11-18-88

DYNAMAC CORPORATION

1/311

ATRAZINE SRR

VOLUME II. DISSIPATION STUDIES (FIELD)

ACCUMULATION STUDIES

Task 1: Review and Evaluation of Individual Studies

Task 2: Environmental Fate Assessment

November 18, 1988

Final Report

Contract No. 68-02-4250

Submitted to:
Environmental Protection Agency
Arlington, VA 22202

Submitted by:
Dynamac Corporation
The Dynamac Building
11140 Rockville Pike
Rockville, MD 20852

DISSIPATION STUDIES
(FIELD)

DATA EVALUATION RECORD

STUDY 28

CHEM 080803 Atrazine §164-1

FORMULATION--XX--DRY FLOWABLE

FICHE/MASTER ID 40431336

White, S. 1987b. Field dissipation study on Aatrex Nine-O for terrestrial uses on bareground, Ripon, California. Laboratory Study No. 1641-86-71-01-21E-23. Prepared by Landis Associates, Inc., Valdosta, GA; Agri-Growth Research, Inc., Hollandale, MN; and Minnesota Valley Testing Labs., Inc., New Ulm, MN; and submitted by Ciba-Geigy Corporation, Greensboro, NC.

DIRECT REVIEW TIME = 5

REVIEWED BY: J. Harlin TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

TEL: 468-2500

APPROVED BY: S. Termes
TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 557-2243

SIGNATURE: Nov. 18/1988

CONCLUSIONS:

Field Dissipation - Terrestrial

This study is unacceptable because the analytical method was inadequate to accurately assess the concentration of atrazine and its degradates in field soil (recovery of atrazine from fortified samples ranged from 87 to 147%, and recovery of its degradates ranged from 0 to 179%). However, this study appears to have been conducted according to EPA Data Requirements for Registering Pesticides.

SUMMARY OF DATA BY REVIEWER:

Atrazine (90% dry flowable) applied at a nominal concentration of 18 lb ai/A to a field plot of unvegetated sandy loam soil located in Ripon, California, in July 1986, dissipated with a registrant-calculated half-life of 119 days (r^2 0.453). In the 0- to 6-inch depth, atrazine

was 4.75 ppm (4.75 ppm total residues) immediately posttreatment, decreased to 1.05 ppm (1.20 ppm total) at 90 days and 0.67 ppm (0.94 ppm total) at 120 days, increased to 5.31 ppm (6.24 ppm total) at 180 days, then decreased to 0.50 ppm (0.63 ppm total) at 267 days and 0.20 ppm (0.26 ppm total) at 358 days posttreatment. Two major degradates were . . .

2-amino-4-chloro-6-ethylamino-s-triazine (G-28279, at up to 0.45 ppm) and

2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033, at up to 0.47 ppm).

One minor degradate . . .

2-hydroxy-4-ethylamino-6-isopropylamino-s-triazine (hydroxyatrazine; G-34048) . . .

was ≤0.05 ppm in the 0- to 6-inch depth throughout the study. Atrazine residues (atrazine plus the degradate G-28279, G-30033, and G-34048) were detected in the 6- to 12-inch depth at up to 1.25 ppm (day 180), but were not detected (<0.05 ppm) in the 12- to 18-, 18- to 24-, and 24- to 36-inch depths at any sampling interval.

DISCUSSION:

1. The analytical method was inadequate to accurately assess the concentration of atrazine and its degradates in field soil. Recovery from fortified soil samples ranged from 87 to 147% for atrazine (average 117.2%), 77 to 179% for 2-amino-4-chloro-6-ethylamino-s-triazine (G-28279; average 121%), 70 to 159% for 2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033; average 119.1%), and 0 to 115% (G-34048; average 24.9%).

2. The study author calculated a half-life of 119 days with an r^2 value of 0.453.

3. During the first 14 days following treatment, 0.03 inches of rain were received and air temperatures ranged from 51 to 100°F. Between 14 and 28 days posttreatment, no rain was received and air temperatures ranged from 52 to 98°F. Between 28 and 60 days posttreatment, 0.15 inches of rain were received and air temperatures ranged from 46 to 99°F. During the entire test period, total precipitation was 9.02 inches, and air temperatures ranged from 22 to 104°F. Soil temperatures were not reported. Meteorological data were collected ≈14 miles from the actual test site. It is preferred that such information be measured at the test site, since rainfall and temperatures can vary between sites in close proximity.

4. The freezer storage stability study is included in Study 29. See pertinent conclusions.

5. Although the registrant stated that soil was sampled to a depth of 4 feet, data were provided only to a depth of 3 feet.

6. Based on concentrations measured during spraying, the actual application of atrazine was 6.84-11.70 lb ai/A (average 9.35 ± 2.23 lb ai/A) instead of the nominal concentration of 18 lb ai/A.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Atrazine (90% dry flowable, Ciba-Geigy Corporation) was applied at 18 lb ai/A to an unvegetated field plot (50 x 50 feet) of sandy loam soil (55% sand, 32.5% silt, 12.5% clay, 1.3% organic matter, pH 7.3, CEC 12.2 meq/100 g) that was located in Ripon, California, on July 23, 1986. One untreated control plot was located ≈150 feet from the test plot. Soil (three cores per plot; 0- to 6-, 6- to 12-, 12- to 18-, 18- to 24-, 24- to 36-, and 36- to 48-inch depths) from the treated and control plots was sampled prior to treatment, immediately after treatment, and at intervals up to 360 days posttreatment using a combination of excavation and hydraulic probe techniques. The samples from each depth were air-dried and mixed to yield one composite sample for analysis. The samples were stored frozen prior to analysis.

A portion of each soil sample was extracted by refluxing for 2 hours using methanol:water (90:10). The extracts were then concentrated and analyzed for atrazine, G-30033, and G-28279 by GC with nitrogen-phosphorus detection. Additional soil was Soxhlet-extracted overnight in methanol:water (80:20) in order to extract G-34048 from the soil. The extract was evaporated to remove methanol and filtered through a XAD-4 column. Aliquots of the extracts were analyzed by HPLC. The detection limits were 0.05 ppm for atrazine and its degradates.

Atrazine

Page_____ is not included in this copy.

Pages 8 through 28 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

✓ FIFRA registration data.

____ The document is a duplicate of page(s) _____.

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

DATA EVALUATION RECORD

STUDY 29

CHEM 080803 Atrazine §164-1

FORMULATION--XX--DRY FLOWABLE

FICHE/MASTER ID 40431337

White, S. M. 1987a. Field dissipation study on Aatrex Nine-O for terrestrial uses on bareground, Hollandale, MN. Laboratory Study No. 1641-86-71-01-21E-25. Prepared by Landis Associates, Inc., Valdosta, GA; Agri-Growth Research, Inc., Hollandale, MN; and Minnesota Valley Testing Labs., Inc., New Ulm, MN; and submitted by Ciba-Geigy Corporation, Greensboro, NC.

DIRECT REVIEW TIME = 8

REVIEWED BY: J. Harlin TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

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SIGNATURE: Nov. 18/1988

CONCLUSIONS:

Field Dissipation - Terrestrial

This study is unacceptable because the analytical method was inadequate to accurately assess the concentration of atrazine and its degradates in field soil (recovery of atrazine from fortified samples ranged from 65 to 147%, and recovery of its degradates ranged from 32 to 179%). However, this study appears to have been conducted according to EPA Data Requirements for Registering Pesticides.

Ancillary Study - Freezer Storage Stability

This study is unacceptable because details of the methodology were incomplete, so that it could not be determined if atrazine and its degradates were stable in frozen soil.

SUMMARY OF DATA BY REVIEWER:

Field Dissipation - Terrestrial

Atrazine (90% dry flowable) was applied at a nominal concentration of 20 lb ai/A to a field plot of unvegetated loam soil located in Minnesota in July, 1986. In the 0- to 6-inch depth, atrazine was 4.23 ppm (5.06 ppm ppm total residues) immediately after treatment, increased to 10.15 ppm (11.66 ppm total) at 14 days, decreased to 5.34 ppm (6.75 ppm total) at 28 days, and was 2.90 ppm (4.88 ppm total) at 360 days post-treatment. The major degradate was . . .

2-hydroxy-4-ethylamino-6-isopropylamino-s-triazine (hydroxy-atrazine; G-34048) . . .

at up to 1.21 ppm.

2-Amino-4-chloro-6-isopropylamino-s-triazine (G-30033) and

2-amino-4-chloro-6-ethylamino-s-triazine (G-28279) . . .

were <1 ppm in the 0- to 6-inch depth throughout the study. In deeper soil, total atrazine residues (atrazine plus the degradates G-34048, G-30033, and G-28279) were most concentrated at 360 days posttreatment; residues were detected in the 6- to 12-inch depth at up to 1.14 ppm, in the 12- to 18-inch depth at up to 0.24 ppm, in the 18- to 24-inch depth at up to 0.14 ppm, in the 24- to 36-inch depth at up to 0.15 ppm, and in the 36- to 48-inch depth at up to 0.08 ppm.

Ancillary Study - Freezer Storage Stability

Atrazine ranged from 0.985 to 1.188 ppm, G-28279 ranged from 0.930 to 1.400 ppm, and G-30033 ranged from 0.988 to 1.996 ppm in soil fortified at ≈1 ppm and stored frozen for up to 145 days. Concentrations did not appear to be related to length of storage.

DISCUSSION:

Field Dissipation - Terrestrial

1. The analytical method was inadequate to accurately assess the concentration of atrazine and its degradates in field soil. Recovery from fortified soil samples ranged from 65 to 147% for atrazine (average 100.4%), 60 to 165% for 2-amino-4-chloro-6-ethylamino-s-triazine (G-28279; average 107.3%), 60 to 179% for 2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033; average 106.8%), and 32 to 147% for 2-hydroxy-4-ethylamino-6-isopropylamino-s-triazine (G-34048; average 81.9%).

2. Data for soil samples taken immediately after treatment appear to be inaccurate, either because of uneven application to the bare soil in the field or analytical errors. Atrazine was applied to the test plots at a nominal concentration of 20 lb ai/A; measurements during spraying ranged

from 13.93 to 20.50 lb ai/A (average 16.28 ± 2.65 lb ai/A). Assuming that 1 acre, 6-inch depth equals 2,000,000 pounds of soil, the expected concentration of atrazine in the soil immediately after treatment would be ≈10 ppm (7-10 ppm based on spray drift data). However, the measured concentration at time 0 was 4.23 ppm of atrazine (5.06 ppm total residues) which was much lower than expected. Also, subsequent samples are much more concentrated than day 0; at 14 and 28 days, the measured concentration of atrazine was 10.15 and 5.34 ppm, respectively.

3. The study author calculated a half-life of 369 days with an r^2 value of 0.379. Because of the poor correlation, the study author stated that the calculated half-life was inaccurate.

4. The ground was frozen for 30% of the study period. The pattern of decline of atrazine would be typical of cold winter areas; atrazine would be expected to dissipate faster in warmer areas.

5. During the first 14 days following treatment, 2.95 inches of rain were received, air temperatures ranged from 56 to 90°F, and soil temperatures (4-inch depth) ranged from 64 to 91°F. Between 14 and 28 days posttreatment, 0.93 inches of rain were received, air temperatures ranged from 56 to 92°F, and soil temperatures ranged from 67 to 100°F. During the entire test period, total precipitation was ≈28.36 inches, and high and low air temperatures ranged from -1 to 97°F and -17 to 75°F, respectively.

Ancillary Study - Freezer Storage Stability

1. Insufficient details about the methodology were provided to review. The soil was described only as being obtained in New Ulm, Minnesota. It was not stated whether each soil sample represents a separate treatment, or if soil was treated, homogenized, and then subsampled. It was not stated if the soils were frozen moist or dry. The freezer temperature was not reported.

