US ERA ARCHIVE DOCUMENT

		Shaughnessy No.: 109702
Too		Date Out of EFGWB:
To:		ct Manager tration Division (H-7505C)
From:	Envir	Abramovitch, Chief onmental Chemistry Review Section #3 onmental Fate and Ground Water Branch/EFED (H-7507C)
Through:		Jacoby, Chief onmental Fate and Ground Water Branch/EFED (H-7507C)
Attached,	pleas	e find the EFGWB review of
Reg./File	# :	
Common Nan	ne :	Cypermethrin.
Type Produ	uct :	Insecticide.
Product Na	ame :	Fligene CI, Siperin, Polytrin, Ammo, Arrivo, Basathrin,
		Aimcocyper, Cypermar, Cyperkill, Cynoff, Cyperguard, Kafil
		Super, Demon, Cyperator, Ralothrin, Sunmerin, Barricade,
		Flectron, Folcord, Ripcord, Ustaad, and Cyrux.
Company Na	ame :	FMC Corporation.
Purpose	:	Review of photodegradation in water and on soil, and mobility
•		(batch equilibrium and aged column leaching) studies.
Date Recei	ived:	EFGWB # (s):
Action Cod	ie :	
Deferrals to:		Ecological Effects Branch, EFED
		Science Integration and Policy Staff, EFED
		Non-Dietary Exposure Branch, HED
		Dietary Exposure Branch, HED
•		Toxicology Branch I, HED
		Toxicology Branch II. HED

1. CHEMICAL: Common name:

Cypermethrin.

Chemical name (C.A.):

(RS)-Cyano(3-phenoxyphenyl)-methyl(1RS)-3-(2,2-dichloro-ethenyl)-2,2-dimethylcyclopropanecarboxylate.

Trade name(s):

Fligene CI, Siperin, Polytrin, Ammo, Arrivo, Basathrin, Amcocyper, Cypermar, Cyperkill, Cynoff, Cyperguard, Kafil Super, Demon, Cyperator, Ralothrin, unmerin, Barricade, Flectron, Folcord, Ripcord, Ustaad, and Cyrux.

Structure:

Formulations:

Emulsifiable concentrate, ULV concentrate, and wettable powder.

Physical/Chemical properties:

Molecular formula: C.H.,Cl,NO,.

Molecular weight: 416.3.

Physical state (mixed isomers): Viscous yellowish-brown semi-solid.

Melting point: 60-80 C.

Solubility (20 C): 0.01-0.2 mg/L water; >450 g/L acetone, chloroform, cyclohexanone, xylene; 337

g/L ethanol; 103 g/L hexane.

2. TEST MATERIAL:

Studies 1-4: Active ingredient.

3. STUDY/ACTION TYPE:

Review of photodegradation in water and on soil, and mobility (batch equilibrium and aged column leaching) studies.

4. STUDY IDENTIFICATION:

Curry, S.J. 1991. Leaching of "C-cypermethrin in soil following aerobic aging. Laboratory Project ID: 191E3190E1. Unpublished study performed and submitted by FMC Corporation, Princeton, NJ. (42129002)

Estigoy, L., L.O. Ruzo, and K. Shepler. 1991a. Photodegradation of ["C-acid] and ["C-alcohol]cypermethrin in buffered aqueous solution at pH 7 by natural sunlight. PTRL Project No. 247/248W. PTRL Report No. 247/248W-1. FMC Study No. 191E1290E1. FMC Report No. PC-0163. Unpublished study performed by Pharmacology and Toxicology Research Laboratory, Richmond, CA, and submitted by FMC Corporation, Princeton, NJ. (42141501)

Estigoy, L., L.O. Ruzo, and K. Shepler. 1991b. Photodegradation of ["C]-acid] and ["C-alcohol]cypermethrin in/on soil by natural sunlight. PTRL Project No. 249/250W. FMC Study No. 191E1390E1. FMC Report No. PC-0159. Unpublished study performed by Pharmacology and Toxicology Research Laboratory, Richmond, CA and submitted by FMC Corporation, Princeton, NJ. (42129001)

Froelich, L.W. 1991. Soil mobility studies: Adsorption/desorption studies of cypermethrin. Laboratory Project ID: 191E3290E1... Unpublished study performed and submitted by FMC Corporation, Princeton, NJ. (42129003)

5. REVIEWED BY:

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Date:

7. CONCLUSION:

6.

- 8. RECOMMENDATIONS:
- 9. BACKGROUND:
 - A. <u>Introduction</u>

B. <u>Directions for Use</u>

Cypermethrin is a stomach and contact insecticide registered for use to control a wide range of pests, particularly lepidoptera, in cotton, fruit, and vegetables. Cypermethrin is a member of the pyrethroid family of chemicals. Single active ingredient formulations include emulsifiable concentrate, ULV concentrate (in vegetable oil carrier for cotton), and wettable powder. For cotton application, cypermethrin can be tank mixed with other cotton-registered products. Some or all applications of cypermethrin may be classified as RUP.

10. <u>DISCUSSION OF INDIVIDUAL TESTS OR STUDIES:</u>

Refer to attached reviews.

11. COMPLETION OF ONE-LINER:

12. CBI APPENDIX:

All data reviewed here are considered "company confidential" by the registrant and must be treated as such.



Environmental Services

CYPERMETHRIN

TASK 1: REVIEW AND EVALUATION OF INDIVIDUAL STUDIES

June 8, 1992

Initial Draft Report

Contract No. 68D20057

Submitted to: Environmental Protection Agency Arlington, VA 22202

Submitted by: Dynamac Corporation The Dynamac Building 2275 Research Boulevard Rockville, MD 20850-3262

CYPERMETHRIN

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INTRODUCTION

Cypermethrin is a stomach and contact insecticide registered for use to control a wide range of pests, particularly lepidoptera, in cotton, fruit, and vegetables. Cypermethrin is a member of the pyrethroid family of chemicals. Single active ingredient formulations include emulsifiable concentrate, ULV concentrate (in vegetable oil carrier for cotton), and wettable powder. For cotton application, cypermethrin can be tank mixed with other cotton-registered products. Some or all applications of cypermethrin may be classified as RUP.

