DP Barcode: D160299,160301,160304,160680,161379,161577

PC Code No.: 056502

Date Out: JUL 3 1 1991

To: Lois Rossi
Product Manager PM #74
Special Review and Reregistration Division (H7508C)

From: Emil Regelman, Supervisory Chemist
Chemistry Review Section #2
Environmental Fate & Ground Water Branch
Environmental Fate & Effects Division (H7507C)

Thru: Henry Jacoby, Chief
Environmental Fate & Ground Water Branch
Environmental Fate & Effects Division (H7507C)

Attached, please find the EFGWB review of...

Reg./File # : 056502

Chemical Name : Pentachloronitrobenzene

Type Product : Fungicide

Product Name :

Company Name : AMVAC Chemical Corp. and Uniroyal Chemical Co.

Purpose : Response to 1987 Registration Standard.

:

Action Code : 660 EFGWB #(s): 91-0333,-0339,-0340,-0375,-0416,-0425

Date Received : 2/25/91 Total Review Time: 18 days

Deferrals to:

__ EEB/EFED __ DEB/HED __ OREB/HED

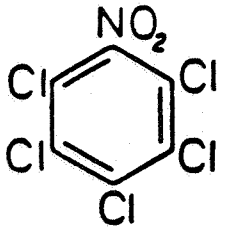
__ TB1/HED __ TB2/HED __ RSB/RD

1. CHEMICAL:

Common name: PCNB

Chemical name: Pentachloronitrobenzene

Structure:



Physical/Chemical properties:

Molecular formula: $C_6Cl_5NO_2$
Molecular weight: 295.3
Physical state: colorless needles
Density at 25°C: 1.718
Solubility at 25°C: 0.44 ppm in water, soluble in benzene and chloroform.
Vapor pressure at 25°C: 1.13×10^{-4} mmHg
Melting point: 141-145°C

2. TEST MATERIAL:

See attached reviews of individual studies.

3. STUDY/ACTION TYPE:

Addendum to the PCNB Registration Standard.

4. STUDY IDENTIFICATION:

Bowman, B. 1988. Determination of the photolysis rate of PCNB on the surface of soil. Laboratory project ID: ABC Final Report No. 36007. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by Uniroyal Chemical Co., Inc., Middlebury, CT. (41014201)

Cranor, W. 1990. Supplemental report to: aerobic soil metabolism of ^{14}C-pentachloronitrobenzene, MRID 41384501. Laboratory Project ID: ABC Final Supplemental Report No. 368241. Performed by Analytical Bio-Chemistry Laboratories, Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA. (41713202)

Cranor, W. 1990. Supplemental report to: amended final report to: anaerobic soil metabolism of ^{14}C-pentachloronitrobenzene, original final report MRID 41384301, amended final report MRID to be determined.

2

Laboratory Project ID: ABC Final Supplemental Report No. 36825. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA. (41713203)

Cranor, W., and C. Patterson. 1989. Anaerobic soil metabolism of ^{14}C-pentachloronitrobenzene. Laboratory Project ID: ABC Final Report No. 36825. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA. (41384301)

Cranor, W., and C. Patterson. 1990. Amended final report to: anaerobic soil metabolism of ^{14}C-pentachloronitrobenzene, MRID 41384301. Laboratory Project ID: ABC Amended Final Report No. 36825. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA. (41686001)

Dykes, J., and M. Carpenter. 1989. Determination of the photolysis rate of ^{14}C-PCNB in pH 5 aqueous solution. Laboratory Project ID: ABC Amended Final Report No. 36822. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA. (41384401)

Dykes, J. 1990. Soil adsorption/desorption with ^{14}C-PCNB. Laboratory Project ID: ABC Final Report No. 38405. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc. Columbia, MO., and submitted by AMVAC Chemical Corporation, Los Angeles, CA. (41648201)

Judy, D., B. Jacobson, and W. Guyton. 1990. Terrestrial field dissipation for PCNB in potatoes. Laboratory Project ID: ABC Final Report No. 36971. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and Agri-Growth Research, Inc., Hollandale, MN, and submitted by AMVAC Chemical Corporation, Los Angeles, CA. (41721401)

Kabler, K. 1990. Supplement to: Photodegradation Study of ^{14}C-PCNB on Soil Surface (MRID 41004801). Laboratory Project ID: ABC Final Report No. 368231. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA. (41713201)

Rice, F., B. Jacobson, and B. Guyton. 1990. Terrestrial field dissipation for PCNB in broccoli. Laboratory Project ID: ABC Final Report No. 36927. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and Plant Sciences, Inc., Watsonville, CA, and submitted by AMVAC Chemical Corporation, Los Angeles, CA. (41747001)

5. REVIEWED BY:

Dana Spatz
Chemist, ECRS #2
EFGWB/EFED/OPP

Date: JUL 22 1991

6. APPROVED BY:

Emil Regelman
Supervisory Chemist, ECRS #2
EFGWB/EFED/OPP

Date: JUL 31 1991

7. CONCLUSIONS:

PHOTODEGRADATION IN WATER (41384401), AMVAC Chemical Corporation

This study cannot be used to fulfill the Photodegradation in Water data requirement.

This study is unacceptable for the following reasons:

a. The mass balances were incomplete in the irradiated samples; recoveries were as low as 44.9-64.6% of the applied at 7 days posttreatment for the sensitized and non-sensitized samples, respectively. Volatilization was neither measured nor controlled; the ampules were sealed during the exposure period and opened for analysis with no provision to trap volatiles.

b. Degradates present at up to approximately 80% of the radioactivity in the irradiated solutions were not characterized. The study author reports that "Virtually all of the degradation was seen as one peak from the reverse phase HPLC... This peak did not correspond to any of the degradate standards supplied by the sponsor." It was reported that the degradate(s) will be identified in a supplemental study.

c. The stability of PCNB under refrigeration was not addressed in this study. Samples were stored under refrigeration for approximately 2 months before analysis.

[^{14}C]PCNB at a nominal concentration of 0.3 µg/mL photodegraded with registrant-calculated half-lives of 2.46 days in sensitized (approximately 1% acetone) and 2.50 days in nonsensitized sterile aqueous buffer solutions (pH 5) that were irradiated with an artificial light source (xenon lamp) at 23-26°C for 7 days. In the nonsensitized irradiated samples, [^{14}C]PCNB was 99.4% of the radioactivity remaining immediately posttreatment, 67.2% at day 2, 63.5% on day 3, and 19.9% on day 7 posttreatment. In the sensitized irradiated samples, [^{14}C]PCNB was 99.1% of the radioactivity remaining immediately posttreatment, 76.5% at day 2, 57.3% at day 3, and 34.5% at 7 days posttreatment. Uncharacterized

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radioactivity increased from 0.6-0.9% of the radioactivity immediately posttreatment to 65.5-80.1% of the radioactivity remaining at 7 days posttreatment. [^{14}C]PCNB was 97.5-100.6% of the radioactivity in the sensitized and nonsensitized dark control solutions.

The material balances were 44.9-100% in the irradiated solutions (sensitized and nonsensitized) and 93.2-112.3% in the dark control solutions (sensitized and nonsensitized).

PHOTODEGRADATION ON SOIL (41713201), AMVAC Chemical Corporation

This study involved additional voluntary identification work performed in order to try and identify two unknown photolysis products which accounted for 3.5% and 4% of the initial dose of the test compound at the terminal sample point in a photodegradation on soil study originally reviewed and accepted by EFGWB on July 12, 1989; MRID # 41004801. The conclusions from that study were that PCNB photodegrades on the surface of sandy loam soil exposed to filtered Xenon Arc light system with a half-life of 80 days. Photoproducts were not identified as no degradate accounted for more than 4% of the activity in the test samples.

In this current identification study, the test methods included a modified TLC system in conjunction with HPLC, with quantitation of the analytes by LSC of the ^{14}C-zones/ fractions.

The integrity of PCNB in the stored extracts was confirmed by HPLC. The HPLC analyses demonstrated the stability of PCNB upon extended storage at -20°C (>2 years). A degradate accounting for 3.6% of the recovered ^{14}C-activity was detected in the second exposed sample replicate, however, no degradates were noticed in the other samples. The average percent parent was 93.2% in the exposed study samples and 100.4% for the non-exposed study samples.

In conclusion, further attempts to identify two unknown degradates observed in the day 30 exposed sample extracts from the original study were made using a modified TLC system in conjunction with HPLC, however, there was not a sufficient level of either degradate in the samples (<4% initial dose) so the analyses were terminated.

PHOTODEGRADATION ON SOIL (41014201), Uniroyal Chemical Company

This study cannot be used to fulfill the Photodegradation on Soil data requirement.

This study is unacceptable for the following reasons:

a. The soils were dosed with 10.5 μg parent equivalents per gram of soil, however, after 30 days, only 6.84 μg/g were recovered. The photolysis half-life appears to be based upon loss due to poor recoveries rather than on photodegradation of PCNB.

b. The day 0 sampling showed that only 90.8% of the extracted material was parent PCNB. The reason for this loss was not apparent.

c. Reference was made to confirming the identity of the residues using GC and GC/MS, however, the data verifying the identifications were not provided.

d. The complete set of spectra used in the analysis of residues was not provided for review and validation.

e. The dark control portion of the study was repeated because the initial dark control samples were inadvertently exposed to light for approximately 25 hours. The procedure in the dark sample repeat was different from the original procedure used for the exposed samples and the first set of dark controls (soil was air-dried at room temperature rather than in an oven at 120°C for 3 days, and different types of sealed vials were used). The results from the two dark control trials differed significantly. The registrant-calculated half-life was 367 days in the samples which were irradiated 1 day and 99.7 days in the repeat. No explanation was given by the registrant for why the repeat had a shorter half-life than the original dark control, especially when the original was mistakenly irradiated for 1 day. A concern about the sterility of the soils arises.

f. The r^2 value for the first-order degradation in the exposed system was an unacceptable 0.82. No explanation for this poor result was provided.

g. Material balance was only 80.6% on day 30 for the dark control.

Uniformly ring-labeled [^{14}C]PCNB (radiochemical purity 98.3%) degraded with a half-life of 22.2 days on sandy loam soil irradiated with artificial light (xenon arc lamp) at 25 ±2°C for 30 days. The only degradate identified in the irradiated soil was pentachloroaniline (PCA). No degradates were identified in the dark controls.

AEROBIC SOIL METABOLISM (41713202), AMVAC Chemical Corporation

This document was submitted by the registrant to supplement "Aerobic Soil Metabolism of [^{14}C]Pentachloronitrobenzene" (MRID 41384501), an experiment previously reviewed by EFGWB in a report dated January 16, 1991. That study, while scientifically sound, did not fulfill the aerobic soil metabolism data requirement for the following reasons:

a. Residue identification was incomplete. Unidentified extractable ^{14}C-residues were observed at the TLC origin throughout the study. These residues increased from an average day-0 level of 0.16 ppm in soil to a maximum of 0.64 ppm at 122 days posttreatment.

b. The analytical methodology employed to separate and identify residues was insufficient. In this study, one-dimensional TLC with either one of two solvent systems was used to identify residues. Because TLC does not give confirmatory residue identification, HPLC was also employed, however, this only tentatively confirmed the presence of parent PCNB. HPLC did not give confirmatory identification of any degradates.

   Although GC-MS is preferred whenever feasible, HPLC and then 2-dimensional TLC may prove to be acceptable if the HPLC and TLC systems employ different mobile phases and the separations achieved are unequivocal.

c. The soil extracts were stored frozen prior to TLC and HPLC analyses. Examination of HPLC data in Appendix I revealed that the analyses were performed 5-9 months after the sampling date. Freezer storage stability data for PCNB and its degradates in soil extracts were not provided.

In order to provide the needed information samples from the original study were used. A soil extract that had been frozen for approximately 2 years was re-analyzed and a soil sample from the same sampling interval was extracted and the extract was analyzed. The data from the analyses of the frozen extract and soil were compared to the original data for the corresponding sampling interval.

The frozen soil sample was extracted by a method "similar to the procedure employed in the original study". The soil was extracted three times with methanol:1 N acetic acid (80:20). Aliquots of this soil extract and the original frozen extract were analyzed by TLC on silica gel plates developed in hexane:toluene:acetone (7:3:1, v:v:v). Reverse-phase HPLC had been used in the original study to confirm TLC results. There was poor correlation for quantification between the two methods (TLC and reverse-phase HPLC). To resolve this problem, in the supplemental document, the extracts were partitioned with hexane and analyzed by normal-phase HPLC on a silica gel column eluted with a hexane:chloroform gradient with UV (254 nm) detection. Fractions were collected and analyzed by LSC. The recovery of radioactivity by the HPLC procedure was 76.7-95.7%.

In the original analysis of the 30-day posttreatment soil extract, PCNB was 82.4% and 96.2% of the extracted radioactivity by TLC and HPLC, respectively, and the degradate

pentachloroaniline (PCA)

was 8.7% by TLC and not detected by HPLC. In the reanalysis of this extract after 2 years of freezer storage, parent PCNB was 90.6% and 91.2% of the extracted radioactivity by TLC and HPLC, respectively; PCA was 6.5% and 3.9% by TLC and HPLC, respectively, and an unidentified

degradate(s) was 4.9% as determined by HPLC (this degradate was not detected by TLC).

In the extract of the frozen soil sample, parent PCNB was 86.1% and 91.3% of the extracted radioactivity by TLC and HPLC, respectively; PCA was 8.6% and 4.7% by TLC and HPLC, respectively, and an unidentified degradate(s) was 4.0% as determined by HPLC. This degradate was not detected by TLC. In the supplemental document, uncharacterized residues remaining in the aqueous phase of both extracts after the hexane partitioning step were present at up to 9% of the extracted radioactivity.

This supplement does adequately address the deficiencies in the original study. These studies fulfill the aerobic soil metabolism data requirement.

Pentachloronitrobenzene (PCNB) dissipated with a half-life of 77 days from sandy loam soil that was incubated in the dark at approximately 25°C. The major degradate was pentachloroaniline (PCA), and the degradate pentachlorothioanisole (PCTA) was also isolated. Volatilized residues included PCNB, PCA, and carbon dioxide.

ANAEROBIC SOIL METABOLISM (41384301, 41686001, 41713203), AMVAC Chemical

This study cannot be used to fulfill the Anaerobic Soil Metabolism data requirement.

[^{14}C]PCNB degraded with a half-life of 9.0 days in sandy loam soil anaerobically incubated in the dark at 25.0-26.9°C following 30 days of aerobic incubation. The degradates identified were pentachloroaniline (PCA) and pentachlorothioanisole (PCTA).

This study is scientifically sound, but does not meet Subdivision N guidelines for the following reason:

> Degradates present in the soil extracts (found at origin of TLC plate) at up to 7% of total soluble residues, were not identified.

In order for this study to fulfill the anaerobic soil metabolism data requirement, the registrant must identify all degradates present at ≥0.01 ppm.

LEACHING-ADSORPTION/DESORPTION (41648201), AMVAC Chemical Corporation

This study is acceptable and partially fulfills the Leaching-Adsorption/Desorption data requirement by providing information on the mobility (batch equilibrium) of unaged PCNB in silt loam, clay, sandy loam, and sand soils.

[^{14}C]PCNB was slightly mobile to immobile in silt loam, clay, sandy loam, and sand soils. Freundlich K_{ads} values were 7.25 for the sand soil, 15.61 for the sandy loam soil, 18.25 for the silt loam soil, and 51.91 for the clay soil.

No additional data on the mobility of unaged PCNB in soil are required at this time. However, data are needed on the mobility of PCNB degradates in soil.

SOIL FIELD DISSIPATION (41721401), AMVAC Chemical Corporation

This study is acceptable and can be used towards satisfying the Soil Field Dissipation data requirement.

PCNB (2-EC, 2 lb/gallon EC), applied at 25 lb ai/A, dissipated very slowly with a registrant-calculated half-life of 324 days from loam soil (0-6" layer) in Minnesota that was treated in July 1988. The pesticide was incorporated (4- to 6-inch depth) immediately after application, and the plot was planted to potatoes. Total PCNB residues [PCNB, pentachloroaniline (PCA), and pentachlorothioanisole (PCTA)] dissipated with a half-life of 436 days. Two degradates were identified: pentachloroaniline (PCA), at a maximum of 1.1 ppm at 515 days, and pentachlorothioanisole (PCTA), at a maximum of 0.25 ppm at 125 and 295 days. PCNB and its degradates were not detected below the 12-inch soil depth.

This study may be used to support registration of PCNB applied at or below 25 lb ai/A; additional data are required for use sites at which the registered application rate is greater. For complete fulfillment of the terrestrial field dissipation data requirement, acceptable data are required at two field sites for both the EC and granular formulations, applied at the maximum use rate.

SOIL FIELD DISSIPATION (41747001), AMVAC Chemical Corporation

This study cannot be used to fulfill the Soil Field Dissipation data requirement.

This study is unacceptable for the following reason:

The reported application rate of 30 lbs ai/A was not confirmed by the soil residue measurements taken immediately posttreatment. The actual application rate calculated from soil analysis was approximately 45% of the nominal rate of 30 lb ai/A (which is equivalent to 15.0 ppm in the 0- to 6-inch soil depth). Filter paper discs, placed on the test soil before the application of PCNB, contained only 12-55% of the expected concentration (average 29 ± 13%), and the time 0 soil sample contained only 6.8 ppm of PCNB residues. The authors suggest that the volatility of PCNB may explain this loss. This explanation may account for some of the loss, however, a better explanation may be related to the fact that

the concentration of PCNB in the spray solution was less than expected since it appears from the data that the test spray solution contained only 47% of the theoretical concentration of PCNB.

It should also be noted that the product label recommends soil incorporation to a depth of 4-6 inches immediately after application for effective control. No mention of soil incorporation was made in the report.

PCNB (75% WP), applied at 30 lb ai/A, dissipated very slowly with a registrant-calculated half-life of 305 days from sandy loam soil in California that was treated in September 1988. The plot was planted the next day to broccoli. Total PCNB residues [PCNB, pentachloroaniline (PCA), and pentachlorothioanisole (PCTA)] dissipated with a half-life of 401 days. In the 0- to 6-inch soil depth, parent PCNB averaged 6.7 ppm immediately after treatment. Two degradates were identified: pentachloroaniline (PCA), at a maximum of 0.39 ppm at 556 days, and pentachlorothioanisole (PCTA), at a maximum of 0.35 ppm at 275 days. PCNB and its degradates were not detected below the 12-inch soil depth.

ENVIRONMENTAL FATE ASSESSMENT

Studies have shown that PCNB does not hydrolyze under sterile aqueous conditions at pH 5, 7 or 9 at 25°C, and it photodegrades very slowly ($t_{1/2}$: ≈80 days) on the surface of soil exposed to a filtered Xenon Arc light system. PCNB degraded with a half-life of 77 days from sandy loam soil that was incubated in the dark at approximately 25°C. The major degradate was pentachloroaniline (PCA), and the degradate pentachlorothioanisole (PCTA) was also isolated. PCNB degraded with a half-life of 9.0 days in sandy loam soil anaerobically incubated in the dark at 25.0-26.9°C following 30 days of aerobic incubation. The degradates identified were PCA and PCTA. PCNB is slightly mobile to immobile in soils and is not expected to leach to ground water. Freundlich k_{ads} values were 7.25 for a sand soil, 15.61 for a sandy loam soil, 18.25 for a silt loam, and 51.91 for a clay soil. The mobility of its degradates PCA, PCTA, PCB, and the contaminant HCB, has not been adequately defined. PCNB demonstrated significant volatilization from soil; at 7 days posttreatment, approximately 80% of the applied had volatilized from a sandy loam incubated at 25°C. The vapor pressure of PCNB is 1.13×10^{-4} mm Hg at 25°C.

Several soil field dissipation studies have been conducted, however, only one has been found to be acceptable. In the acceptable study, PCNB (2EC) applied at 25 lb ai/A and incorporated to a 4-6" depth, dissipated very slowly with a calculated half-life of 324 days from loam soil in Minnesota that was planted to potatoes. This study, as well as the other unacceptable soil field dissipation studies do indicate that PCNB tends to remain in the 0-6 inch layer and that it is persistent. Half-lives in the 0-6 inch layer were: 305 days in a sandy loam broccoli plot in California; 128 days in a loamy sand soil in California planted with broccoli; 193 days in a sandy loam potato plot in Minnesota; 116 days in a sandy loam peanut plot in Georgia; and 35 days in a sandy loam turf plot in California. PCNB degradates were also persistent in these studies. These residues were detected sporadically at depths below the 0-6 inch layer at low concentrations, however, it was not clear from the data whether there was actual leaching through the soil profile or if there was a contamination problem during sampling.

Although the study was not acceptable, the data from a confined accumulation in rotational crops study did demonstrate that leafy vegetables, root crops, and small grains accumulate significant amounts of PCNB residues (yet to be identified) even after being planted 12 months posttreatment. Some examples of the level of residues found in crops planted 12 months after application of PCNB are: 0.61 ppm in mature lettuce, 1.5 ppm in turnip roots, 25.9 ppm in wheat straw, 8.0 ppm in wheat hulls, and 0.38 ppm in wheat grain.

PCNB also accumulates in fish. An accumulation in fish study demonstrated that PCNB residues (yet to be defined) bioconcentrate in the tissues of bluegill sunfish. BCF's were 370x, 1800x, and 960x for edible, viscera, and whole fish, respectively. However, after the 21-day

depuration period, 81-84% of the accumulated residues were eliminated from the fish tissues.

In summary, PCNB is expected to be persistent in the environment. The degradates PCA, PCTA, PCB and the contaminant HCB are also persistent. PCNB is not likely to leach to ground water, however, the potential for its degradates to leach has not been clearly defined. PCNB is moderately volatile and may dissipate through volatilization unless measures such as soil incorporation are taken. There is a tendency for residues of PCNB to accumulate in crops rotated several months after application and also to bioaccumulate in fish.

8. RECOMMENDATIONS:

The studies that must be conducted to fulfill the outstanding data requirements are summarized below. Because there are two registrants generating data independently in response to the Registration Standard, a separate list of data requirements has been generated for each company.

UNIROYAL CHEMICAL CO. SATISFIED

Hydrolysis 161-1 (40972601)
Leaching-Adsorption/Desorption 163-1 (00114168, 00114181)

NOT SATISFIED

Photodegradation in Water 161-2
Photodegradation in Soil 161-3
Aerobic Soil Metabolism 162-1
Anaerobic Soil Metabolism 162-2
Laboratory Volatility 163-2
Field Volatility 163-3
Soil Field Dissipation 164-1
Confined Accumulation in Rotational Crops 165-1
Accumulation in Fish 165-4

RESERVED

Long-Term Soil Field Dissipation 164-5
Field Accumulation in Rotational Crops 165-2

AMVAC CHEMICAL CORP. SATISFIED

Hydrolysis 161-1 (40865301)
Photodegradation on Soil 161-3 (41004801, 41713201)
Aerobic Soil Metabolism 162-1 (41384501, 41713202)
Unaged Leaching-Adsorption/Desorption 163-1 (41648201)
Laboratory Volatility 163-2 (41178001)

NOT SATISFIED

Photodegradation in Water 161-2
Anaerobic Soil Metabolism 162-2
Aged Leaching-Adsorption/Desorption 163-1
Field Volatility 163-3
Soil Field Dissipation 164-1
Confined Accumulation in Rotational Crops 165-1
Accumulation in Fish 165-4

RESERVED

Long-Term Soil Field Dissipation 164-5
Field Accumulation in Rotational Crops 165-2

9. BACKGROUND:

PCNB (pentachloronitrobenzene) is a fungicide registered for use on a variety of terrestrial food crop (field and vegetable) and terrestrial nonfood (ornamentals and turf) use sites. Single active ingredient formulations consist of 10-40% D, 75% WP, 2-30% G, 23.4-26.5% EC, 20% FlC, and 20-25% RTU-L. PCNB may be formulated with several compounds including fenaminosulf, thiram, captan, truban, carbaryl, disulfoton, phorate, cycloheximide, malathion, and carboxin. PCNB is generally used as a seed treatment, a spray using ground equipment, or a granular soil application.

10. DISCUSSION OF INDIVIDUAL TESTS OR STUDIES:

See attached DER's.

11. COMPLETION OF ONE-LINER:

Amended as appropriate.

12. CBI APPENDIX:

Not applicable.

Environmental Fate & Effects Division
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY
PENTACHLORONITROBENZENE
Last Update on July 22, 1991
[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

LOGOUT	Reviewer:	Section Head:	Date: JUL 3 1 1991

Common Name:PENTACHLORONITROBENZENE
Smiles Code:Cl-c(c(Cl)c(Cl)c1N(=O)=O)c(Cl)c1Cl
PC Code # : 56502 CAS #:82-68-8 Caswell #:

Chem. Name :PENTACHLORONITROBENZENE

Action Type:Fungicide

Trade Names:QUINTOZENE; PCNB; BRASSICOL; TERRACLOR
(Formul'tn):WP; EC; GRANULES; RTU = 24% SOLUTION
Physical State: colorless needles

Use :SOIL FUNGICIDE/SEED DRESSING
Patterns :Cotton, peanuts, beans, cabbage, broccoli, turf, ornamentals
(% Usage) :
:

Empirical Form: $C_6Cl_5NO_2$
Molecular Wgt.: 295.34 Vapor Pressure: 1.13E -4 Torr
Melting Point : °C Boiling Point: °C
Log Kow : 5.45 pKa: @ °C
Henry's : 1.00E -4 Atm. M3/Mol (Measured) 9.98E -5 (calc'd)

Solubility in ...				Comments
Water	0.44E	ppm	@20.0 °C	
Acetone	E	ppm	@ °C	
Acetonitrile	E	ppm	@ °C	
Benzene	E	ppm	@ °C	
Chloroform	E	ppm	@ °C	
Ethanol	E	ppm	@ °C	
Methanol	E	ppm	@ °C	
Toluene	E	ppm	@ °C	
Xylene	E	ppm	@ °C	
	E	ppm	@ °C	
	E	ppm	@ °C	

Hydrolysis (161-1)
[V] pH 5.0:STABLE
[V] pH 7.0:STABLE
[V] pH 9.0:STABLE
[ ] pH :
[ ] pH :
[ ] pH :

Environmental Fate & Effects Division
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY
PENTACHLORONITROBENZENE
Last Update on July 22, 1991
[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

Photolysis (161-2, -3, -4)
[ ] Air :
[V] Soil :80 DAYS ON SdLm; Xe ARC
[S] Water:4 DAYS; Xe ARC
[ ] :
[ ] :
[ ] :

Aerobic Soil Metabolism (162-1)
[S] 4.7 MONTHS IN SANDY LOAM
[S] 7.6 " " CLAY
[S] 9.7 " " MUCK
[S] 4 MONTHS IN SANDY CLAY LOAM
[V] HALF-LIFE OF 77 DAYS IN SANDY LOAM; PCA MAJOR DEGRADATE, PCTA
[ ] WAS ALSO ISOLATED. VOLATILES: PCNB, PCA, CO2
[ ]

Anaerobic Soil Metabolism (162-2)
[S] HALF-LIFE OF 9 DAYS IN SANDY LOAM; PCA AND PCTA IDENTIFIED
[ ]
[ ]
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Anaerobic Aquatic Metabolism (162-3)
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Aerobic Aquatic Metabolism (162-4)
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Environmental Fate & Effects Division
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY
PENTACHLORONITROBENZENE
Last Update on July 22, 1991
[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

Soil Partition Coefficient (Kd) (163-1)
[V] UNAGED: 7.2 SAND, 15.6 SANDY LOAM, 18.2 SILT LOAM, 51.9 CLAY
[ ]
[ ]
[ ]
[ ]
[ ]

Soil Rf Factors (163-1)
[S] 0.00 IN HAGERSTOWN SiClLm
[S] RELATIVELY IMMOBILE IN SiLm,
[ ] SAND, AND SdLm
[ ]
[ ]
[ ]

Laboratory Volatility (163-2)
[V] 0.062 to 0.171 µg/cm2/hr (4.99 x10-4 to 1.30 x 10-3 g/m3)
[ ] from sandy loam soil

Field Volatility (163-3)
[ ]
[ ]

Terrestrial Field Dissipation (164-1)
[S] IN 22 POTATO FIELDS IN DENMARK, T1/2 AVGD. 434 DAYS WITH
[ ] RANGE OF 117 TO 1059 DAYS.
[ ]
[V] 2-EC APPLIED AT 25 LB AI/A DISSIPATED VERY SLOWLY WITH A HALF-
[ ] LIFE OF 324 DAYS FROM LOAM SOIL IN MINNESOTA (POTATOES). PCNB WAS
[ ] INCORPORATED IMMEDIATELY AFTER APPLICATION. TWO DEGRADATES WERE
[ ] IDENTIFIED; PCA (1.1 PPM AT 515 DAYS) AND PCTA (0.25 PPM AT 125
[ ] AND 295 DAYS). PCNB AND ITS DEGRADATES NOT DETECTED BELOW THE
[ ] 12-INCH SOIL DEPTH.
[ ]

Aquatic Dissipation (164-2)
[ ]
[ ]
[ ]
[ ]
[ ]
[ ]

Forestry Dissipation (164-3)
[ ]
[ ]

Environmental Fate & Effects Division
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY
PENTACHLORONITROBENZENE
Last Update on July 22, 1991
[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

Long-Term Soil Dissipation (164-5)
[ ]
[ ]

Accumulation in Rotational Crops, Confined (165-1)
[ ]
[ ]

Accumulation in Rotational Crops, Field (165-2)
[ ]
[ ]

Accumulation in Irrigated Crops (165-3)
[ ]
[ ]

Bioaccumulation in Fish (165-4)
[S] BLUEGILL SUNFISH BCF: 1043 X FOR WHOLE FISH. DEPURATION
[ ] WAS FAIRLY RAPID.

Bioaccumulation in Non-Target Organisms (165-5)
[S] Channel Catfish: 250x (edible), 630x (viscera), 460x (whole)
[ ] Bluegill: 120x (edible), 560x (viscera), 330x (whole)

Ground Water Monitoring, Prospective (166-1)
[ ]
[ ]
[ ]
[ ]

Ground Water Monitoring, Small Scale Retrospective (166-2)
[ ]
[ ]
[ ]
[ ]

Ground Water Monitoring, Large Scale Retrospective (166-3)
[ ]
[ ]
[ ]
[ ]

Ground Water Monitoring, Miscellaneous Data (158.75)
[ ]
[ ]
[ ]

Environmental Fate & Effects Division
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY
PENTACHLORONITROBENZENE
Last Update on July 22, 1991
[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

Field Runoff (167-1)
[ ]
[ ]
[ ]
[ ]

Surface Water Monitoring (167-2)
[ ]
[ ]
[ ]
[ ]

Spray Drift, Droplet Spectrum (201-1)
[ ]
[ ]
[ ]
[ ]

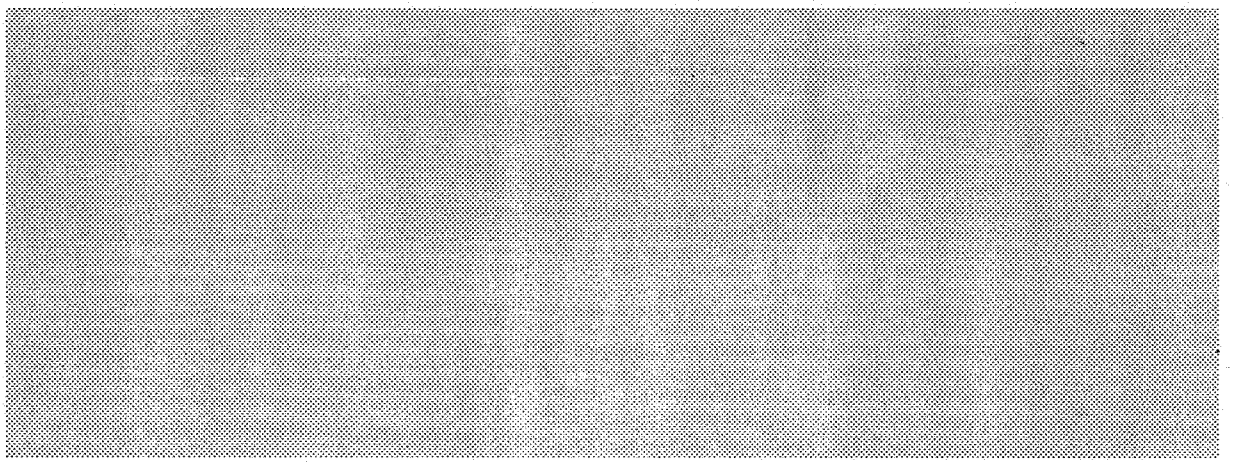
Spray Drift, Field Evaluation (202-1)
[ ]
[ ]
[ ]
[ ]

Degradation Products

Pentachloroaniline (PCA)
Pentachlorothioanisole (PCTA)
Pentachlorobenzene (PCB)
Hexachlorobenzene (HCB) IS AN IMPURITY

Environmental Fate & Effects Division
PESTICIDE ENVIRONMENTAL FATE ONE LINE SUMMARY
PENTACHLORONITROBENZENE
Last Update on July 22, 1991
[V] = Validated Study [S] = Supplemental Study [U] = USDA Data

Comments



References: ODW HA,
Writer : RWH, DSS

PCNB

Table of Contents

DATA EVALUATION RECORD

STUDY 1

CHEM 056502 PCNB §161-2

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41384401

Dykes, J and M. Carpenter. 1989. Determination of the photolysis rate of ^{14}C-PCNB in pH 5 aqueous solution. Laboratory Project ID: ABC Amended Final Report No. 36822. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA.

REVIEWED BY: N. Shishkoff TITLE: Staff Scientist

EDITED BY: W. