [ $^{14}\text{C}$ ]residues were 0.29-1.4% of the applied radioactivity (Table VI, pp. 20, 21).

To determine the viability of the soil samples, bacterial and fungal populations were evaluated by total plate counts prior to treatment; data indicated that the soils were viable prior to treatment and (Table III, p. 17).

## DATA SUMMARY

The soil mobility (column leaching) of radiolabeled [ $\Delta$ - $^{14}\text{C}$ ]difenoconazole (radiochemical purity 98%), at a nominal application rate of 10 ppm (equivalent to 0.2 lb/A), was studied in loam (New York), sandy loam, loamy sand, and loam (Texas) soil columns which were leached with distilled water over 45 days. However, the results were questionable because soils were leached with distilled water rather than 0.01-0.02 *N*  $\text{CaCl}_2$  solution (see Comment #1).

### Loam soil (from New York)

Based on LSC analysis, most of the [ $^{14}\text{C}$ ]residues retained in the soil column following leaching remained in the 0- to 1-inch depth (86%; Tables VII, XI, p. 22, 26). Residues were also detected in each one-inch soil section from 1- to 12-inches (total residues <3.4%; reviewer-calculated). Only [ $^{14}\text{C}$ ]residues in the 0- to 1-inch depth were characterized. Based on the characterization data reported for the acetone extracts (containing 80.7% of the applied radioactivity), the parent compound was present at 75.2% of the applied radioactivity in the 0- to 1-inch depth (Appendix, Table 7, p. 48). The minor degradates CGA-142856, CGA-71019, CGA-205375, and CGA-205374 (chemical names not provided; structures presented in Appendix, Table 1, pp. 41, 42) were each present at  $\leq 2.3\%$  of the applied radioactivity in the 0- to 1-inch depth. Four unidentified minor degradates were each  $\leq 0.58\%$  of the applied radioactivity in the 0- to 1-inch depth. Nonextractable [ $^{14}\text{C}$ ]residues accounted for 15.8% of the applied radioactivity (Appendix, Table 4, p. 45). Following extraction with hexane, an additional

9.0% of the applied radioactivity was extracted from the soil (Appendix, Table 10, p. 51); 87.8% of the recovered radioactivity was parent. Total [ $^{14}\text{C}$ ]residues in the leachate were 0.89% of the applied radioactivity (reviewer-calculated mean; Table XI, p. 20).

Material balances (based on LSC analysis) following leaching were 95.3-98.4% of the applied radioactivity with the exception of 77.9% of the applied (one of three replicates; Table XV, p. 30).

#### Sandy loam soil

Based on LSC analysis, most of the [ $^{14}\text{C}$ ]residues retained in the soil column following leaching remained in the 0- to 1-inch depth (94.1%; Tables VIII, XII, p. 23, 27). Residues were also detected in each one-inch soil section from 1- to 12-inches (total residues <3.9%; reviewer-calculated). Only [ $^{14}\text{C}$ ]residues in the 0- to 1-inch depth were characterized. Based on the characterization data reported for the acetone extracts (containing 73.3% of the applied radioactivity), the parent compound was present at 68.6% of the applied radioactivity in the 0- to 1-inch depth (Appendix, Table 6, p. 47). The minor degradates CGA-142856, CGA-71019, CGA-205375, and CGA-205374 (chemical names not provided; structures presented in Appendix, Table 1, pp. 41, 42) each accounted for  $\leq 1.5\%$  of the applied radioactivity in the 0- to 1-inch depth. Four unidentified minor degradates each accounted for  $\leq 0.57\%$  of the applied radioactivity in the 0- to 1-inch depth. Nonextractable [ $^{14}\text{C}$ ]residues accounted for 24.9% of the applied radioactivity (Appendix, Table 4, p. 45). Following extraction with hexane, an additional 14.0% of the applied radioactivity was extracted from the soil (Appendix, Table 9, p. 50); 93.8% of the recovered radioactivity was parent. [ $^{14}\text{C}$ ]Residues were not detected in the leachate (Table VI, p. 20).

Material balances (based on LSC analysis) following leaching were 107.1-107.4% of the applied radioactivity with the exception of 79.5% of the applied (one of three replicates; Table XVI, p. 31).

#### Loamy sand soil

Based on LSC analysis, most of the [ $^{14}\text{C}$ ]residues retained in the soil column following leaching remained in the 0- to 1-inch depth (86.7%; Tables IX, XIII, p. 24, 28). Residues were also detected in each one-inch soil section from 1- to 12-inches (total residues <5.9%; reviewer-calculated). Only [ $^{14}\text{C}$ ]residues in the 0- to 1-inch depth were characterized. Based on characterization data reported for the acetone extracts (containing 103% of the applied radioactivity), the parent compound was present at 93.1% of the applied radioactivity in the 0- to 1-inch depth (Appendix, Table 5, p. 46). The minor degradates CGA-142856, CGA-71019, CGA-205375, and CGA-205374 (chemical names not provided; structures presented in Appendix, Table 1, pp. 41, 42) each accounted for  $\leq 3.1\%$  of the applied radioactivity in the 0- to 1-inch depth. Five

unidentified minor degradates each accounted for  $\leq 1.1\%$  of the applied radioactivity. Nonextractable [ $^{14}\text{C}$ ]residues accounted for 6.5% of the applied radioactivity (Appendix, Table 4, p. 45). Total [ $^{14}\text{C}$ ]residues in the leachate were 1.6% of the applied radioactivity (reviewer-calculated mean; Table VI, p. 20).

Mean material balances (based on LSC analysis) following leaching were 95.3% of the applied radioactivity (Table XVII, p. 32).

#### Loam soil (from Texas)

Based on LSC analysis, most of the [ $^{14}\text{C}$ ]residues retained in the soil column following leaching remained in the 0- to 1-inch depth (98.9%; Tables X, XIV, p. 25, 29). Residues were also detected in each one-inch soil section from 1- to 12-inches (total residues  $< 2.9\%$ ; reviewer-calculated). Only [ $^{14}\text{C}$ ]residues in the 0- to 1-inch depth were characterized. Based on the characterization data reported for the acetone extracts (containing 87.0% of the applied radioactivity), the parent compound was present at 78.9% of the applied radioactivity in the 0- to 1-inch depth (Appendix, Table 8, p. 49). The minor degradates CGA-142856, CGA-71019, CGA-205375, and CGA-205374 (chemical names not provided; structures presented in Appendix, Table 1, pp. 41, 42) each accounted for  $\leq 2.0\%$  of the applied radioactivity in the 0- to 1-inch depth. Five unidentified minor degradates each accounted for  $\leq 0.87\%$  of the applied radioactivity in the 0- to 1-inch depth. Nonextractable [ $^{14}\text{C}$ ]residues accounted for 9.5% of the applied radioactivity (Appendix, Table 4, p. 45). Total [ $^{14}\text{C}$ ]residues in the leachate were 0.42% of the applied radioactivity (reviewer-calculated mean; Table VI, p. 21).

Material balances (based on LSC analysis) following leaching were 95.7-97.7% of the applied radioactivity with the exception of 113.8% of the applied (one of three replicates; Table XVIII, p. 33).

#### COMMENTS

1. The experimental method was inadequate because the soil columns were leached with distilled water rather than 0.01-0.02 *N* CaCl<sub>2</sub> solution as required by Subdivision N Guidelines. The use of distilled water could cause particles to disperse, thereby decreasing the rate of infiltration and leaching. Also, the use of distilled water may lead to the removal of sorbed ions from soil particles, thereby affecting the degree of adsorption of the test material.
2. The study author did not state whether any of the soils used in the study were the same soil utilized in an aerobic soil metabolism study. However, the reviewer noted that a loam soil from Texas was used in an aerobic soil metabolism study of difenoconazole (MRID 42245132).



3. The method detection limit was reported for LSC, but not for TLC. Both limits of detection and quantitation should be reported to allow the reviewer to evaluate the adequacy of the method for the determination of the test compound and its degradates.
4. Material balances of individual replicates were 76.8%, 79.5%, and 112.9% of the applied radioactivity in the New York loam, sandy loam, and Texas loam soils, respectively (Table XV-XVIII, pp. 30-32). The study author did not provide an explanation as to why material balances were not between 90-110% of the applied radioactivity as required by Subdivision N Guidelines.
5. Two of the four soils utilized were classified as loam soils. Subdivision N Guidelines require that a minimum of four different soil types be used for unaged soil column leaching studies. In addition, soil series names were not provided. Instead, soils were referred to by their geographic location of collection.
6. The reviewer calculated the total [<sup>14</sup>C]residues detected in the soil columns below the 0- to 1-inch depth by summing the percent of the applied radioactivity recovered from each one-inch soil section (segments 2-12; Tables XI-XIV, pp. 26-29).
7. The study author did not report the chemical name for difenoconazole; the reviewer reported the name that was provided in the soil photolysis study (MRID 42245130). Additionally, the chemical names were not provided for the degradates CGA-142856, CGA-71019, CGA-205375, and CGA-205374; structures were presented in the Appendix (Table 1, pp. 41, 42). In future studies submitted to EPA, it is necessary that the chemical names and structures be reported for the parent and its degradates.

RIN 0509-04

EFED Review for MRID # 422451-37

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Pages 9 through 36 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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