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DATA EVALUATION RECORD

STUDY 1

CHEM 109702

Cypermethrin

§161-2

FORMULATION--OO--ACTIVE INGREDIENT

STUDY ID 42141501

Estigoy, L., L.O. Ruzo, and K. Shepler. 1991a. Photodegradation of ["C-acid] and ["C-alcohol]cypermethrin in buffered aqueous solution at pH 7 by natural sunlight. PTRL Project No. 247/248W. PTRL Report No. 247/248W-1. FMC Study No. 191E1290E1. FMC Report No. PC-0163. Unpublished study performed by Pharmacology and Toxicology Research Laboratory, Richmond, CA, and submitted by FMC Corporation, Princeton, NJ.

DIRECT REVIEW TIME = 16

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CONCLUSIONS:

Degradation - Photodegradation in Water

- This study cannot be used to fulfill data requirements. 1.
- These data are considered to be of uncertain value and should not be 2. used to predict the environmental behavior of cypermethrin and its degradates.
- This study is unacceptable for the following reason: 3.

Photodegradation of cypermethrin and its degradates may have been confounded with microbial degradation. The untreated buffer solution incubated with the phenyl ring-labeled sample set and the untreated buffer solutions incubated with both dark controls contained gram negative bacilli at the termination of the experiments. The study authors stated that the untreated buffer solution incubated with the cyclopropyl ring-labeled sample set showed "no growth of gram negative bacilli" after 30 days; it could not be determined from the limited methodology if tests were conducted to determine the presence of other forms of microorganisms.

In addition, this study does not meet Subdivision N guidelines for the following reasons:

the concentration of cosolvent was 20% by volume, and

one degradate (Unknown A), comprising >10% of the applied radioactivity in the cyclopropyl ring-labeled solution, was not identified.

4. Since the solutions may not have been sterile for the duration of the experiments, and since photodegradation could not be distinguished from microbial degradation, the problems with this study cannot be resolved with the submission of additional data. A new study must be submitted.

METHODOLOGY:

Cyclopropyl ring-labeled ["Clcypermethrin (labeled at the 1-carbon position; radiochemical purity 95.4%, specific activity 300,200 dpm/ug, FMC Corporation) was dissolved in acetonitrile. Aliquots (2 mL) of the stock solution were mixed with filter-sterilized (0.22 um) 0.0048 M pH 7 phosphate buffer solution (8 mL) in quartz and foil-covered Pyrex tubes. The resulting solutions were vortexed; the final concentration of ["C]cypermethrin and acetonitrile in the solutions was 0.1 ppm and 20% by volume, respectively. The tubes were placed at a 60 vertical angle in a circulating water bath located outdoors in Richmond, California (37.45·N, 122.26·W) between March and April, 1991; the samples were maintained at 22.6-28.8 C (mean 25.1 \pm 0.6 C). The daily sunlight intensity ranged from 57 to 24592 uW/cm2; the daily light energy ranged from 3.44 to 10.56 Wminute/cm'; and the cumulative light energy for the 30-day period was 281.59 W-minute/cm² (Table VA). A thermocouple and a photodetector probe were located inside the water bath to monitor environmental conditions. Filter-sterilized air was continuously drawn through the sample tubes, then through ethylene glycol (one tube) and 10% NaOH (two tubes) trapping solutions (Figure 4). Tubes of untreated buffer solution were incubated with the samples to monitor for bacterial contamination at the start and completion of the experiment. Duplicate tubes of the irradiated and dark control solutions were

collected for analysis at 0, 8, 14, 22, and 30 days posttreatment. The trapping solutions were collected for analysis at 14 and 30 days posttreatment.

Phenyl ring-labeled ["Clcypermethrin (uniformly labeled; radiochemical purity 96.9%, specific activity 188,800 dpm/ug, FMC Corporation) was dissolved in acetonitrile and mixed with phosphate buffer solution as previously described. The samples were irradiated with sunlight in Richmond, California, between January and March, 1991. During the experiment, the temperature of the solutions was maintained at 21.6-27.1 C (mean 25.3 ± 0.5 C). The daily sunlight intensity ranged from 59 to 86479 uW/cm²; the daily light energy ranged from 1.14 to 9.07 W-minute/cm² and the cumulative light energy for the 35-day period was 250.84 W-minute/cm² (Table VB). Duplicate tubes of the irradiated and dark control solutions were collected for analysis at 0, 7, 14, 21, 28, and 35 days posttreatment. The trapping solutions were collected for analysis at 14 and 35 days posttreatment.

All samples were "generally" analyzed within 72 hours after collection, and were stored frozen (<0 C) when not in use. The samples were combined with an acetonitrile rinse of their respective sample tubes, and aliquots of the combined solutions were analyzed for total radioactivity using LSC. Additional aliquots of the solutions were analyzed for cypermethrin and its degradates by HPLC using a Supelco C-18 column eluted with a water to acetonitrile linear gradient, and with radioactivity and UV (254 nm) detection. Column eluate fractions (0.5 mL) were collected and analyzed by LSC. HPLC recovery efficiencies averaged 95.4 \pm 6.3% for the cyclopropy1 label and $97.5 \pm 9.0\%$ for the phenyl label; the quantification and detection limits were 50 and 10 dpm, respectively. Additional aliquots of the final samples (30 or 35 days) were analyzed by twodimensional TLC on silica gel plates developed in cyclohexane (saturated with formic acid):ether (3:1, v:v) in the first direction and toluene (saturated with formic acid):ether (10:1, v:v) in the second direction. Samples were cochromatographed with reference standards of cypermethrin and the degradates (1RS)-cis, trans-3-(2,2dichloroviny1)-2,2-dimethylcyclopropanecarboxylic acid (DV acid); (RS)-carbamoy1(3-phenoxypheny1)-methy1(1RS)-cis, trans-3-(2,2dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate (cyperamide); and 3-phenoxybenzoic acid (MPB acid). ["C]Compounds were identified by comparison to the R, values of the reference standards. Radioactive areas were located using a linear analyzer or autoradiography, and identified by comparison to the standards.

Aliquots of the trapping solutions were analyzed for total radioactivity using LSC. ["C]Residues in the NaOH trapping solutions were confirmed to be CO, by barium chloride precipitation.

Aliquots of the untreated buffer solutions were analyzed using MacKonkey and blood plates; no additional microbial analytical methodology was provided.

In an attempt to generate additional degradate material for identification, an acetonitrile solution of cyclopropyl ring-labeled ["C]cypermethrin was mixed with pH 7 buffer solution (0.4 and 1.6 L, respectively) and irradiated with sunlight for 30 days. There was a constant flow of air through the solution. At 30 days, an aliquot of the irradiated solution was analyzed using HPLC as described; the remaining solution was extracted with ethyl acetate. The extracted aqueous solution was lyophilized; and the resulting residues were redissolved in water. Aliquots of the aqueous solution were analyzed by HPLC using a Dionex PCX-500 column eluted with an acidified water:acidified acetonitrile gradient, and with radioactivity and UV (254 nm) detection. Additional aliquots were analyzed by two-dimensional TLC as described.

DATA SUMMARY:

Cyclopropyl and phenyl ring-labeled ["C]cypermethrin (radiochemical purities ≥95.4%), at 0.1 ppm, photodegraded with registrant-calculated half-lives of 36.1 and 20.2 days, respectively, in pH 7 aqueous buffered solutions that were irradiated with sunlight in California for 30 or 35 days. ["C]Cypermethrin degraded slightly (<10% of the applied) in nonsterile dark control solutions during 30-35 days of incubation. The irradiated phenyl ring-labeled solution and both dark control solutions were not sterile during the experiment, and the cyclopropyl ring-labeled solution was described only as being free of gram negative bacilli. In the irradiated solutions, the major degradates identified were 3-(2,2-dimethylcyclopropanecarboxylic acid (DV acid) and 3-phenoxybenzoic acid (MPB acid).

Cyclopropyl ring-labeled ["Clcypermethrin (labeled at the 1-carbon position; radiochemical purity 95.4%), at 0.1 ppm, photodegraded with a registrant-calculated half-life of 36.1 days in "gram negative bacilli"-free, pH 7 aqueous buffered solutions that were irradiated outdoors at 22.6-28.8 C for 30 days in California. The cumulative sunlight energy for the 30-day period was 281.59 W-minute/cm². In duplicate irradiated samples at 30 days posttreatment, cypermethrin was 47.7 and 55.3% of the applied,

3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylic acid (DV acid)

was 3.8 and 10.1%, and Unknown A was 18.5 and 23.9% (Table VIA). Also at 30 days, up to 24 unidentified minor ["C]compounds, each present at \leq 4% of the applied, totaled 18.4 and 27.6% of the applied, and "CO, totaled 5.8%.

In the nonsterile dark control solutions at 30 days posttreatment, cypermethrin comprised 81.3-87.4% of the applied, Unknown A was 5.4-5.9%, unidentified ["C]compounds totaled 10.3-13.9%, and "CO, was 1.5%.

During the experiment, the material balance for the irradiated samples ranged from 96.2 to 111.5% of the applied, and for the dark controls ranged from 87.9 to 104.3% (Table IIA).

Phenyl ring-labeled ["Clcypermethrin" (uniformly labeled; radiochemical purity 96.5%), at 0.1 ppm, photodegraded with a registrant-calculated half-life of 20.2 days in nonsterile pH 7 aqueous buffered solutions that were irradiated outdoors at 21.6-27.1 C for 35 days in California. The cumulative light energy for the 35-day period was 250.84 W-minute/cm. In duplicate irradiated samples, ["C]cypermethrin was 86.9 and 91.3% of the applied immediately posttreatment, 57.9 and 69.9% at 14 days, 39.3 and 51.9% at 21 days, and 22.4 and 28.2% at 35 days (Table VIB). The major degradate,

3-phenoxybenzoic acid (MPB acid)

increased to a maximum of 33.3-35.9% of the applied at 35 days posttreatment; minor degradates included up to 29 unidentified ["C]compounds, each at $\leq 8.9\%$ and totaling up to 35.0% of the applied, and one unidentified ["C]compound that was detected only once, at 11.2% in one of duplicate samples collected at 7 days. "CO totaled 1.4% of the applied during the 35-day experiment.

In the nonsterile dark control solutions at 35 days posttreatment, cypermethrin was 82% of the applied, MPB acid was 6.3%, 24 unidentified degradates totaled 11.7%, and "CO, was ≤ 0.1 %.

During the experiment, the material balance for the irradiated samples ranged from 83.6 to 114.5% of the applied, and for the dark controls ranged from 84.8 to 111.7% (Table IIB).

COMMENTS:

1. The photodegradation of cypermethrin and its degradates in the buffer solutions may have been confounded with microbial degradation. Untreated buffer solutions were incubated with the treated samples to serve as solution sterility checks; however, the value of the checks were limited because the solutions were tested only on MacKonkey and Blood agars for gram negative enterics (sewage coliforms, streptococcus, staphylococcus, etc.). Also, the relationship between the sterility of the untreated buffers and the samples was tenuous. Because of the pattern of contamination, contamination appeared to have occurred either during the transfer of the stock solution into the sample tubes or during incubation, suggesting that individual tubes rather than an entire sample set were contaminated (in this case, it would have been more accurate to test the sterility of the samples themselves).

The untreated buffer stock solution tested immediately posttreatment did not contain gram negative bacilli. The untreated solutions incubated with the phenyl ring-labeled sample set and the untreated

buffer solutions incubated with both dark controls contained gram negative bacilli at the termination of the experiments. The cyclopropyl ring-labeled sample set was reported to contain no gram negative bacilli. The study authors stated that "Since minimal degradation occurred in the dark controls and there was agreement between the light exposed acid and alcohol labels, it is anticipated that no adverse impact on the study results occurred." It is true that degradation in the dark controls was <10% of the applied (half-lives >300 days). In fact, the rate at which cypermethrin degraded in the contaminated phenyl ring-labeled solution (acid dabel) was approximately twice that of cypermethrin in the cyclopropyl ring-labeled solution (alcohol label), 20 compared to 35 days, respectively.

- 2. The concentration of cosolvent (acetonitrile) used by the study authors was 20% by volume; Subdivision N guidelines require that the concentration of cosolvent not exceed 1% in volume. It was stated that the high concentration of acetonitrile was needed because of the low solubility of cypermethrin in water, which was reported to be 0.3 ppm at 25 C; however, the concentration of cypermethrin used in this experiment was only 0.1 ppm. It is possible that the study authors failed to differentiate between the solubility of cypermethrin and its tendency to readily adsorb to glass.
- 3. In the cyclopropyl ring-labeled solutions, one unidentified degradate, Unknown A, comprised 20% of the applied by 30 days posttreatment. Subdivision N guidelines require that all degradates >10% of the applied radioactivity by identified. The study authors stated that, to date, further identification had not been successful, and that attempts to identify the compound are continuing. In addition, in one of the two phenyl ring-labeled solutions sampled at 7 days posttreatment, one unidentified degradate was detected at 11.2% of the applied. The study authors considered this compound to be "transient", and apparently did not attempt to identify the compound further.
- 4. Because the concentration of cypermethrin in solution was variable (as a result of the tendency of cypermethrin to adsorb to the container), the study authors defined "% of the applied" as the percentage of the average concentration of ["C]residues in solution at a given sampling interval, and adjusted the data accordingly. EPA was informed of this approach in advance, and did not object (Appendix A4, EPA return fax dated March 26, 1991). The actual concentration of ["C]residues in solution was never determined, however, because an acetonitrile rinse of the sample tubes was added to the sample solutions prior to analysis.
- 5. The registrant-calculated half-life for the cyclopropyl label dark control was 332.5 days. The authors stated that a half-life was not calculated for the phenyl label dark control, since only three data points were obtained, and the results were similar to those of the cyclopropyl label dark controls.

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DATA EVALUATION RECORD

STUDY 2

CHEM 109702

Cypermethrin

§161-3

FORMULATION--OO--ACTIVE INGREDIENT

STUDY ID 42129001

Estigoy, L., L.O. Ruzo, and K. Shepler. 1991b. Photodegradation of ["C]-acid] and ["C-alcohol]cypermethrin in/on soil by natural sunlight. PTRL Project No. 249/250W. FMC Study No. 191E1390E1. FMC Report No. PC-0159. Unpublished study performed by Pharmacology and Toxicology Research Laboratory, Richmond, CA and submitted by FMC Corporation, Princeton, NJ.

DIRECT REVIEW TIME = 8

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CONCLUSIONS:

Degradation - Photodegradation on Soil

- 1. This study can be used to fulfill data requirements.
- 2. Cypermethrin degraded with a registrant-calculated half-life of approximately 55 days on sandy loam soil that was irradiated with natural sunlight in California. In the corresponding dark controls, cypermethrin decreased from 86.4-105.0% of the applied immediately posttreatment to 72.9-78.4% at 35 days. In both the irradiated and dark control soils, (RS)-carbamoyl(3-phenoxyphenyl)-methyl(1RS)-cis,trans-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate (cyperamide) was the only degradate comprising >10% of the applied.

During the 35-day experiments, the cumulative light energy was 243-251 W-minute/cm², and soil surface temperatures ranged from 11.5 to 34.6 C.

- 3. This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the photodegradation of cyclopropyl and phenyl ring-labeled ["C]cypermethrin on sandy loam soil.
- 4. No additional information on the photodegradation of cypermethrin is required at this time.

METHODOLOGY:

Sieved (2 mm) sandy loam soil (76% sand, 13% silt, 11% clay, 1.8% organic matter content, pH 6.9, CEC 5.2 meq/100 g) was mixed with distilled water, and the resulting slurries were applied to Petri dishes, then air-dried. The soil was remoistened with deionized water to increase the soil moisture to 75% of field capacity.

Cyclopropyl ring-labeled ["Clcypermethrin (labeled at the 1-carbon position; radiochemical purity 95.4%, specific activity 97,500 dpm/ug, FMC Corporation), dissolved in acetonitrile, was evenly applied at 20 ppm to the soil surfaces. The samples were placed perpendicular to the sun's path within two water-jacketed stainless steel chambers located outdoors in Richmond, California (37.45.N, 122.26·W) between February and March, 1991 (Figure 4). One chamber was covered with a quartz plate, the second with a rubber-covered glass plate that excluded light. The daily sunlight intensity ranged from 56 to 86479 uW/cm'; the daily light energy ranged from 1.74 to 10.03 W-minute/cm², and the cumulative light energy for the 35-day period was 242.74 W-minute/cm² (Table VA). Sunlight intensity and energy were measured with a photodetector probe oriented at a 30. angle with respect to the vertical, and located approximately 4 feet above and 6 feet behind the soil chambers. The soil samples were maintained at 11.5-34.6 C (mean 23.7 \pm 0.4) in the irradiated chamber and 12.0-32.3 C (mean 23.8 \pm 0.3) in the dark chamber using circulating coolant through the water jacket; the temperatures were monitored using thermocouples attached to the soil surface. Humidified air was continuously drawn through the sample tubes, then through ethylene glycol (one tube) and 10% NaOH (two tubes) trapping solutions (Figure 4). Duplicate dishes of irradiated and dark control soil were removed for analysis at 0, 7, 14, 21, 28, and 35 days posttreatment. The trapping solutions were sampled at 21 and 35 days posttreatment.

Phenyl ring-labeled ["Clcypermethrin (uniformly labeled; radiochemical purity 96.9%, specific activity 188,800 dpm/ug, FMC Corporation) was dissolved in acetonitrile and applied to Petri dishes containing sandy loam soil as previously described. The samples were irradiated with sunlight in Richmond, California,

between January and March, 1991. The daily sunlight intensity ranged from 59 to 86479 uW/cm²; the daily light energy ranged from 1.14 to 9.07 W-minute/cm², and the cumulative light energy for the 35-day period was 250.84 W-minute/cm² (Table VB). During the experiment, the temperature of the irradiated soil was maintained at 19.8-34.6 C (mean 24.6 ± 0.2), and of the dark control was maintained at 12.3-29.3 C (mean 24.5 ± 0.1). Duplicate dishes of irradiated and dark control soil were removed for analysis at 0, 7, 14, 21, 28, and 35 days posttreatment. The trapping solutions were sampled at 21 and 35 days posttreatment. All samples were stored frozen at <0 C when not in use.

The soil was transferred into centrifuge tubes, then extracted with acetone by shaking on a wrist action shaker for 16 hours. The slurries were centrifuged and the supernatant removed, and the soil was then sequentially extracted once with acetone and once with acetone:water (90:10, v:v), each time by shaking for 1 hour. The acetone and acetone:water extracts were combined, and aliquots were analyzed for total radioactivity by LSC. Additional aliquots of the extracts were analyzed for cypermethrin and its degradates by HPLC using a Supelco C-18 column eluted with a water to acetonitrile linear gradient, and with radioactivity and UV (254 nm) detection. Column eluate fractions (0.5 mL) were collected and analyzed by LSC. HPLC recovery efficiencies averaged 99.2 \pm 10.6% for the cyclopropyl label and $101.5 \pm 5.0\%$ for the phenyl label; the quantification and detection limits were 50 and 10 dpm, respectively. Additional aliquots of the final samples (30 or 35 days) were analyzed by twodimensional TLC on silica gel plates developed in cyclohexane (saturated with formic acid):ether (3:1, v:v) in the first direction and toluene (saturated with formic acid):ether (10:1, v:v) in the second direction. Samples were cochromatographed with reference standards of cypermethrin and the degradates (1RS)-cis, trans-3-(2,2dichlorovinyl)-2,2-dimethylcyclopropanecarboxylic acid (DV acid); (RS)-carbamoyl(3-phenoxyphenyl)-methyl(1RS)-cis,trans-3-(2,2dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate (cyperamide); 3phenoxybenzoic acid (MPB acid); and 3-phenoxybenzaldehyde cyanohydrin (MPB Ald cyanohydrin). ["C]Compounds were identified by comparison to the R, values of the reference standards. Radioactive areas were located using a linear analyzer or autoradiography, and identified by comparison to the standards.

The extracted soil was air-dried and a subsample was analyzed by LSC following combustion. Soil samples retaining >10% of the applied radioactivity following extraction were refluxed with acetonitrile: water (70:30, v:v) for 1 hour, then filtered. The supernatant was analyzed using LSC and TLC as described, and the refluxed soil was analyzed using LSC following combustion.

Aliquots of the trapping solutions were analyzed for total radioactivity using LSC. ["C]Residues in the NaOH trapping solutions were confirmed to be CO, by barium chloride precipitation.

DATA SUMMARY:

Cyclopropyl and phenyl ring-labeled ["C]cypermethrin (radiochemical purities ≥95.4%), at 20 ppm, degraded with a registrant-calculated half-life of approximately 55 days on sandy loam soil that was irradiated with natural sunlight in California for 35 days. In the corresponding dark controls, ["C]cypermethrin decreased from 86.4-105.0% of the applied immediately posttreatment to 72.9-78.4% at 35 days (Tables VIA and VIB). During the 35-day experiments, the cumulative light energy was 243-251 W-minute/cm², and soil surface temperatures ranged from 11.5 to 34.6 C in the irradiated chamber and 12.0 to 32.3 C in the dark chamber. Material balances for the irradiated soil and the dark controls were 91.5-108.6% of the applied immediately posttreatment and 89.6-104.2% at 35 days (Tables IIA and B).

In the irradiated soil at 35 days posttreatment, cyclopropyl and phenyl ring-labeled ["C]cypermethrin comprised 63.0-69.9% of the applied in the soil, the degradate

(RS)-carbamoyl(3-phenoxyphenyl)-methyl(1RS)-cis,trans-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate (cyperamide)

comprised 8.9-10.5%, and numerous unidentified ["C]compounds, each <4.5%, totaled 1.5-5.3% (Tables VIA and VIB). During the experiments, unextracted ["C]residues increased from 1.1-4.8% of the applied immediately posttreatment to 14.9-34.0% at 28 days, then decreased to 9.0-15.2% at 35 days; when 50-80% of the unextracted ["C]residues were extracted by refluxing, the majority (quantitative data not provided) were identified as ["C]cypermethrin (Tables VIA, VIB, VIIA, and VIIB). After 35 days, "CO, totaled 4.0% of the applied from the soil treated with cyclopropyl ring-labeled ["C]cypermethrin and 0.5% from the soil treated with phenyl ring-labeled ["C]cypermethrin; volatilized organic ["C]residues totaled <0.1%.

In the dark control soil at 35 days posttreatment, ["C]cypermethrin comprised 72.9-78.4% of the applied, cyperamide was 12.6-13.5%, unidentified acetone-extractable compounds totaled ≤2.2%, and unextracted ["C]residues were 7.4-10.2% (Tables VIA and VIB). After 35 days, "CO, totaled 1.3% of the applied from the soil treated with cyclopropyl ring-labeled ["C]cypermethrin and 0.6% from the soil treated with phenyl ring-labeled ["C]cypermethrin; volatilized organic ["C]residues totaled <0.1%.

COMMENTS:

1. The results of the TLC analyses of ["C]residues extracted from the soil by refluxing were not presented as numerical values, but only as copies of the autoradiographs.

- 2. Half-lives were calculated by the study authors based on the concentration of cypermethrin in the acetone and acetone:water sample extracts (data presented in Tables VIA and VIB). Cypermethrin extracted from the soil by refluxing was not considered.
- 3. A proposed degradation pathway of cypermethrin is presented in Figure 11.

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DATA EVALUATION RECORD

STUDY 3

CHEM 109702

Cypermethrin

FORMULATION -- OO -- ACTIVE INGREDIENT

STUDY ID 42129003

Froelich, L.W. 1991. Soil mobility studies: Adsorption/desorption studies of cypermethrin. Laboratory Project ID: 191E3290E1. Unpublished study performed and submitted by FMC Corporation, Princeton, NJ.

DIRECT REVIEW TIME = 4

REVIEWED BY: J. Harlin

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K. Ferguson

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CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

- This study can be used towards the fulfillment of data requirements. 1.
- Cypermethrin was immobile in sand, sandy loam, and silt loam soils, 2. and slightly mobile in clay loam soil.
- This study is acceptable and partially fulfills EPA Data Requirements 3. for Registering Pesticides by providing information on the mobility (batch equilibrium) of unaged cypermethrin in sand, sandy loam, silt loam, and clay loam soils.

4. No additional information is needed on the mobility of unaged cypermethrin in soil at this time. The requirement for data on the mobility of aged cypermethrin has also been fulfilled (Study 4; MRID 42129002).

METHODOLOGY:

Sand, sandy loam, silt loam, and clay loam soils (Table I) were airdried and sieved (2 mm). Based on the results of preliminary experiments, a 1:20 soil:solution ratio and a 24-hour equilibration period were selected for the definitive study. In addition, Teflon tubes were selected for use in the definitive study because cypermethrin adsorbed to Teflon surfaces significantly less than it did to glass, polycarbonate, or polysulfone surfaces.

To determine adsorption, aliquots of a 0.01 N CaCl solution were added to sterile Teflon tubes. Cyclopropyl ring-labeled ["C]cypermethrin (labeled at the 1-carbon position, radiochemical purity 98.1%, specific activity 56.3 mCi/mg, Amersham), dissolved at nominal concentrations of 0.005, 0.04, 0.1, 0.2, or 0.4 ug/mL in 0.01 M CaCl, solutions containing 2% acetonitrile, was added to each tube. The solutions were allowed to equilibrate with the wall surfaces for 30 minutes, then aliquots of the solutions were analyzed by LSC. Soil (1 g) was added to each tube. The slurries were equilibrated for 24 hours at 25 \pm 1 C in a shaking water bath in the dark. Following equilibration, the slurries were centrifuged, and the supernatants were decanted and their volume recorded. Triplicate aliquots of the supernatants were analyzed for total radioactivity by LSC.

To determine desorption, the supernatants from the adsorption samples were replaced with pesticide-free 0.01 N CaCl, solution and the soil:solution slurries were shaken for 24 hours in the dark. After the desorption period, the slurries were centrifuged and the supernatants were decanted. Aliquots of the supernatants were analyzed by LSC. The soils were dried with a nitrogen stream, and triplicate subsamples were analyzed by LSC following combustion. The remaining dry soil was removed from the centrifuge tubes, which were then rinsed with acetonitrile, and duplicate aliquots of the acetonitrile rinses were analyzed by LSC.

DATA SUMMARY:

Based on batch equilibrium experiments, cyclopropyl ring-labeled ["C]cypermethrin (labeled at the 1-carbon position, radiochemical purity 98.1%), at nominal concentrations of 0.005, 0.04, 0.1, 0.2, and 0.4 ug/mL, was immobile in sand, sandy loam, and silt loam soils, and slightly mobile in clay loam soil:solution slurries (1:20) that were equilibrated for 24 hours at 25 ± 1 C. Freundlich K. values were 657 for sand, 1163 for sandy loam, 1897 for the silt loam, and

416 for clay loam soils; respective $K_{\rm e}$ values were 285652, 110752, 72405, and 18326 (Table XII).

Following desorption in pesticide-free CaCl, solution (1:20 soil:solution ratio) for 24 hours, 3.72-18.88% of the radioactivity that had been adsorbed to the soils was desorbed (Tables III-VI). Freundlich K. values were 1263 for the sand soil, 191 for the sandy loam soil, 602 for the silt loam soil, and 262 for the clay loam soil.

The material balances for the definitive study were 63.8-79.1% for the sand soil, 74.5-137.0% for the sandy loam soil, 82.6-114.5% for the silt loam soil, and 95.7-111.7% for the clay loam soil (Tables XIII-XVI).

COMMENTS:

- 1. The study author suggested that the low material balance obtained for the sand soil may have been due to particulate matter (possibly organic matter) floating in the supernatant and decanted with the aqueous phase. The author stated that if these particles had adsorbed ["C]cypermethrin, then the combustion analysis for the sand soil would yield lower amounts of radioactivity, thereby decreasing the recovery.
- 2. Recovery efficiencies and method detection limits were not reported.
- 3. The solubility of cypermethrin was reported to be 0.3 ppm at pH 7 and 25 C.

109702 CYPERMETHRIN RIN 8036-92
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DATA EVALUATION RECORD

STUDY 4

Cypermethrin CHEM 109702

FORMULATION -- OO -- ACTIVE INGREDIENT

STUDY ID 42129002

Curry, S.J. 1991. Leaching of "C-cypermethrin in soil following aerobic aging. Laboratory Project ID: 191E3190E1. Unpublished study performed and submitted by FMC Corporation, Princeton, NJ.

DIRECT REVIEW TIME = 16

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SIGNATURE:

CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

This study can be used towards the fulfillment of data requirements. 1.

Aged (30 days) cypermethrin residues were of low mobility in columns 2. of sandy loam soil that were leached with 20 inches of 0.01 N CaCl.; 73.2-92.8% of the applied radioactivity remained in the treated layer and upper 6 cm of the soil columns. The predominant compounds in the soil were cypermethrin, (1RS)-cis, trans-3(2,2-dichloroethenyl)-2,2dimethylcyclopropanecarboxylic acid (cis/trans-DCVA), and 3phenoxybenzoic acid (mPB acid)/3-phenoxyphenylmethanol (mPB alcohol).

- 3. This study is acceptable and partially fulfills EPA Data Requirements for Registering Pesticides by providing information on the mobility (column leaching) of aged ["C]cypermethrin residues in sandy loam soil.
- 4. No additional information is needed on the mobility of aged cypermethrin in soil at this time. The requirement for data on the mobility of unaged cypermethrin has also been fulfilled (Study 3, MRID 42129003).

METHODOLOGY:

Sieved (2-mm), air-dried sandy loam soil (76% sand, 13% silt, 11% clay, 1.8% organic matter content, pH 6.9, CEC 5.2 meq/100 g) was weighed (30 g) into biometer flasks and treated at 0.55 ppm with either cyclopropyl ring-labeled ["C]cypermethrin (labeled at the 1carbon position; radiochemical purity 98.2%, specific activity 56.3 mCi/mMol, Amersham) or phenyl ring-labeled ["C]cypermethrin (uniformly labeled; radiochemical purity 96.9%, specific activity 35.4 mCi/mMol, Sigma Chemical), dissolved in ethanol. The ethanol was allowed to evaporate, and each sample was mixed using a spatula. The soil moisture was adjusted to 75% of field moisture capacity at 0.33 bar. The sidearm of each flask contained a 0.2 M KOH solution for trapping evolved "CO. The flasks were sealed with rubber stoppers and the treated soil samples were incubated aerobically in the dark at approximately 25 C. At weekly intervals, the flasks were removed and weighed, and the soil moisture was adjusted to maintain 70-80% of field capacity. At 30 days posttreatment, the flasks of soil were removed for analysis; trapping solutions were sampled and refreshed at various intervals during the incubation. Following the 30-day aging period, the soil was air-dried and ground. Subsamples were analyzed by LSC following combustion.

Duplicate 19.5-inch glass columns (25-mm id) were filled with sieved (2-mm) sandy loam soil (Figure 1). The soils were saturated with 0.01 N CaCl, by capillary action and allowed to drain to approximate soil moisture at field capacity conditions; the length of the soil columns after saturation was at least 30 cm. A 10-g (dry weight) subsample of the aged, treated soils was evenly distributed on top of each soil column, and the treated layer was covered with 10 g of untreated soil and filter paper. The columns were wrapped with aluminum foil and leached with the equivalent of 20 acre-inches of a 0.01 N CaCl, solution. Leaching was completed within 28-52 hours. The leachate was collected in 100-mL fractions, aliquots of which were analyzed using LSC. The remaining leachate was pooled prior to further analysis. Following leaching, the soil columns were frozen, then thawed for 10-30 minutes, and the partially frozen soil core was extruded from the column. The top layer containing the aged soil and 10 g of overlaid untreated soil were removed, and the remaining soil was divided into five 6-cm segments that were analyzed by LSC following combustion. Following removal of the soil, the glass

columns were rinsed sequentially with 2% Isoclean and methanol; the rinse from each column was combined into a single sample and analyzed for total radioactivity by LSC.

Subsamples of the soil collected during the aerobic incubation period and from all soil segments that contained >0.01 ppm of ["C]residues were extracted in a blender with acetonitrile:water (70:30, v:v) for 5 minutes (Figure 2). The slurries were centrifuged and the supernatant decanted; the extraction procedure was repeated two more times. The remaining soil was transferred to a round-bottom flask, refluxed for 30 minutes with acetonitrile:water (70:30, v:v), and centrifuged as described. The supernatants from each extraction and the reflux were combined to form a single soil extract; the volume was measured and duplicate aliquots were analyzed using LSC. The soil extract was adjusted to pH 2 with concentrated HC1 and the extract was immediately partitioned three times with equal volumes of methylene chloride. The organic and aqueous fractions were separated and analyzed by LSC. The methylene chloride fraction was dried over anhydrous sodium sulfate, partially concentrated by rotary evaporation, and then further concentrated under a nitrogen stream. The concentrated methylene chloride fraction was redissolved in "suitable solvents" and analyzed by one-dimensional TLC and reversephase HPLC.

For HPLC analyses, the extracts were analyzed on a Beckman Ultrasphere ODS column and eluted with a 20% 0.01M acetic acid:80% acetonitrile isocratic gradient and with radioactive flow monitoring and UV (214 nm) detection. Column fractions were collected and analyzed by LSC. Radioactive peaks were compared to the retention times of unlabeled reference standards of unlabeled cis/trans cypermethrin, (1RS)-cis,trans-3(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid (cis/trans-DCVA), 3-phenoxybenzoic acid (mPB acid), and 3-phenoxyphenylmethanol (mPB alcohol) that had been chromatographed in the same systems. The identities of cypermethrin and its degradates were confirmed using GC/MS. The quantitation limit for cypermethrin and its degradates was 0.001 ppm. HPLC column recoveries ranged from 68.8-to 107.5%.

For TLC analyses, the extracts were dissolved in methylene chloride (phenyl ring-labeled) or hexane:methyl t-butyl ether (50:50; cyclopropyl ring-labeled) and spotted on normal phase silica gel plates, which were then developed in toluene:ethyl acetate:acetic acid (75:25:1, v:v:v). ["C]Compounds were located using autoradiography, quantified using a TLC linear analyzer, and identified by comparison to reference standards. Unlabeled reference standards, which were cochromatographed with the phenyl ring-labeled extracts but on separate TLC plates under similar conditions for the cyclopropyl ring-labeled samples, were located with short-wavelength UV light. The quantitation limit for cypermethrin and its degradates was 0.001 ppm.

The leachate fractions from each column were combined and analyzed by LSC. The combined leachate fractions were adjusted to pH 2 and partitioned with methylene chloride. The methylene chloride fractions were concentrated and analyzed as described for the soil methylene chloride fractions.

The KOH trapping solutions from the aerobic aging portion of the study were analyzed for "CO, using two referenced methods (not provided for review). In one method, aliquots of the trapping solutions were acidified with 2 N HCl, then saturated with nonradioactive CO, (dry ice chips), and the remaining solution was analyzed by LSC. In the second method, the identity of CO, was confirmed by barium carbonate precipitation.

DATA SUMMARY:

Based on column leaching studies, aged (30 days) ["C]cypermethrin residues had low mobility in 30-cm columns of sandy loam soil that were leached with 20 inches of 0.01 N CaCl,; \geq 73.2% of the radioactivity applied to the soil columns (% TRR) remained in the surface 6 cm (Tables VII and VIII). The major degradates extracted from the soil were

(1RS)-cis,trans,-3(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid (cis/trans-DCVA)

in soil columns treated with the cyclopropyl ring-labeled ["C]cypermethrin residues, and

3-phenoxybenzoic acid (mPB acid) plus

3-phenoxyphenylmethanol (mPB alcohol)

in columns treated with the phenyl ring-labeled ["C]cypermethrin residues.

Cyclopropyl ring-labeled ["Clcypermethrin: Of the total radioactivity applied to duplicate soil columns, 43.73-64.36% remained in the treated soil layer, 11.36-26.84% was recovered from the 0- to 6-cm segment, 2.31-3.94% was distributed throughout the 12-to 30-cm segments, and 13.42-15.65% was recovered from the leachates (Tables VIII and IX). The recovery from the soil columns was 88.24-93.81% of the material applied to the column (Table X).

Based on reverse-phase HPLC analyses of methylene chloride extracts of the soil segments and leachates, cypermethrin comprised 24.2-36.5% of the applied in the treated layer, 5.0-15.4% in the 0- to 6-cm segment, and 0.3-0.4% in the leachates (Table XIII). DCVA was 2.8-4.8% of the applied in the treated layer, 1.3-2.2% in the 0- to 6-cm segment, and 10.1-13.2% in the leachates. Unidentified ("diffuse")

["C]residues totaled 5.7-6.0% of the applied throughout the soil columns and in the leachates.

In the aged (30 days) soil prior to leaching, 47.1% of the ["C]residues were cypermethrin, 19.9% were DCVA, 5.6% were unidentified extractable, and 24.0% were unextracted (Tables XI and XIII). Cumulative "CO, totaled 21.84% of the applied (Table IV). The material balance during the 30-day aging period was 102.82% of the applied (Table V).

Phenyl ring-labeled ["Clcypermethrin: Of the total radioactivity applied to duplicate soil columns, 20.83-23.96% remained in the treated soil layer, 68.46-72.32% was recovered from the 0- to 6-cm segment, 0.64-6.94% was distributed throughout the 6- to 30-cm segments, and 0.57-0.83% was recovered from the leachates (Tables VII and IX). The recovery from the soil columns was 94.31-101.53% of the material applied to the column (Table X).

Based on reverse-phase HPLC analyses of methylene chloride extracts of the soil segments and leachates, cypermethrin comprised 8.9-10.3% of applied in the treated layer, 28.6-34.2% in the 0- to 6-cm segment, and 2.2% in the 6- to 12-cm segment (Table XII). The degradates mPB acid and mPB alcohol were not differentiated and together comprised 1.2-1.4% of the applied in the treated layer, 3.6-4.3% in the 0- to 6-cm segment, and 0.5% in the 6- to 12-cm segment (one column). Unidentified ("diffuse") radioactivity comprised 4.2-5.0% of the applied throughout the soil columns.

In the aged (30 days) soil prior to leaching, 45.6% of the ["C]residues were cypermethrin, 6.3% were mPB acid/mPB alcohol, 5.1% were unidentified extractable, and 38.6% were unextracted (Tables XI and XII). Cumulative "CO, totaled 23.10% of the applied (Table III). The material balance during the 30-day aging period was 88.84% of the applied (Table V).

COMMENTS:

- 1. The treated soil was aged for slightly longer than one half-life; following the 30-day aging period, cypermethrin comprised 45.6-47.1% of the applied (Tables XII and XIII). The study author stated that the aerobic aging procedure was based on the results of an aerobic metabolism study in which a half-life of approximately 60 days was determined.
- 2. The study author stated that TLC analyses of extracts from the cyclopropyl ring-labeled samples were subject to severe matrix effects, resulting in sample R, values for soil and leachate extracts that were considerably lower than the standards. Therefore, TLC regions were identified by matching patterns, instead of by direct comparison of the R, values.

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- 3. The material balance for the 1-month aerobic aging of the phenyl ring-labeled samples was 88.8% (Table V). The study author suggested that the missing radioactivity may have been "CO, not adsorbed by an oversaturated KOH trapping solution during the first 5 days of aging.
- 4. The soil samples, leachates, and organic and aqueous fractions were frozen when not in use. The study author stated that no significant degradation of the parent compound occurred in treated, unaged soil that was stored frozen for 10 weeks.
- 5. The application rate of 0.55 ppm (0.1 lb ai/A) was reported to be the highest recommended rate for a single application of the test substance.

109702 CYPERMETHRIN	RIN 8036-92
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REFERENCES

The following studies were reviewed:

Curry, S.J. 1991. Leaching of "C-cypermethrin in soil following aerobic aging. Laboratory Project ID: 191E3190E1. Unpublished study performed and submitted by FMC Corporation, Princeton, NJ. (42129002)

Estigoy, L., L.O. Ruzo, and K. Shepler. 1991a. Photodegradation of ["C-acid] and ["C-alcohol]cypermethrin in buffered aqueous solution at pH 7 by natural sunlight. PTRL Project No. 247/248W. PTRL Report No. 247/248W-1. FMC Study No. 191E1290E1. FMC Report No. PC-0163. Unpublished study performed by Pharmacology and Toxicology Research Laboratory, Richmond, CA, and submitted by FMC Corporation, Princeton, NJ. (42141501)

Estigoy, L., L.O. Ruzo, and K. Shepler. 1991b. Photodegradation of ["C]-acid] and ["C-alcohol]cypermethrin in/on soil by natural sunlight. PTRL Project No. 249/250W. FMC Study No. 191E1390E1. FMC Report No. PC-0159. Unpublished study performed by Pharmacology and Toxicology Research Laboratory, Richmond, CA and submitted by FMC Corporation, Princeton, NJ. (42129001)

Froelich, L.W. 1991. Soil mobility studies: Adsorption/desorption studies of cypermethrin. Laboratory Project ID: 191E3290E1. Unpublished study performed and submitted by FMC Corporation, Princeton, NJ. (42129003)

APPENDIX CYPERMETHRIN AND ITS DEGRADATES

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(RS)-Cyano(3-phenoxyphenyl)-methyl(1RS)-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate (Cypermethrin)

3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylic acid (DV acid; Study 1) (1RS)-3(2,2-dichloroethenyl)-2,2-dimethylcyclo-propanecarboxylic acid (DCVA; Study 4)

(RS)-carbamoyl(3-phenoxyphenyl)-methyl(1RS)-cis,trans-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate (Cyperamide)

3-Phenoxybenzoic acid (mPB Acid)

3-Phenoxyphenylmethanol (mPB Alcohol)