2. No freezer storage stability data were provided for G-34048.

3. Apparently the soil was not analyzed immediately after treatment to confirm the application rate.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Field Dissipatation - Terrestrial

Atrazine (90% dry flowable, Ciba-Geigy Corporation) was applied at 20 lb ai/A to an unvegetated field plot (50 x 50 feet) of loam soil (47.5% sand, 37.5% silt, 25% clay, 6.2% organic matter, pH 7.9, CEC 23.9 meq/100 g) that was located in Hollandale, Minnesota, on July 2, 1986. One untreated control plot was located ≈150 feet from the test plot. Soil (three cores per plot; 0- to 6-, 6- to 12-, 12- to 18-, 18- to 24-, 24- to 36-, and 36- to 48-inch depths) from the treated and control plots was sampled prior to treatment, immediately after treatment, and at intervals up to 360 days posttreatment using a combination of excavation and hydraulic probe techniques. The samples from each depth were air-dried, then mixed to yield one composite sample for analysis. The samples were frozen until analysis.

A portion of each soil sample was extracted by refluxing for 2 hours with methanol:water (90:10). The extracts were concentrated and analyzed for atrazine, G-30033, and G-28279 by GC with nitrogen-phosphorous detection. In order to extract G-34048 from the soil, additional soil was Soxhlet-extracted overnight in methanol:water (80:20). The extract was evaporated to remove methanol and filtered through a XAD-4 column. Aliquots of the extracts were analyzed by HPLC. The detection limits were 0.05 ppm for atrazine and its degradates.

Ancillary Study - Freezer Storage Stability

Soil from New Ulm, Minnesota, was treated with either atrazine, G-28279, or G-30033 at ≈1 ppm. The soil was stored frozen (temperature and moisture content unspecified) for up to 145 days. Samples were analyzed on days 1, 7, and 145 using methods described in the field dissipation portion of this study.

Atrazine

Page____ is not included in this copy.

Pages 34 through 57 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

__✓__ FIFRA registration data.

____ The document is a duplicate of page(s) _____.

____ The document is not responsive to the request.

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

DATA EVALUATION RECORD

STUDY 30

CHEM 080803 Atrazine §164-1

FORMULATION--XX--DRY FLOWABLE

FICHE/MASTER ID 40431338

White, S. M. 1987d. Field dissipation study on Aatrex Nine-O for terrestrial uses on corn, Ripon, CA. Laboratory Study No. 1641-86-71-01-06B-22. Prepared by Landis Associates, Inc., Valdosta, GA; Research for Hire, Porterville, CA; and Minnesota Valley Testing Labs., Inc., New Ulm, MN; and submitted by Ciba-Geigy Corporation, Greensboro, NC.

DIRECT REVIEW TIME = 12

REVIEWED BY: J. Harlin TITLE: Staff Scientist

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APPROVED BY: S. Termes
TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 557-2243

SIGNATURE: Nov. 18/1988

CONCLUSIONS:

Field Dissipation - Terrestrial

This study is unacceptable because the analytical method was inadequate to accurately assess the concentration of atrazine and its degradates in field soil (recovery of atrazine from fortified samples ranged from 65 to 150%, and recovery of its degradates ranged from 0 to 195%). However, this study appears to have been conducted according to EPA Data Requirements for Registering Pesticides.

SUMMARY OF DATA BY REVIEWER:

Atrazine (90% dry flowable), applied at a nominal concentration of 3.96 lb ai/A to a field plot of sandy loam soil located in California in July, 1986, dissipated with a registrant-calculated half-life of 58 days (r^2 0.952). In the 0- to 6-inch depth, atrazine was 1.15 ppm (1.15 ppm

total residues) immediately after treatment, increased to 2.82 ppm (2.82 ppm total) at 7 days, decreased to 1.18 ppm (1.18 ppm total) at 14 days, decreased to 0.50 ppm (0.74 ppm total) at 60 days, and was 0.02 ppm (0.53 ppm total) at 358 days posttreatment. The major degradate was . . .

2-hydroxy-4-ethylamino-6-isopropylamino-s-triazine (hydroxyatrazine; G-34048) at up to 0.49 ppm.

2-Amino-4-chloro-6-isopropylamino-s-triazine (G-30033), and

2-amino-4-chloro-6-ethylamino-s-triazine (G-28279) . . .

were ≤0.08 ppm in the 0- to 6-inch depth throughout the study. Atrazine residues (atrazine plus the degradates G-34048, G-30033, and G-28279) were detected in the 6- to 12-inch depth at up to 0.31 ppm (day 267), but were not detected (<0.05 ppm) in the 12- to 18-, 18- to 24-, and 24- to 36-inch depths at any sampling interval.

DISCUSSION:

1. The analytical method was inadequate to accurately assess the concentration of atrazine and its degradates in field soil. Recovery from fortified soil samples ranged from 65 to 150% for atrazine (average 100.7%), 51 to 195% for 2-amino-4-chloro-6-ethylamino-s-triazine (G-28279; average 96.0%), 56 to 164% for 2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033; average 107.7%), and 0 to 114% for 2-hydroxy-4-ethylamino-6-isopropylamino-s-triazine (G-34048; average 24.8%).

2. During the first 7 days following treatment, 0.03 inches of rain were received and air temperatures ranged from 51 to 99°F. Between 7 and 14 days posttreatment, no rain was received and air temperatures ranged from 54 to 100°F. Between 14 and 60 days posttreatment, 0.15 inches of rain were received and air temperatures ranged from 46 to 99°F. During the entire test period, total precipitation was ≈9.02 inches, and air temperatures ranged from 22 to 104°F.

3. The freezer storage stability study included with this document was reviewed in Study 1 of this addendum.

4. Field records provided in the original document indicate that the plots were irrigated. The dates and amounts of irrigation were not reported.

5. Although the registrant stated that the atrazine was applied as a preemergent to corn, there in no indication in the original document if or when the corn was planted.

6. Although the registrant stated that soil was sampled to a depth of 4 feet, data were provided only to a depth of 3 feet.

7. Based on concentrations measured during spraying, the actual application of atrazine was 1.06-4.53 lb ai/A (2.94 ± 1.40 lb ai/A).

MATERIALS AND METHODS

MATERIALS AND METHODS:

Atrazine (90% dry flowable, Ciba-Geigy Corporation) was applied at 3.96 lb ai/A to a field plot (50 x 50 feet) of sandy loam soil (55.0% sand, 32.5% silt, 12.5% clay, 1.3% organic matter, pH 7.3, CEC 12.2 meq/-100 g) that was located in Ripon, California, on July 23, 1986. One untreated control plot was located ≈150 feet from the test plot. Soil (three cores per plot; 0- to 6-, 6- to 12-, 12- to 18-, 18- to 24-, and 24- to 36-inch depths) from the treated and control plots was sampled prior to treatment, immediately after treatment, and at intervals up to 358 days posttreatment using a combination of excavation and hydraulic probe techniques. The samples from each depth were air-dried, then mixed to yield one composite sample for analysis. The samples were frozen until analysis.

A portion of each soil sample was extracted by refluxing for 2 hours with methanol:water (90:10). The extracts were concentrated and analyzed for atrazine, G-30033, and G-28279 by GC with nitrogen-phosphorous detection. In order to extract G-34048 from the soil, additional soil was Soxhlet-extracted overnight in methanol:water (80:20). The extract was evaporated to remove methanol and filtered through a XAD-4 column. Aliquots of the extracts were analyzed by HPLC. The detection limits were 0.05 ppm for atrazine and its degradates.

Atrazine

Page_____ is not included in this copy.

Pages 62 through 83 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

✓ FIFRA registration data.

____ The document is a duplicate of page(s) _____.

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

DATA EVALUATION RECORD

STUDY 31

CHEM 080803 Atrazine §164-1

FORMULATION--XX--DRY FLOWABLE

FICHE/MASTER ID 40431339

White, S. M. 1987c. Field dissipation study on Aatrex Nine-O for terrestrial uses on corn, Hollandale, MN. Laboratory Study No. 1641-86-71-01-06B-24. Prepared by Landis Associates, Inc., Valdosta, GA; Agri-Growth Research, Inc., Hollandale, MN; and Minnesota Valley Testing Labs., Inc., New Ulm, MN; and submitted by Ciba-Geigy Corporation, Greensboro, NC.

DIRECT REVIEW TIME = 5

REVIEWED BY: J. Harlin TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

TEL: 468-2500

APPROVED BY: S. Termes
TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 557-2243

SIGNATURE: Nov. 18/1988

CONCLUSIONS:

Field Dissipation - Terrestrial

This study is unacceptable because the analytical method was inadequate to accurately assess the concentration of atrazine and its degradates in field soil (recovery of atrazine from fortified samples ranged from 76 to 150%, and recovery of its degradates ranged from 42 to 183%). However, this study appears to have been conducted according to EPA Data Requirements for Registering Pesticides.

SUMMARY OF DATA BY REVIEWER:

Atrazine (90% dry flowable) was applied at a nominal concentration of 4.4 lb ai/A to a field plot of loam soil planted to corn located in Minnesota in June, 1986. In the 0- to 6-inch depth, atrazine was 1.20 ppm (1.37 ppm total residues) immediately after treatment, increased

to 1.40 ppm (1.59 ppm total) at 2 days, ranged from 0.48 to 1.00 ppm (0.90-1.17 ppm total) with no discernable pattern between 7 and 290 days, and was 0.37 ppm (0.91 ppm total) at 360 days posttreatment. The major degradates were . . .

2-hydroxy-4-ethylamino-6-isopropylamino-s-triazine (hydroxy-atrazine; G-34048) at up to 0.20 ppm, and

2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033) at up to 0.30 ppm.

2-Amino-4-chloro-6-ethylamino-s-triazine (G-28279) . . .

was ≤0.1 ppm in the 0- to 6-inch depth throughout the study. Atrazine residues (atrazine plus the degradates G-34048, G-30033, and G-28279) were detected in the 6- to 12-inch depth at up to 0.29 ppm (days 0 and 95), but were not detected (<0.05 ppm) in the 12- to 18-, 18- to 24-, and 24- to 36-inch depths at any sampling interval.

DISCUSSION:

1. The analytical method was inadequate to accurately assess the concentration of atrazine and its degradates in field soil. Recovery from fortified soil samples ranged from 76 to 150% for atrazine (average 111.7%), 76 to 183% for 2-amino-4-chloro-6-ethylamino-s-triazine (G-28279; average 108.5%), 56 to 143% for 2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033; average 107.6%), and 42 to 142% for 2-hydroxy-4-ethylamino-6-isopropylamino-s-triazine (G-34048; average 83.3%).

2. The study author calculated a half-life of 291 days with an r^2 value of 0.56.

3. The ground was frozen for 30% of the study period. The pattern of decline of atrazine would be typical of cold winter areas; atrazine would be expected to dissipate faster in warmer areas.

4. During the first 2 days following treatment, 1.15 inches of rain were received, air temperatures ranged from 68 to 92°F, and soil temperatures (4-inch depth) ranged from 72 to 93°F. Between 2 and 7 days posttreatment, 0.80 inches of rain were received, air temperatures ranged from 52 to 90°F, and soil temperatures ranged from 62 to 85°F. Between 7 and 13 days posttreatment, 0.42 inches of rain were received, air temperatures ranged from 55 to 86°F, and soil temperatures ranged from 64 to 93°F. During the entire test period, total precipitation was ≈28.36 inches, and air temperatures ranged from -17 to 97°F.

5. The freezer storage stability study included with this document was reviewed in Study 1 of this addendum.

6. Although the registrant stated that soil was sampled to a depth of 4 feet, data were provided only to a depth of 3 feet.

7. Based on concentrations measured during spraying, the actual application of atrazine was 2.63-3.36 lb ai/A (2.95 ± 0.27 lb ai/A).

MATERIALS AND METHODS

MATERIALS AND METHODS:

Atrazine (90% dry flowable, Ciba-Geigy Corporation) was applied at 4.4 lb ai/A to a field plot (50 x 50 feet) of loam soil (47.5% sand, 37.5% silt, 25% clay, 6.2% organic matter, pH 7.9, CEC 23.9 meq/100 g) that was located in Hollandale, Minnesota, on June 20, 1986. The plot had been planted to field corn on June 17, 1986. One untreated control plot was located ≈150 feet from the test plot. Soil (three cores per plot; 0- to 6-, 6- to 12-, 12- to 18-, 18- to 24-, and 24- to 36-inch depths) from the treated and control plots was sampled prior to treatment, immediately after treatment, and at intervals up to 360 days posttreatment using a combination of excavation and hydraulic probe techniques. The samples from each depth were air-dried, then mixed to yield one composite sample for analysis. The samples were frozen until analysis.

A portion of each soil sample was extracted by refluxing for 2 hours with methanol:water (90:10). The extracts were concentrated and analyzed for atrazine, G-30033, and G-28279 by GC with nitrogen-phosphorous detection. In order to extract G-34048 from the soil, additional soil was Soxhlet-extracted overnight in methanol:water (80:20). The extract was evaporated to remove methanol and filtered through a XAD-4 column. Aliquots of the extracts were analyzed by HPLC. The detection limits were 0.05 ppm for atrazine and its degradates.

Atrazine

Page____ is not included in this copy.

Pages 89 through 112 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

✓ FIFRA registration data.

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DATA EVALUATION RECORD

STUDY 32

CHEM 080803 ATRAZINE §164-1

FORMULATION--TYPICAL END-USE PRODUCT, EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 40431335
Klaine, S.J. 1987. Field dissipation study of atrazine in West Tennessee soil. Conducted by the Department of Biology, Memphis State University, Memphis, TN. Laboratory Study No. TX-431A. Completed October 22, 1987. Submitted by Ciba-Geigy Corporation, Greensboro, NC.

REVIEWED BY: Silvia C. Termes TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 557-2243

SIGNATURE: Nov. 16, 1988

CONCLUSIONS:

This study, which can only be considered supplemental to field dissipation studies, is unacceptable for the following reasons: (1) information on the percentages of sand-clay-silt was not given; (2) while the 1985 application was sampled through a 238-day period, the 1986 application was only sampled through a 43-day period, with no explanation given for this; (3) not all of the results of the soil-depth samples said to have been analyzed were shown; (4) the 1986 application shows only analyses down to 20-30 cm depth.

Atrazine was chosen in this study because of its abundant use throughout the USA, physical/chemical properties (summarized in Table I; refer to pertinent references at the end of the study), biological parameters, and availability of analytical methods.

MATERIALS AND METHODS

Site: Located at Agricenter International, Shelby County, SW Tennessee. Altitude 300 ft above sea level. Western 10% of the county is on the Mississippi river flood plain. Average annual temperature in Memphis is 16.7 deg. C. Average annual precipitation is approximately 127 cm (50 in), most of it occurring in winter and early spring, with a second period of heavy precipitation (with showers and thunderstorms) in late spring and early summer. The predominant surface waters (other than the Mississippi) are the Wolf and the Loosahatchie Rivers. However, they are not important sources for municipal, industrial, and agricultural use. Groundwater, because of the abundance, high quality, and relatively low pumping and treatment costs, is used primarily as water source. The county is predominantly industrial, but agriculture provides a large amount of income, with soybeans, cotton, corn, and wheat being the most important crops. The Agricenter is located in the south center of the county, with 1000 acres of fertile farmland. A watershed area approximately 18.21 ha (45 acres) was chosen. Other criteria for choosing the site were the proximity with Memphis State University and to the Wolf River.

Soil: Primary soil type is a Falaya silt loam, which is somewhat poorly drained, very silty. Surface layer is a brown, friable silt loam about 6 inches thick; underlying material is a friable silt loam containing brown and gray mottles, which extends to a depth of several feet. Available water capacity is high. Upon rainwater leaching, the soil becomes strongly acidic. A soil analysis is shown in Table II (P and K content is moderately high). The watershed was divided into several segments (Figure 1) to facilitate sampling.

Agronomic practices: Agronomic activities are summarized in Table III. Ten ha (24 acres) of the north watershed were planted in corn; 8 ha (21 acres) were set aside (in accordance with a federal program) during 1985 and 1986. Fertilizers: nitrogen was supplied in the form of urea, phosphorus as diammonium polyphosphate, and potassium as potash (first application of first year); second application consisted of ammonium nitrate.

Atrazine application: Atrazine was applied with a 12-row boom; the boom contained 25 nozzles, 61 cm (24 in) above the soil surface.

Application rates- 1985: 1.27 kg/ha (1.14 lb/acre)
1986: 1.67 kg/ha (1.5 lb/acre).

Atrazine was used as the emulsifiable concentrate and following label requirements. Spray operations were conducted during periods of low temperature and wind velocities.

Soil sampling: North watershed was divided into 4-subareas and each subarea sampled at five depths: 0-20 cm; 20-40 cm; 40-60 cm; 60-80 cm; 80-100 cm. Sampling was done with a split-tube core sampler (approximately 2-cm diameter). Background atrazine was measured at each depth interval. Analyses were performed immediately after sampling. Sampling times after atrazine application were 0, 1, 3, 7, 14, 28, 56, 84, and 238 days (in 1985) and 0, 1, 3, 7, 22, and 43 days (in 1986).

Determination of atrazine application rate: This was determined by two methods. One method consisted of randomly placing glass-fiber filter pads (1.2 um) through the watershed. The pads were collected after application, combined, and sent for analysis. The other method consisted in timing the nozzles and calculating the spray volumes discharged by the 12-row boom used to apply the atrazine (this was done immediately preceding application on May 17, 1985 and on May 7, 1986). Soil sampling at day 0 also serve to confirm the application rate.

Soil sampling: Soil samples were analyzed for pH, organic content, and water content. Analyses were performed in accordance to standard ASTM methods.

Analysis of atrazine in soils: Each core was divided into subsamples representing the different depths: 0-10 cm, 10-20 cm, 20-30 cm, 30-40 cm, 40-50 cm, 60-70 cm, and 90-100 cm. Atrazine analyses were performed in triplicate. For 1985, values are reported through 238 days after application. The extraction procedure used is shown in Figure 2. Atrazine standards were prepared from samples obtained from Ciba-Geigy.

REPORTED RESULTS AND DISCUSSION

Atrazine levels

a) Prior application: 0.0011 ug/g (1985); 0.0077 ug/g (1986), based on soil cores in planted area. The 1986 value was higher because it was taken in Jan. and not before atrazine application in May. Top 20-cm of soil.

b) Day 0: Average of 0.69 ug/g or 9.2 ug/cm^2 at the surface; this represents 94% of the atrazine (9.7 ug/cm^2) as measured on the filter pads; top 10-cm, 1985.

Average of 1.29 ug/g or 16.9 ug/cm^2 at the soil surface; this represents 108% of the applied atrazine (15.6 ug/cm^2) as measured by the filter pad technique; top 10-cm, 1986.

Atrazine concentrations with time

Following atrazine application in 1985, 36% of the applied atrazine remained in the soil after 28 days, 6% after 84 days, and 2% after 238 days. Results are shown in Table IV. Figures 3 and 4 show atrazine levels at different depths. On day 7, following first runoff event after application, levels in the 10-20 cm depth were 0.0091 ug/g. Levels at this depth increased approximately 400% during next 3-weeks and decreased to 0.0080 ug/g on day 84. This migration was attributed to runoff events (5/23; 6/10). Atrazine concentration never exceeded 0.01 ug/g in the 20-30 cm depth.

Table V show the atrazine levels at different depths for 1986 application (also Figures 5 and 6). Between days 7 and 22, atrazine levels in the 10-20 cm depth decreased, which was attributed to the lack of rain. After 7 days of application, approximately 50% of the atrazine remained in the soil 0-10 cm depth, 41% after 22 days, and 23% after 43 days. Between days 22 and 43, levels increased about 300% in the 10-20 cm depth (caused by several rainfalls).

Atrazine half-life

A first-order kinetics was assumed in the calculation of the decay of atrazine. Using data from the field soil for the 1985 application, a half-life of atrazine of approximately 20 days was calculated. For 1986, the data from the field soil indicate that two distinct exponential periods exist: an initial period exhibiting higher reduction constants (ending between 7 and 22 days, with several small rainfall events with little runoff) and a second exponential period exhibiting lower reduction constants (significant rainfall after 28 days of application may be responsible). The "half-life" for the initial period was about 7-days; for the second period it was about 20-days.

Nutrient concentrations and physical conditions of soils

These are summarized in Tables VI-XI.

Crop performance

Excellent development of the corn was observed due to the sufficient and well-distributed rainfall from the second planting date through the end of June 1985. The growth of Johnson grass was heavy during early summer; there was also problem with smartweed and cocklebur; insects were not a problem. Corn yield was only 38 bushels/A in 1985 (attributed to maize dwarf mosaic virus carried by the Johnson grass). Corn development was slower in 1986. Rainfall was not well-distributed and the problems with the Johnson grass and the virus were more severe.

REVIEWER'S COMMENTS

This study can only be considered as supplemental. Although a complete elemental analysis of the soil was provided, no information on the percentages of sand-clay-silt was given. While the 1985 application was sampled through a 238-day period, the 1986 application was only sampled through a 43-day period.

Not all of the results of the soil-depth samples said to have been analyzed were shown. The 1986 application shows only analyses down to the 20-30 cm depth.

PERTINENT DATA TABLES AND FIGURES

Atrazine

Page_____ is not included in this copy.

Pages 119 through 139 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

✓ FIFRA registration data.

____ The document is a duplicate of page(s) _____.

____ The document is not responsive to the request.

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

DATA EVALUATION RECORD

STUDY 33

CHEM 080803 Atrazine §§164-3 and 165-5

FORMULATION--04--GRANULAR (G)

FICHE/MASTER ID 40431340
Schofield, M. 1986. Combined field dissipation and aquatic nontarget organism accumulation studies on Aatrex Nine-O for forestry use at Oregon City, Oregon. ABC Report No. 32989. Prepared by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by Ciba-Geigy Corporation, Greensboro, NC.

DIRECT REVIEW TIME = 24

REVIEWED BY: J. Harlin TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

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APPROVED BY: S. Termes
TITLE: Chemist
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TEL: 557-2243

SIGNATURE: Nov. 18/1985

CONCLUSIONS:

Field Dissipation - Forestry

The forestry dissipation study may fulfill data requirements if acceptable freezer storage stability data and an adequate description of the forest site is submitted.

Field Accumulation - Aquatic Nontarget Organisms

This portion of the study can only be considered to provide supplemental information.

SUMMARY OF DATA BY REVIEWER:

Field Dissipation - Forestry

Field Accumulation - Aquatic Nontarget Organisms

Atrazine (Aatrex Nine-O, 90% G) was applied aerially at 4 lb ai/A to 10 acres of an immature Douglas fir forest located in Oregon City, Oregon, on April 4, 1985. In tree foliage samples, atrazine was 168.2-294.2 ppm immediately posttreatment, 76.7-88.0 ppm at 7 days, 6.6-10.5 ppm at 29 days, and 1.6-3.2 ppm at 88 days posttreatment. The registrant-calculated half-life for atrazine in foliage was 13 days. At 88 days posttreatment, the degradates . . .

2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033) and

2-amino-4-chloro-6-ethylamino-s-triazine (G-28279) . . .

were isolated at maximum concentrations of 0.357 ppm (11% of the recovered) and 0.061 ppm (2% of the recovered), respectively.

In leaf litter samples, atrazine was 73.1-114.2 ppm immediately post-treatment, 21.8-27.9 ppm at 29 days, 7.2-8.1 ppm at 88 days, and 0.60-3.4 ppm at 364 days posttreatment; the registrant-calculated half-life was 66 days. 2-Amino-4-chloro-6-isopropylamino-s-triazine was isolated at a maximum concentration of 0.73 ppm (3.3% of the recovered) at 14 days posttreatment; 2-amino-4-chloro-6-ethylamino-s-triazine was isolated at a maximum concentration of 0.30 ppm (1% of the recovered) at 29 days posttreatment.

In soil (0- to 6-inch depth) that was not covered with leaf litter, atrazine concentrations were variable with no discernible pattern, ranging from 0.075 to 4.3 ppm. 2-Amino-4-chloro-6-isopropylamino-s-triazine was isolated at up to 0.118 ppm (10.7% of the recovered; 128 days) in the 0- to 6-inch depth; the degradate 2-amino-4-chloro-6-ethyl-amino-s-triazine was <0.05 ppm (not detected) at all sampling intervals. In the 6- to 12- and 12- to 18-inch soil depths, atrazine was <0.05-0.432 ppm and <0.05-0.110 ppm, respectively.

In soil under leaf litter, atrazine concentrations were variable in the 0- to- 6-inch depth, ranging from 0.077 to 4.7 ppm, and were ≤0.088 ppm in the 6- to 12- and 12- to 18-inch depths.

Atrazine was <0.1 ppb in stream water (sampled up to 88 days posttreatment), <0.05 ppm in stream sediment (sampled up to 90 days posttreatment), and <0.05 ppm in fish (sampled up to 29 days posttreatment).

DISCUSSION:

Field Dissipation - Forestry

1. All samples (except water samples) were stored frozen for various lengths of time before analysis. However, no storage stability data were provided to show that atrazine was stable in the samples for the length of storage. Since the water samples were shipped without refrigeration, a stability study was conducted using water samples. Recoveries from water samples fortified with atrazine at 0.05-1.0 µg/mL ranged from 92 to 164%.

2. The forest site was incompletely described. The location of foliage samples within the forest canopy, the density of the canopy, the thickness and composition of the litter layer, the rate of flow of water in the stream, and the degree of exposure of the stream to the atrazine spray during application were not stated. It was not stated whether the foliage was sampled inside or outside the crown.

3. The silt loam soil was misclassified in the study. Using the USDA Soil Textural Classification System, the soil classified by the study author as a silt loam soil was determined to be a loam soil. It is described as a loam soil in this report.

4. The registrant stated that an accurate half-life could not be calculated for soil under leaf litter, possibly due to the uneven distribution of leaf litter at the test site after reforestation. In addition, an accurate half-life could not be calculated for exposed soil. The data showed poor correlation for the first-order kinetics with a correlation coefficient value of -0.515 and an estimated half-life of 135 days. Exclusion of outlier data obtained from day 1 and 3 soil samples resulted in improved correlation (correlation coefficient of -0.924) and a calculated half-life of 87 days.

5. Soil sampled under the leaf litter on day 88 contained 0.089 ppm of 2-amino-4-chloro-6-isopropylamino-s-triazine. The registrant stated that it is likely these levels were caused by an external contamination or interference in the analysis.

Field Accumulation - Aquatic Nontarget Organisms

1. It could not be determined whether bottom, middle, and surface feeding fish from the native fish population were analyzed, since the registrant did not specify the species of fish analyzed. In addition, edible and nonedible tissues from the native fish population were only analyzed at intervals up to 3 days posttreatment. Analysis of edible, nonedible, and whole tissues taken from caged rainbow trout indicated that atrazine residues were <0.05 ppm at intervals up to 29 days posttreatment.

2. Movement of water into and out of the test site was not reported.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Atrazine (Aatrex Nine-O, 90% G, Ciba-Geigy Corporation) was applied by aircraft at 4 lb ai/A on April 4, 1985, to 10 acres of an immature Douglas-fir forest located in Oregon City, Oregon (45°17'07" N, 122°25'10" W). The test site had been clear cut and burned, then planted with 2-year-old Douglas fir trees in mid-March 1985. The test site was underlain with loam soil, had an elevation ranging from 420 to 600 feet, and a slope on the steepest eastern boundary ranging from 8 to 27°. For sampling purposes, the test area was divided into 72 subplots (each 66 x 73 feet) using a 9 x 8 grid. Prior to treatment, a 3 x 3-foot section of each cell was cleared of surface material to provide exposed soil sites. An untreated plot (50 x 50 feet) served as the control.

Foliage samples, taken by clipping branches from the transplanted trees, were sampled at intervals up to 88 days posttreatment. Soil under leaf litter and exposed soil (each sampled at 0- to 6-, 6- to 12-, and 12- to 18-inch depths), and leaf litter samples were taken at intervals up to 364 days posttreatment. Stream water and sediment samples from near the treatment area were taken for analysis at intervals up to 88 and 90 days posttreatment, respectively. The potential for accumulation of atrazine in fish taken from the stream was also investigated. Native fish (uncharacterized) were sampled at intervals up to 28 days following treatment. In addition, 30 rainbow trout (8 inches in length; fish not further characterized) that had been caged in the stream two weeks prior to the initiation of the study, were sampled at intervals up to 29 days posttreatment. All samples, except water samples, were stored frozen prior to analysis. The water samples were shipped at ambient temperature, then stored under refrigeration until analysis.

Soil samples from the treated and control test sites were ground and refluxed in acetonitrile:water (9:1). The extract plus flask rinses were filtered and combined, then evaporated to dryness on a rotary evaporator. The residues were redissolved in toluene and analyzed for atrazine and its degradates by GC using a Hall electrolytic conductivity detector operating in the nitrogen specific mode. The detection limit was 0.05 ppm. Recoveries from soil samples fortified with atrazine at 0.05-0.5 ppm were 62-100%. Recoveries from soil samples fortified with G-28279 or G-30033 at 0.05-0.5 ppm were 66-98% and 68-97%, respectively.

Foliage samples were ground with dry ice, then blended with chloroform for 10 minutes. The extracts were filtered through anhydrous sodium sulfate into a graduated cylinder; the blender jar was rinsed with chloroform and the rinse was transferred to the graduated cylinder. An aliquot of the chloroform extract was roto-evaporated, and the resulting residues were redissolved in n-hexane. The hexane solution was partitioned twice with acetonitrile. The acetonitrile fractions were combined, partitioned with fresh n-hexane, and evaporated to dryness using a rotary evaporator. The residues were dissolved in carbon tetrachloride and filtered through an aluminum oxide column. Atrazine was eluted from the column in carbon tetrachloride:ethyl ether (95:5), and the degradates G-30033 and G-28279 were eluted in carbon tetrachloride:ethyl ether

(75:25). Both eluates were concentrated. The eluate containing atrazine was extracted with methylene chloride, evaporated to dryness, redissolved in toluene, and analyzed by GC using a Hall electrolytic conductivity detector operating in the nitrogen specific mode. The G-30033/G-28279 residues were redissolved in methylene chloride and filtered through a Florisil column in methylene chloride:acetonitrile (9:1). The eluant was evaporated, dissolved in toluene, and analyzed by GC as described previously. The detection limit was 0.05 ppm. Recoveries from leaf foliage samples fortified with atrazine at 0.05 or 0.5 ppm were 72-86%, and for leaf foliage samples fortified with G-28279 and G-30033, recoveries were 67-80% and 90-104%, respectively.

Leaf litter samples were ground, refluxed with acetonitrile:water (9:1), and filtered. An aliquot of the extract was roto-evaporated, then partitioned three times with methylene chloride. The methylene chloride fractions were filtered through anhydrous sodium sulfate, combined, and dried; the resulting residues were redissolved in n-hexane. The n-hexane solutions were further extracted and analyzed by GC as described for the foliage samples. The detection limit was 0.05 ppm. Reported recoveries from leaf litter samples fortified with atrazine at 0.05-0.5 ppm ranged from 62 to 86%. Recoveries from leaf litter samples fortified with G-28279 and G-30033 at 0.05-0.5 ppm were 52-82% and 68-94%, respectively.

Stream sediment samples were air-dried, ground, extracted with acetonitrile:water (9:1), and analyzed according to procedures outlined for leaf litter samples. Stream water samples were filtered and extracted three times with methylene chloride:n-hexane (9:1). The extracts were roto-evaporated, redissolved in toluene, and analyzed by GC. The detection limit was 0.1 ppb. Reported recovery efficiencies from stream sediment samples fortified with atrazine at 0.05-0.5 ppb ranged from 78 to 124%. Recoveries from stream water samples fortified with atrazine at 0.1-1.0 ppb ranged from 68 to 85%.

Whole fish, and edible and nonedible fish tissues were homogenized and extracted with methanol:water (4:1). The extracts were filtered, roto-evaporated, and extracted two times with methylene chloride. The methylene chloride extracts were evaporated to dryness using a rotary evaporator, filtered through aluminum oxide and Florisil columns, and analyzed by GC as described previously. Detection limits for the various fish fractions were 0.05 ppm. Recoveries from edible and nonedible tissues fortified with atrazine at 0.05-0.5 ppm were 63-93%, and recoveries from fish samples fortified with G-28279 or G-30033 at 0.05-0.5 ppm were 56-91% and 78-98%, respectively. Recoveries from whole fish fortified with atrazine at 0.05-0.5 ppm were 84-108%.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

Atrazine

Page_____ is not included in this copy.

Pages 147 through 163 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

✓ FIFRA registration data.

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

ACCUMULATION STUDIES

DATA EVALUATION RECORD

STUDY 34

CHEM 080803 Atrazine §165-1

FORMULATION--90--FORMULATION NOT IDENTIFIED

FICHE/MASTER ID 40431342
Simoneaux, B. 1987a. Uptake and characterization of ^{14}C-atrazine metabolites in greenhouse grown corn. Laboratory Study No. ABR-87093. Prepared and submitted by Ciba-Geigy Corporation, Greensboro, NC.

FICHE/MASTER ID 40431343
Simoneaux, B. 1987b. Uptake and characterization of ^{14}C-atrazine metabolites in greenhouse grown rotational plants. Laboratory Study No. ABR-87103. Prepared and submitted by Ciba-Geigy Corporation, Greensboro, NC.

DIRECT REVIEW TIME = 16

REVIEWED BY: J. Harlin TITLE: Staff Scientist

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SIGNATURE: Nov. 18, 1988

CONCLUSIONS:

Confined Accumulation - Rotational Crops

The study on the uptake and characterization of [^{14}C]atrazine metabolites in greenhouse-grown corn (40431342) is included here because it provided the information on soil treatment, soil characteristics, preparation of test pails, etc., preceding planting and harvesting of the corn prior to rotation of crops. The study provided confirmation of the application rate of atrazine. At the 2 lb ai/A rate used in the study, the amount of pesticide expected in the 0- to 3-inch soil core is 2 ppm; zero-day sampling gave a value of 2.17 ppm.

According to the author, the rate of 2.0 lb ai/A was chosen because it is the recommended rate for the loamy sand soil used in the study. However, higher application rates (up to 8 lb ai/A, preplant and/or preemergence) are known for corn and, therefore, the present study can only support registration for uses at maximum rates of 2 lb ai/A.

At the two rotation intervals studied (108 days posttreatment for lettuce, beets, and soybeans; 363 days posttreatment for spring wheat), residues were still detected in these crops when harvested at maturity. Thus, a 108-day rotation interval is inadequate for lettuce, beets, and soybeans. For winter wheat, the one-year rotation interval is also inadequate.

In the reviewed rotational crop study (40431343), it was also noted that the laboratory and greenhouse methods were incompletely described and that characterization of [^{14}C]residues in plants was inadequate (identity of degradates was not confirmed). Therefore, the submitted study does not fulfill data requirements for confined rotational crop studies.

Based on the rotational crop data reviewed, EFGWB concludes that residues are likely to occur in all crops rotated one year or more following application of atrazine at current use rates. As a consequence, appropriate rotational crop intervals for atrazine cannot be established. The registrant must petition the Dietary Exposure Branch (DEB/HED) for suitable tolerances for all crops to be rotated.

SUMMARY OF DATA BY REVIEWER:

[^{14}C]Atrazine (98% radiochemical purity; specific activity 20.6 Ci/mg) was applied at a nominal rate of 2 lb ai/A to sandy loam pails (greenhouse conditions). At zero-time sampling, the [^{14}C]atrazine equivalents in the 0- to 3-inch soil cores were 2.17 ppm. Corn (target crop) was planted (seeds; planting methods and upkeeping not included in the report, but referenced), and then harvested (that is, removed from the pails) at maturity (15 weeks). The amount of [^{14}C]atrazine equivalents remaining in the 0- to 3-inch soil core was 1.41 ppm (of which 78.8% was nonextractable materials, 8% atrazine, 3.6% hydroxyatrazine, and 2.0% dealkylated hydroxyatrazine). After the corn was harvested, the 1-inch soil layer was tilled.

Rotational crops: Leaf lettuce, sugar beets, and soybeans were planted 108 days after pesticide treatment. The reported amount of [^{14}C]atrazine equivalents in the 0- to 3-inch soil core after 108 days was 1.095 ppm. Although crops were analyzed at intermediate stages of development, the results for each crop harvested at maturity are as follows:

a) Lettuce. After 53 days of planting (161 days posttreatment), the amount of [^{14}C]residues in leaves was 0.089 ppm. Of this, 54.66% were organosoluble residues, 30.15% water-soluble residues, and 22.75% unextractables. The organic fraction was analyzed by TLC alone by cochromatography with standards of atrazine and its degradates and gave 18.7% atrazine, 1.97% G-28279, 6.67% G-30033, and

Ø.22% G-28273. Aqueous-soluble fractions, analyzed by cation exchange column chromatography, are considered to be polar metabolites.

b) Sugar beets. Sugar beets were harvested after 145 days (253 days posttreatment). The total amount of [^{14}C]atrazine equivalents was Ø.Ø46 ppm in tops and Ø.Ø43 ppm in beets. Residues were not characterized.

c) Soybeans. Soybeans were harvested after 136 days of planting (244 days posttreatment). The [^{14}C]atrazine equivalents were Ø.417 ppm (stalks), Ø.259 ppm (pods), and Ø.256 ppm in beans. Organosoluble fractions were 11.4Ø, 5.64, and 19.56% (stalks, pods, and beans, respectively). Aqueous-soluble fractions were 58.22, 47.28, and 34.67% (stalks, pods, and beans, respectively). Nonextractables were 28.Ø2, 37.63, and 46.64% (stalks, pods, and beans, respectively). The organic fraction was not analyzed.

Spring wheat was planted after 363 days (1 year) of treatment and harvested 94 days later. The amount of radioactivity (atrazine equivalents) found was Ø.32Ø ppm in stalks, Ø.Ø4Ø ppm in grain, and Ø.199 ppm in hulls. Percent of total radioactivity in organic-soluble fractions was 15.82 and 11.87 (stalks and hulls, respectively). In water-soluble fraction, 63.8 and 6Ø.22% was found for stalks and hulls, respectively. Nonextractables accounted for 28.31% in stalks and 25.64% in hulls. No data was provided for grain. At the time of planting spring wheat (363 days posttreatment), the amount of total [^{14}C]atrazine residues in the Ø- to 3-inch soil core was Ø.366 ppm.

DISCUSSION:

1. The analytical procedures for the soil and plant extraction procedures were referenced (Analytical Method Nos. AG-223, AG-214, AG-351, AG-252, and AG-276), but the references were not provided to review. Because details of the analytical methods were not available, it was difficult to interpret some of the data presented. Also, the detection limits and recovery efficiencies from fortified samples were not provided for the analytical methods. Information concerning crop maintenance, such as the watering schedule, was referenced (BIOL- 86Ø15 and 87ØØ5) but not provided for review.

2. [^{14}C]Residues were not characterized in the organic extracts of mature sugar beets, and soybeans, and spring wheat. [^{14}C]Residues in the organic extracts of mature lettuce were identified only by cochromatography on TLC plates with reference standards; additional analyses using HPLC and/or GS/MS should have been conducted to confirm the identities of the degradates.

3. The extracts of mature lettuce, soybeans, and wheat were analyzed for total [^{14}C]residues, then repartitioned and reanalyzed. Data for the two analyses were very different. Data for extracts of the immature plants should not be compared to the repartition data for extracts of the mature plants, because analytical methods were different. The study author appears to believe the repartition data are more accurate; therefore,

only the repartition data are reported in the Data Summary.

4. Plant and soil samples were stored frozen prior to analysis; however, no storage stability data were provided.

5. The data were reported in terms of "percent of total radioactivity"; this was interpreted to mean "% of the recovered" in this review.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Ring-labeled [^{14}C]atrazine (radiochemical purity 98.5%, specific activity 20.6 µCi/mg, Ciba-Geigy) was applied at ≈2 lb ai/A to loamy sand soil (81.2% sand, 12% silt, 6.8% clay, and 1.5% organic matter, pH 6.7, CEC 3.2 meq/100 g). The treated soil was divided between 20 greenhouse test pails (12-quart size), and each pail was planted to corn; pails containing untreated soil were similarly planted to corn to serve as controls. The plants were maintained in the greenhouse according to BIOL-86015 (not provided to review). The corn was harvested on July 11, 1986, and the soil was immediately planted to lettuce, sugar beets, soybeans or spring wheat (108 days posttreatment). The treated and control crops were maintained in separate 10 x 12-foot air-conditioned greenhouse cubicles at 50-85°F with a 13- to 14-hour photoperiod; maintenance details were provided in BIOL-87005, which was not provided to review. Leaf lettuce was sampled at 50 and 100% of maturity (140 and 161 days posttreatment). Sugar beets were harvested at 25, 50, and 100% of maturity (184, 214, and 283 days posttreatment). Soybeans were harvested at 25, 50, and 100% of maturity (177, 200, and 274 days posttreatment). Because of problems with the wheat, the spring wheat was replanted on March 23, 1987 (363 days posttreatment); wheat was sampled at 25, 50, and 100% of maturity (400, 420, and 450 days posttreatment). Sampling data are provided in Table 2. Soil cores (0- to 3-, 3- to 6-, 6- to 8-inch depths) were sampled at 0, 28, 77, 105, 108, 202, 253, and 363 days posttreatment. All samples were frozen prior to analysis.

The plant and soil samples were analyzed according to Analytical Method Nos. AG-223, AG-214, AG-351, AG-252, and AG-276. The plant samples were ground, and portions of each sample were analyzed for total radioactivity by LSC following combustion. Plant samples containing >0.05 ppm of radioactivity and soil samples containing >0.1 ppm of radioactivity were extracted using analytical Method Nos. AG-214 and AG-351. Organosoluble [^{14}C]residues were analyzed by TLC, and water-soluble residues were analyzed by ion exchange chromatography. The organosoluble fractions were analyzed by one-dimensional TLC using silica gel plates developed in either ethyl acetate:hexane (70:30) to determine the chloro-triazine derivatives or in chloroform:methanol:formic acid:water (70:25:4:2) for the hydroxy-triazine derivatives. Radioactive areas on the TLC plates were visualized using spark chamber photography, identified by comparison to standards, and quantified by LSC. The aqueous fractions were filtered through a cation exchange column packed with Aminex A-4 resin. [^{14}C]-Polar compounds were eluted from the column with a linear gradient of 0.1 N ammonium carbonate to 1.0 N ammonium carbonate to 0.1 N ammonium hydroxide, followed by a 0.1 N ammonium hydroxide strip (it was not specified how various fractions were quantified).

Atrazine

Page_____ is not included in this copy.

Pages 171 through 196 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

__✓__ FIFRA registration data.

____ The document is a duplicate of page(s) _____.

____ The document is not responsive to the request.

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

DATA EVALUATION RECORD

STUDY 35

CHEM 080803 Atrazine §165-1

FORMULATION--90--FORMULATION NOT IDENTIFIED

FICHE/MASTER ID 00103164
Fischer, W., N. Cargile, and J. Cassidy. 1979a. Degradation of delta-^{14}C-atrazine in field soil and uptake of the degradation products into rotation lettuce. Report No. ABR-79065. Unpublished study received June 10, 1982 under 100-631. Prepared and submitted by Ciba-Geigy Corporation, Greensboro, NC; CDL:070915-A.

FICHE/MASTER ID 00103163
Fischer, W., N. Cargile, and J. Cassidy. 1979b. Degradation of delta-^{14}C-atrazine in field soil and uptake of the degradation products into rotation sugar beets. Report No. ABR-79064. Unpublished study received June 10, 1982 under 100-631. Prepared and submitted by Ciba-Geigy Corporation, Greensboro, NC; CDL:070914-X.

FICHE/MASTER ID 00118936
Fischer, W., N. Cargile, and J. Cassidy. 1979c. Degradation of delta-^{14}C-atrazine in field soil and uptake of the degradation products into rotation winter wheat. Report No. ABR-79063. Unpublished study received June 10, 1982 under 100-631. Prepared and submitted by Ciba-Geigy Corporation, Greensboro, NC; CDL:070914-W.

DIRECT REVIEW TIME = 24

REVIEWED BY: J. Harlin TITLE: Staff Scientist

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SIGNATURE: Nov. 18, 1988

197

CONCLUSIONS:

Confined Accumulation - Rotational Crops

This study is unacceptable and would not fulfill EPA Data Requirements for Registering Pesticides because [^{14}C]residues in plants were inadequately characterized, [^{14}C]residues in soil were not characterized at most sampling intervals, and more importantly, the soil was not sampled until 17 weeks posttreatment (at which time only 20.3% of the [^{14}C]residues were atrazine). The soil was not sampled at all planting intervals, storage stability data were not provided, field test data were incomplete, and the test substance was not completely characterized. In addition, this study would not support registration for uses at application rates higher than 3 lb ai/A. Analytical methods for extraction and analysis of plant and soil samples were not provided.

SUMMARY OF DATA BY REVIEWER:

[^{14}C]Atrazine residues accumulated in winter wheat planted in silt loam soil 67 weeks after the soil had been treated with ring-labeled [^{14}C]-atrazine (purity unspecified, specific activity 33 μCi/mg) at 3 lb ai/A. Total [^{14}C]atrazine residues were 4.91 ppm in whole plants at 25% maturity (7 weeks) and decreased to 0.10 ppm at 50% maturity (32 weeks). [^{14}C]Residues were 0.17 ppm in stalks and 0.06 ppm in heads at 75% maturity, and 0.30 ppm in straw and 0.09 ppm in grain at maturity. [^{14}C]Residues in the organic extracts decreased from 31.5% of the recovered (0.03 ppm) in whole plants at 50% maturity, to 19.0% (0.03 ppm) in stalks at 75% maturity, and to 4.1% of the recovered (0.01 ppm) in straw and <1.7% (not detected) in the grain at maturity. Total radioactivity in the polar extracts ranged from 25.8 to 67.1% of the recovered (minimum in grain at maturity, maximum in stalks at 75% maturity). Unextractable [^{14}C]residues increased from 17.9% of the recovered radioactivity in whole plants at 50% maturity, to 23.7% in stalks at 75% maturity, to 56.5% in straw and 83.0% in grain at maturity.

[^{14}C]Atrazine residues accumulated in lettuce planted at 100 weeks posttreatment. Total [^{14}C]residues were 0.17 ppm in the lettuce leaves at 50% maturity, 0.40 ppm at 75% maturity, and 0.17 ppm at maturity. At maturity, 21.0% of the recovered radioactivity (0.04 ppm) was in the organic extract, 69.5% was in the polar fraction, and 14.9% was unextractable.

[^{14}C]Atrazine residues accumulated in sugar beets planted at 100 weeks posttreatment. Total [^{14}C]residues were 0.14 ppm in whole plants at 25% maturity, 0.03 ppm in whole plants at 75% maturity, and 0.01 ppm in beet roots and 0.10 ppm in beet tops at maturity. In whole beets at 25% maturity, 20.5% of the recovered [^{14}C]residues (0.03 ppm) were in the organic extracts, 39.2% were in the polar extracts, and 30.2% were unextractable. In sugar beet tops at maturity, 12.2% of the recovered [^{14}C]residues (0.01 ppm) were in the organic fraction, 47.1% were in the polar fraction, and 31.6% were unextractable.

2. The soil was not sampled until 17 weeks posttreatment, at which time only 20.3% of the recovered radioactivity was [^{14}C]atrazine. [^{14}C]-Residues in the soil were only characterized at 17, 100, 110, and 122 weeks posttreatment, and only in the 0- to 3-inch depth. That is, zero-time data (confirmation of application rate) is missing.

3. [^{14}C]Residues in plant samples were not adequately characterized. The 7-week wheat samples were analyzed only for total residues; the 38-week wheat samples were only fractioned into organic, polar, and unextractable [^{14}C]residues. The polar [^{14}C]residues from the 32-week wheat samples and "mature" grain and straw samples (possibly the 42-week samples) were analyzed to identify specific compounds, and polar compounds were only identified, not quantified. The 4-and 6-week lettuce samples and the 18-week beet samples were analyzed only for total residues. The mature (8-week) lettuce and the 5- and 22-week beet samples were fractioned into organic, polar, and unextractable [^{14}C]residues, but only the polar [^{14}C]residues were analyzed to identify specific compounds, and polar compounds were only identified, not quantified. The major polar degradate in all plants cochromatographed with G-11957; no further analyses were conducted to confirm the identity of this degradate.

4. Plant and soil samples were frozen prior to analysis; however, no storage stability data were provided.

5. Field test data, such as air and soil temperatures, slope of the field, and depth to the water table, were not complete. Cumulative rainfall from October, 1976 through July, 1978 was 50.42 inches. Cumulative rainfall from May, 1977 to September, 1978 was 48.17 inches.

6. The purity of the test substance was not reported.

7. The data were reported in terms of "percent of total radioactivity"; this term was interpreted to mean "% of the recovered" in this review.

The major [^{14}C]compound in the polar extracts of the 32-week wheat whole plants and "mature" straw and grain, the 8-week lettuce leaves, and the 5-week beet whole plants, and the mature beet tops was . . .

2,4-hydroxy-6-isopropylamino-s-triazine (GS-11957).

Other compounds, described as being present in "minor amounts" in the plants were . . .

2-amino-4-ethylamino-6-hydroxy-s-triazine (GS-17792),

2-amino-4-hydroxy-6-isopropylamino-s-triazine (GS-17794), and

2-ethylamino-4-hydroxy-6-isopropylamino-s-triazine (G-34038).

In the 0- to 3-inch layer of soil from the wheat subplots, total [^{14}C]-residues decreased from 1.72 ppm at 17 weeks posttreatment to 0.95 ppm at 67 weeks (planting) and to 0.74 ppm at 110 weeks (mature harvest). In the 0- to- 3-inch layer of soil from the beet/lettuce subplots, total [^{14}C]residues were 0.89 ppm at 100 weeks (planting) and 0.97 ppm at 122 weeks (mature harvest). [^{14}C]Atrazine was 20.3% of the recovered radioactivity at 17 weeks (wheat subplot) and 7.6% at 122 weeks (beet subplot). Other degradates identified in the soil were . . .

2-hydroxy-4-ethylamino-6-isopropylamino-s-triazine (G-34048; 11.0% of the recovered at 17 weeks, not detected at 110 weeks),

2-amino-4-chloro-6-ethylamino-s-triazine (G-28279; 6.8 and 3.1% of the recovered at 17 and 122 weeks),

2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033; 1.9% of the recovered at 17 weeks, 5.3% at 110 weeks, and 3.1% at 122 weeks), and

2,4-diamino-6-chlorotriazine (G-28273; 2.3 and 0.8% of the recovered at 17 and 122 weeks).

Between 17 and 110 weeks posttreatment, total radioactivity in the 3- to 6-inch depth of the wheat subplots decreased from 1.09 to 0.41 ppm and in the 6- to 9-inch depth decreased from 0.50 to 0.18 ppm. In the beet/lettuce subplots at 122 weeks posttreatment, total radioactivity in the 3- to 6-inch depth was 0.60 ppm and in the 6- to 9-inch depth was 0.17 ppm.

DISCUSSION:

1. The analytical methods for the soil and plant extraction procedures and for identification of atrazine and its degradates were referenced, but the references were not provided to review. The limits of detection for the analytical methods and recoveries from fortified samples were not provided.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Ring-labeled [^{14}C]atrazine (specific activity 33 μCi/mg, Ciba-Geigy Corporation, purity unspecified) was applied at 3 lb ai/A on June 6, 1976, to a small field plot (3 x 6 feet) of silt loam soil (20.4% sand, 59.2% silt, 20.4% clay, 2.0% organic matter, pH 6.2, CEC 13.9 meq/100 g) located in York, Nebraska. Field corn was grown in the plot until September, 1976. One subplot (3 x 3 feet) was planted to winter wheat at 17 weeks posttreatment; the second subplot was planted to lettuce spaced between rows of sugar beets on May 28, 1977 (47 weeks posttreatment). The 1976 winter wheat and the 1977 lettuce and sugar beet plantings failed, and the plots were replanted with winter wheat on September 16, 1977, and with lettuce and sugar beets on May 3, 1978. The wheat was harvested at 7, 32, 38, and 43 weeks postplanting (73.7, 98.7, 104.7, and 109.7 weeks posttreatment). The 38- and 43-week wheat samples (representing 75 and 100% of mature) were separated into heads/grain and stalks/straw. Lettuce was sampled at 4, 6, and 8 weeks postplanting (104, 106, and 108 weeks posttreatment). Sugar beets were sampled at 5, 18, and 22 weeks postplanting (105, 118, and 122 weeks posttreatment). Soil (0- to 3-, 3- to 6-, and 6- to 9-inch depths) was sampled at 17, 47, 67, 74, 99, 100, 105, 110, 118, and 122 weeks posttreatment (equivalent to the first and second wheat plantings, first and second beet/lettuce plantings, and each of four wheat and three beet samplings). Plant and soil samples were kept frozen until analysis.

Total radioactivity in plant and soil samples was determined by LSC following combustion. The wheat samples were extracted according to analytical Method No. AG-255. The lettuce and beet samples were extracted according to analytical Method No. AG-214. Total triazine moieties in plant and soil samples were determined as cyanuric acid according to analytical Method No. AG-284. Chlorotriazines were characterized by TLC using a saturated system of chloroform:methanol:acetic acid:water (100:20:4:2). Polar degradates in the plant samples were identified by ion exchange chromatography using Method No. AG-248.

LETTUCE AND SUGAR BEETS

Atrazine

Page_____ is not included in this copy.

Pages 204 through 229 are not included.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product impurities.

_____ Description of the product manufacturing process.

_____ Description of quality control procedures.

_____ Identity of the source of product ingredients.

_____ Sales or other commercial/financial information.

_____ A draft product label.

_____ The product confidential statement of formula.

_____ Information about a pending registration action.

✓ FIFRA registration data.

_____ The document is a duplicate of page(s) _____.

_____ The document is not responsive to the request.

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

DATA EVALUATION RECORD

STUDY 36

CHEM 080803 Atrazine §165-1

FORMULATION--90--FORMULATION NOT IDENTIFIED

FICHE/MASTER ID 00103167
Hermes, P. and J. Knaack. 1972a. The uptake of aged ^{14}C-atrazine and metabolites in rotation oats and the degradation of atrazine in soil: Report No. GAAC-72019. Unpublished study received June 10, 1982 under 100-631. Prepared and submitted by Ciba-Geigy Corporation, Greensboro, NC; CDL:070915-D.

FICHE/MASTER ID 00103170
Hermes, P. and J. Knaack. 1972b. The uptake of aged ^{14}C-atrazine and metabolites in rotation soybeans and the degradation of atrazine in soil: Report No. GAAC-72032. Unpublished study received June 10, 1982 under 100-631. Prepared and submitted by Ciba-Geigy Corporation, Greensboro, NC; CDL:070915-G.

DIRECT REVIEW TIME = 15

REVIEWED BY: J. Harlin TITLE: Staff Scientist

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SIGNATURE:

Nov. 18, 1988

CONCLUSIONS:

Confined Accumulation - Rotational Crops

This study is unacceptable because the soil was not sampled until 22 weeks posttreatment (at which time only 12.4% of the [^{14}C]residues were atrazine); therefore, the application rate could not be confirmed. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides because [^{14}C]residues in plants were inadequately characterized, [^{14}C]residues in soils were not characterized at most sampling intervals, storage stability data were not provided, field test

data were incomplete, and the test substance was not completely characterized.

SUMMARY OF DATA BY REVIEWER:

[^{14}C]Atrazine residues accumulated in oats that were planted in silt loam soil 52 weeks after the soil had been treated with ring-labeled [^{14}C]-atrazine (purity and specific activity unspecified) at 3 lb ai/A. Total [^{14}C]atrazine residues were 0.461 ppm in immature oat stalks (56 weeks posttreatment); total residues were 0.287 ppm in mature stalks, 0.391 ppm in husks, and 0.252 ppm in grain (67 weeks posttreatment).

[^{14}C]Atrazine residues accumulated in soybeans planted at 52 weeks posttreatment. Total [^{14}C]atrazine residues were 1.93 ppm in immature soybean stalks (56 weeks posttreatment); total residues were 0.775 ppm in mature stalks, 0.737 ppm in pods, and 0.259 ppm in grain (77 weeks posttreatment. Total [^{14}C]residues were 0.247 ppm in soybean meal and 0.007 ppm in soybean oil derived from mature samples. Minor [^{14}C]compounds in the methanol:water extracts of mature soybean stalks and leaves were . . .

2-ethylamino-4-hydroxy-6-isopropylamino-s-triazine (G-34048; 0.039 ppm; 5.0% of the recovered),

2-amino-4-hydroxy-6-isopropylamino-s-triazine (GS-17794; 0.029 ppm; 3.8% of the recovered), and

2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033; 0.005 ppm; 0.7% of the recovered).

Other degradates in the mature soybean stalks and leaves were an unidentified atrazine conjugate (18.4% of the recovered) and the glutathione conjugate of atrazine (3.1% of the recovered); unextractable [^{14}C]residues were 9.0% of the recovered.

In the 0- to 3-inch soil depth, total [^{14}C]residues were 1.130 ppm at 22 weeks posttreatment, 1.585 ppm at 52 weeks (planting), 1.696 ppm at 67 weeks (mature oat harvest), and 1.139 ppm at 77 weeks (mature soybean harvest). [^{14}C]Atrazine decreased from was 0.14 ppm (12.4% of the recovered) at 22 weeks posttreatment, to 0.082 ppm (5.17% of the recovered) at 52 weeks, and to 0.020 ppm (1.76% of the recovered) at 77 weeks. Other degradates identified in the soil were . . .

2-ethylamino-4-hydroxy-6-isopropylamino-s-triazine (G-34048; 27.12-38.45% of the recovered at all intervals with no discernable pattern);

2-amino-4-hydroxy-6-isopropylamino-s-triazine (GS-17794; decreasing from 6.2% of the recovered at 22 weeks to 1.67% at 77 weeks);

2-amino-4-ethylamino-6-hydroxy-s-triazine (GS-17792; <0.06-3.0% of the recovered);

2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033; decreasing from 2.7% of the recovered at 22 weeks to 0.18% at 77 weeks);

2-amino-4-chloro-6-ethylamino-s-triazine (G-28279; decreasing from 1.8% of the recovered at 22 weeks to <0.09% at 77 weeks); and

2,4-diamino-6-chloro-s-triazine (G-28273; <0.06%-0.50% of the recovered).

In the 3- to 6-inch depth, total [^{14}C]residues were 0.10 ppm at 22 weeks posttreatment, 0.251 ppm at 52 weeks, 0.398 ppm at 67 weeks, and 0.154 ppm at 77 weeks; [^{14}C]compounds in the 3- to 6-inch depth at 52 and 67 weeks posttreatment were the same as those identified in the 0- to 3-inch depth. In the 6- to 9-inch depth, total [^{14}C]residues were 0.081 ppm at 52 weeks posttreatment, 0.078 ppm at 67 weeks, and 0.056 ppm at 77 weeks. In the 9- to 12-inch depth, total [^{14}C]residues were 0.079 ppm at 52 weeks posttreatment, 0.066 ppm at 67 weeks, and 0.040 ppm at 77 weeks.

DISCUSSION:

1. The soil was not sampled immediately posttreatment; therefore, the application rate could not be confirmed. Only 12.4% of the recovered radioactivity (0.14 ppm) was [^{14}C]atrazine at the first sampling interval (22 weeks posttreatment). [^{14}C]Residues in the soil were only characterized in the 0- to 3-inch depth at 22, 52, 67, and 77 weeks posttreatment and in the 3- to 6-inch depth at 52 and 67 weeks posttreatment.

2. [^{14}C]Residues in plant samples were not adequately characterized. The oat samples (4-week stalks and 15-week stalks, husks, and grain) were analyzed only for total residues. The 4-week soybean stalk and 25-week soybean pod, grain, meal, and oil samples were analyzed only for total residues. Degradates were only characterized in mature (25-week) stalks and leaves.

3. Plant and soil samples were frozen prior to analysis; however, no storage stability data were provided.

4. Field test data, such as precipitation, air and soil temperatures, slope of the field, and depth to the water table, were not provided.

5. The purity and specific activity of the test substance were not reported.

6. The test soil was not completely characterized.

7. The data were reported in terms of "percent of total radioactivity"; this term was interpreted to mean "% of the recovered" in this review.

8. TLC analysis of soil and plant samples was conducted using one or two solvent systems; however, development of the plates in three solvent systems of different polarity is recommended for maximum confidence in separation of [^{14}C]compounds.

9. A leafy vegetable crop (such as lettuce, mustard, or spinach) and a root vegetable crop (such as beets or carrots) were not planted as rotational crops.

10. The limits of detection for the analytical methods and recovery efficiencies from fortified samples were not provided.

11. Additional soil samples were analyzed from a bare subplot of treated soil that was rototilled in May, 1971. At 77 weeks posttreatment, total [^{14}C]residues were 1.445 ppm in the 0- to 3-inch depth, 0.395 ppm in the 3- to 6-inch depth, 0.110 ppm in the 6- to 9-inch depth, and 0.087 ppm in the 9- to 12-inch depth. In the 0- to 3-inch depth, [^{14}C]atrazine was 0.023 ppm (1.59% of the recovered). Other degradates identified in the 0- to 3-inch depth were G-34048 (37.71% of the recovered), GS-17794 (4.29% of the recovered), GS-17794 (4.29% of the recovered), GS-17792 (0.69% of the recovered), G-30033 (0.27% of the recovered), G-28279 (0.20% of the recovered), G-28273 (0.06% of the recovered).

12. In an addendum to the original report dated January 13, 1973, it was determined that [^{14}C]residues persisted in soil sampled from the rototilled subplot (no crops planted) that was left fallow during 1972. At 101 and 124 weeks posttreatment, [^{14}C]residues in the upper 9 inches were 1.67-1.96 ppm. Analysis of soil samples taken from the fallow subplot at 101 and 124 weeks posttreatment indicated the presence of degradates isolated in the soybean and oat subplots; the major degradate identified was G-34048 (27.1-33.3% of the recovered radioactivity).

MATERIALS AND METHODS

MATERIALS AND METHODS:

Ring-labeled [^{14}C]atrazine (Ciba-Geigy Corporation, purity and specific activity unspecified) was applied at 3 lb ai/A on June 6, 1970, to a small field plot (3 x 5 feet) of silt loam soil (soil not further characterized) located in New Paltz, New York. Field corn was grown in the plot and harvested during 1970 (dates unspecified). In May 1971, the field plot was divided into subplots (each 2 x 3 feet), one of which was planted to oats and the other planted to soybeans (each crop planted at 52 weeks posttreatment). The oats were harvested at 4, 7, 10, and 15 weeks postplanting (52, 55, 62, and 77 weeks posttreatment). The 15-week mature oat samples were separated into stalks, husks, and grain. Soybeans were sampled at 4 and 25 weeks postplanting (52 and 77 weeks posttreatment). The 25-week mature soybean samples were separated into stalks, pods, and grain. Soil (0- to 3-, 3- to 6-, 6- to 9-, and 9- to 12-inch depths) was sampled at 22, 52, 67, and 77 weeks posttreatment. Plant and soil samples were kept frozen until analysis.

Total radioactivity in plant and soil samples was determined by LSC following combustion. Ground soybean seed samples were added to hexane, the mixture was filtered, and the filtrate was brought to volume with hexane. Aliquots of the filtrate were analyzed for total radioactivity by LSC, and the unextractable [^{14}C]residues were analyzed by LSC following combustion. The stalks and leaves from mature soybean plants were ground, extracted with methanol:water (90:10) under reflux for 2 hours and the extract was filtered. Aliquots of the extract were analyzed for total radioactivity by LSC following combustion. The extract was concentrated to dryness using rotary evaporation, and residual water, if present, was removed by azeotropic distillation with acetonitrile. The residue was dissolved in dry methanol, adsorbed on silica gel, and allowed to air dry. The dried gel was applied as a slurry in hexane to a silica gel column packed in hexane. The sample was chromatographed according to a referenced chromatographic procedure using various solvents to elute the metabolites. The methanol:water extracts were analyzed by TLC on Eastman Kodak sandwich plates developed in chloroform:methanol:formic acid:water (80:15:4:2). Total radioactivity was determined by LSC. Soil samples were extracted with acetonitrile:water (9:1), refluxed for one hour, and filtered. The acetonitrile:water extract was analyzed according to analytical method No. AG-179. Chlorotriazines were characterized by TLC using a solvent system of hexane:ethyl acetate (65:35). The hydroxy triazines were analyzed by TLC using a solvent system of chloroform:methanol:acetone (3:5:1.5).

Atrazine

Page_____ is not included in this copy.

Pages 236 through 268 are not included.

The material not included contains the following type of information:

____ Identity of product inert ingredients.

____ Identity of product impurities.

____ Description of the product manufacturing process.

____ Description of quality control procedures.

____ Identity of the source of product ingredients.

____ Sales or other commercial/financial information.

____ A draft product label.

____ The product confidential statement of formula.

____ Information about a pending registration action.

✓ FIFRA registration data.

____ The document is a duplicate of page(s) _____.

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

DATA EVALUATION RECORD

STUDY 37

CHEM 080803 Atrazine §165-1

FORMULATION--90--FORMULATION NOT IDENTIFIED

FICHE/MASTER ID 00103153
Cassidy, J. 1972. A rotational metabolism study of delta-^{14}C-atrazine in soil and soybeans 52 to 72 weeks after herbicide application to soil: M2-01-3P, M2-01-45: Report No. GAAC-72137. Unpublished study received June 10, 1982 under 100-631 and submitted by Ciba-Geigy Corp., Greensboro, NC; CDL:070914-L.

DIRECT REVIEW TIME = 12

REVIEWED BY: J. Harlin TITLE: Staff Scientist

EDITED BY: K. Patten TITLE: Task Leader

APPROVED BY: W. Spangler TITLE: Project Manager

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APPROVED BY: S. Termes
TITLE: Chemist
ORG: EFGWB/EFED/OPP
TEL: 557-2243

SIGNATURE: Nov. 18/1988

CONCLUSIONS:

Confined Accumulation - Rotational Crops

This study is unacceptable and would not fulfill EPA Data Requirements for Registering Pesticides because [^{14}C]residues in plants were not adequately characterized, [^{14}C]residues in soil were not characterized at most sampling intervals, and more importantly, the soil was not sampled until 52 weeks posttreatment (at which time only 19.0% of the [^{14}C]residues were atrazine). Also, the soil was not sampled at all planting intervals, storage stability data were not provided, field test data were incomplete, and the test substance was not completely characterized. In addition, this study would not support registration for uses at application rates higher than 3 lb ai/A.

SUMMARY OF DATA BY REVIEWER:

[^{14}C]Atrazine residues accumulated in soybeans that were planted in silt loam soil one year after the soil had been treated with ring-labeled [^{14}C]atrazine (purity unspecified, specific activity 27 μCi/mg) at 3 lb ai/A. Total [^{14}C]atrazine residues were 0.67 ppm in 5-week-old whole plants, 0.48 ppm in 10-week-old whole plants, and 0.33 ppm in 15-week-old plants. [^{14}C]Residues in mature plant parts (20 weeks) were 0.31 ppm in stalks, 0.09 ppm in beans, 0.27 ppm in pods, 0.10 ppm in meal, and 0.03 ppm in bean oil. In 5-week-old whole plants, 19.4% of the recovered radioactivity (0.13 ppm) was in the organic extract, 71.6% was in the polar fraction, and 13.4% was unextractable. In mature stalks, 6.5% of the recovered radioactivity (0.02 ppm) was in the organic extract, 54.8% was in the polar fraction, and 35.5% was unextractable. The major [^{14}C]compound identified in the polar extracts of the 5-week whole plants was . . .

2-(N-acetylalany-3-thio)-4-ethylamino-6-isopropylamino-s-triazine (16.0% of the recovered).

An unidentified major degradate comprised 29.3 and 16.7% of the recovered, respectively, in 5-week whole plants and mature stalks. Minor degradates isolated in the 5-week whole plants and mature stalks were . . .

2-ethylamino-4-hydroxy-6-isopropylamino-s-triazine (G-34048),

2-amino-4-hydroxy-6-isopropylamino-s-triazine (GS-17794),

2-amino-4-ethylamino-6-hydroxy-s-triazine (GS-17792),

2-ethylamino-4-[α-glutamyl-(alanyl-3-thio)-glycyl]-6-isopropylamino-s-triazine, and

2-[N-bis(2-amino-2-carboxyethyl)sulfide]-4-ethylamino-6-isopropylamino-2-triazine.

Five minor unidentified degradates were also isolated in the 5-week whole plants and mature stalks. In addition, other compounds, described as being present in "minor amounts" in the 5-week whole plants were . . .

2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033),

2-amino-4-chloro-6-ethylamino-s-triazine (G-28279), and

2,4-diamino-6-chloro-s-triazine (G-28273).

Total [^{14}C]residues in the 0- to 3-inch soil depth were 2.31 ppm at 52 weeks posttreatment, 1.54 ppm at 57 weeks, 1.48 ppm at 62 weeks, and 1.64 ppm at 72 weeks posttreatment. [^{14}C]Atrazine was 19.0% of the recovered radioactivity at 52 weeks and 5.4% at 72 weeks. Other degradates identified in the soil were . . .

2-ethylamino-4-hydroxy-6-isopropylamino-s-triazine (G-34048; 31.6 and 33.8% of the recovered at 52 and 72 weeks),

2-amino-4-chloro-6-isopropylamino-s-triazine (G-30033; 2.6 and 0.6% of the recovered at 52 and 72 weeks),

2-amino-4-hydroxy-6-isopropylamino-s-triazine (GS-11794; 0.9 and 2.5% of the recovered at 52 and 72 weeks),

2-amino-4-chloro-6-ethylamino-s-triazine (G-28279; 0.9 and 0.5% of the recovered at 52 and 72 weeks), and

2-amino-4-ethylamino-6-hydroxy-s-triazine (GS-17792; 0.4 and 0.7% of the recovered at 52 and 72 weeks).

In the 3- to 6-inch depth, total radioactivity increased from 0.34 ppm at 52 weeks posttreatment to 1.44 ppm at 57 weeks, and then decreased to 0.79 ppm at 62 weeks and 0.12 ppm at 72 weeks posttreatment. Total [^{14}C]residues in runoff samples taken 4 and 8 inches from the edge of the test plot ranged from 0.04 to 0.26 ppm.

DISCUSSION:

1. The analytical methods for the soil and plant extraction procedures and for identification of atrazine and its degradates were referenced, but the references were not provided to review. The limits of detection for the analytical methods and recoveries from fortified samples were not provided.

2. The soil was not sampled until 52 weeks posttreatment, at which time only 19.0% of the recovered radioactivity was [^{14}C]atrazine. [^{14}C]Residues in the soil were only characterized at 52, 57, 62, and 72 weeks posttreatment. That is, confirmation of application rate is missing.

3. [^{14}C]Residues in plant samples were not adequately characterized. The 10- and 15-week whole plant, and mature (20-week) bean, pod, meal, and oil samples were analyzed only for total residues. The 5-week whole plant and mature (20-week) stalk samples were fractionated into organic, polar, and unextractable [^{14}C]residues, but only the polar [^{14}C]residues were analyzed to identify specific compounds.

4. Plant and soil samples were frozen prior to analysis; however, no storage stability data were provided.

5. Field test data, such as precipitation, air, and soil temperatures, slope of the field, and depth to the water table, were incomplete.

6. The purity of the test substance was not reported.

7. The data were reported in terms of "percent of total radioactivity"; this term was interpreted to mean "% of the recovered" in this review.

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8. Discrepancies existed between the data for plant samples analyzed by TLC and ion-exchange chromatography. This reviewer used the TLC data in summarizing the extractable and unextractable [^{14}C]residues in the plant samples.

9. According to EPA Guidelines, crops should be rotated at 30 days post-treatment (to simulate crop failure conditions), 120 days posttreatment, and at 1 year following treatment. In the present study, soybeans were planted one year following application of [^{14}C]atrazine to the test site.

10. A leafy vegetable crop (such as lettuce, mustard, or spinach) and a small grain crop (such as wheat or barley), were not planted as rotational crops.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Ring-labeled [^{14}C]atrazine (specific activity 27.7 μCi/mg, Ciba-Geigy Corporation, purity unspecified) was applied at 3 lb ai/A in May 1971 to a small field plot (3 x 5 feet) of silt loam soil (14.0% sand, 67.2% silt, 18.8% clay, 2.5% organic matter, pH 5.6, CEC 13.2 meq/100 g) located in York, Nebraska. Sorghum was grown in the plot until May, 1972. The plot was then planted to soybeans on May 17, 1972. The soybeans were harvested at 5, 10, 15, and 20 weeks postplanting (52, 57, 62, and 72 weeks posttreatment). Soil (0- to 3- and 3- to 6-inch depths) was sampled on the same schedule as described for the plant samples. Plant and soil samples were kept frozen until analysis.

Total radioactivity in soil and plant samples was determined by LSC following combustion. The plant samples were extracted according to analytical Method No. AG-214. The mature beans were pulverized, extracted with hexane, and analyzed for total radioactivity by LSC. The soil samples were extracted according to analytical Method No. AG-220. The organic fractions of plant and soil samples were analyzed by TLC according to analytical Method No. AG-204. Polar degradates in the 5- and 20-week-old soybean stalks were identified by ion exchange chromatography using a methanol:water (9:1) extract of ground plant material. The plant samples were extracted with methanol, the extract was evaporated to near dryness and dissolved in 0.1 N ammonium carbonate adjusted to pH 4.0. The buffered extract was chromatographed on glass columns containing Aminex A-5 with a linear gradient from 0.1 N ammonium carbonate at pH 4.0 to 1.0 N ammonium carbonate at pH 6.0. Fractions were collected and analyzed for total radioactivity by LSC.

Atrazine

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____ Description of quality control procedures.

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DATA EVALUATION RECORD

STUDY 38

CHEM 080803 Atrazine §165-4

FORMULATION--00--ACTIVE INGREDIENT

FICHE/MASTER ID 40431344
Forbis, A.D. 1986. Uptake, depuration, bioconcentration, and metabolite characterization of ^{14}C-atrazine by bluegill sunfish (Lepomis macrochirus). ABC Report No. 34737. Prepared by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by Ciba-Geigy Corporation, Greensboro, NC.

DIRECT REVIEW TIME = 16

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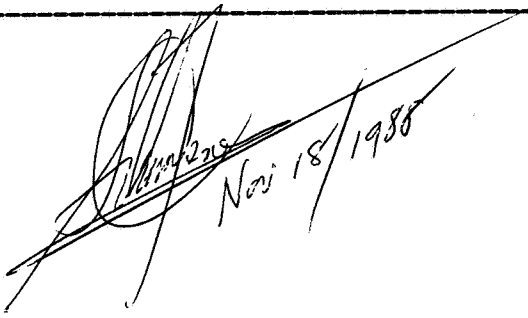

CONCLUSIONS:

Laboratory Accumulation - Fish

This study is acceptable and fulfills EPA Data Requirements for Registering Pesticides by providing information on the accumulation of [^{14}C]atrazine in laboratory fish.

SUMMARY OF DATA BY REVIEWER:

Total [^{14}C]atrazine residues accumulated in bluegill sunfish with maximum bioconcentration factors of 7.7x, 12x, and 15x in edible tissues (body, muscle, skin, skeleton), nonedible tissues (fins, head, internal organs), and whole fish, respectively, during 28 days of exposure to uniformly ring-labeled [^{14}C]atrazine (radiochemical purity >99%, specific activity 25.6 µCi/mg) at 0.10 ppm in a flow-through system. Maximum concentrations of [^{14}C]residues were 0.81 ppm in edible tissues (day 28), 1.6 ppm in nonedible tissues (days 7-28), and 1.3 ppm in whole fish (days 3, 14,

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and 28). Based on TLC analysis of 21- and 28-day fish samples, atrazine was 0.35-0.40 ppm in the edible tissues, 0.99-1.0 ppm in the nonedible tissues, and 0.085-0.087 ppm in the whole fish. One degradate, . . .

2-amino-4-chloro-6-ethyl-amino-s-triazine (G-28279),

comprised 5-11% of the total residues in the 21- and 28-day fish extracts. Three minor degradates . . .

2-amino-4-chloro-6-isopropyl-amino-s-triazine (G-30033),

2,4-diamino-6-chloro-s-triazine (G-28273), and

2-ethylamino-4-hydroxy-6-isopropylamino-s-triazine (G-34048)

were isolated at <5% of the total residues. After 21 days of depuration, [^{14}C]residues were 0.21 ppm in edible tissues, 0.38 ppm in nonedible tissues, and 0.28 ppm in whole fish; depuration rates were 74, 76, and 78%, respectively.

Throughout the study, the temperature of the treated water was 20-21°C, the pH ranged from 8.0 to 8.3, and the dissolve oxygen content ranged from 8.0 to 9.0 mg/L; values were comparable to the control aquarium. Total [^{14}C]residues in the treated water were 0.10-0.11 ppm during the exposure period.

DISCUSSION:

1. In the original document, it was stated that additional water samples from days 21 and 28 of the exposure period and day 14 of the depuration period were taken for possible metabolite characterization. However, it was unclear if the results of the TLC analysis of water were provided (no raw TLC data were provided). General degradate characterization statements made by the study authors may have been intended to include both the fish and the water samples. The study authors stated that the majority of the extracted [^{14}C]residues were identified as [^{14}C]atrazine; only two fish samples contained >0.05 ppm of residues that were not [^{14}C]atrazine.

2. The detection limit for the TLC method was not reported.

3. The source of the test substance was not specified.

4. The mortality of the fish during the study was not reported.

5. Although it was stated that TLC analysis of fish samples taken at day 14 of the depuration period was conducted, no raw data were provided.

6. A preliminary study was performed to determine the LC_{50} value of atrazine for bluegill sunfish. The 96-hour LC_{50} value was >10 mg/L. In view of these results, the study author chose an exposure level of 0.10 ppm (1/67 of the 96-hour LC_{50} of 6.7 mg/L provided by Ciba-Geigy Corporation) for the bioaccumulation study.

MATERIALS AND METHODS

MATERIALS AND METHODS:

Bluegill sunfish (Lepomis macrochirus; average length and weight of 57 mm and 7.3 g, respectively) were held in culture tanks on a 16-hour daylight photoperiod for ≥14 days prior to the initiation of the study. Flow-through aquatic exposure systems were prepared using two 100-L aquaria. Aerated well water (Table 1) was provided to each aquarium at a rate of ≈8.7 turnovers per day. The aquaria were immersed in a water bath and maintained at 22 ± 2°C.

Bluegill sunfish (120) were placed in each aquarium, and one aquarium was continuously treated with uniformly ring-labeled [^{14}C]atrazine (radiochemical purity >99%, specific activity 25.6 μCi/mg, source unspecified) at 0.10 ppm. The second aquarium served as an untreated control. Following a 28-day exposure period, the [^{14}C]atrazine-treated water was replaced with untreated water for a 14-day depuration period. The treated water was sampled prior to introducing the fish, and then water samples and fish (6, 15, or 25) were taken from the treated and control aquaria after 4 hours and 1, 3, 7, 14, 21, and 28 days of exposure. During the depuration period, water samples, and [^{14}C]atrazine-treated and untreated fish were taken on days 1, 3, 7, 10, 14, and 21 days.

Radioactivity in the water samples was quantified using LSC. The detection limit was 0.001 ppm. Aliquots of the water samples were extracted two times with methylene chloride. The extracts were combined, concentrated under a stream of nitrogen, and duplicate aliquots were analyzed for total radioactivity by LSC.

Pooled samples (3 fish) of whole fish, edible tissues (body, muscle, skin, skeleton) and nonedible tissues (fins, head, internal organs) were homogenized with dry ice and analyzed for total radioactivity using combustion and LSC. Reported recoveries of [^{14}C]residues from whole fish, edible tissues, and nonedible tissues fortified with 6612 dpm/50 μL of [^{14}C]atrazine ranged from 95 to 106%. The detection limits for whole fish, edible tissues, and nonedible tissues were 0.0476, 0.0467, and 0.0468 ppm, respectively.

Additional fish were sampled on days 21 and 28 of the exposure period and on day 14 of the depuration period, and were divided into edible, nonedible, and whole fish fractions for degradate identification. The edible and nonedible tissue fractions were extracted sequentially with methylene chloride:acetonitrile (50:50) and acetonitrile for 3 minutes in Sorvall blender cups; the samples were decanted through a glass fiber filter after each extraction. After the final extraction, the filter and blender cup were rinsed with methylene chloride. The methylene chloride:acetonitrile and acetonitrile extracts, and the methylene chloride rinses were combined, concentrated on a roto-evaporator (only acetonitrile remained), and placed in a separatory funnel. The extract was partitioned twice with hexane; the two hexane washes were combined and extracted with fresh acetonitrile. The acetonitrile used to extract the hexane was combined with the hexane-extracted acetonitrile. The hexane was concentrated by roto-evaporation, then diluted in methylene chloride.

The hexane extracts were analyzed for total radioactivity using LSC. The acetonitrile fraction was concentrated and again extracted twice with hexane; both the hexane and acetonitrile fractions were analyzed by LSC. The acetonitrile extract was concentrated and diluted with methylene chloride:acetonitrile; an aliquot was analyzed for radioactivity by LSC. An additional aliquot of the acetonitrile extract was filtered through a C18 Sep-Pak cartridge with acetonitrile; [^{14}C]residues remaining on the cartridge were eluted with hexane. The acetonitrile and hexane eluates were concentrated and aliquots were analyzed by LSC. The remaining acetonitrile eluate was concentrated, transferred to a Kuderna-Danish tube with methylene chloride, and evaporated under a stream of nitrogen. The extracts were analyzed by TLC on silica gel plated developed in chloroform:methanol:formic acid:water (80:15:4:2). Unlabeled reference standards were cochromatographed with the samples. Following development, radioactive areas were located and quantified using a TLC radioscanner. Unextractable [^{14}C]residues were analyzed for total radioactivity by LSC following combustion.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS

Atrazine

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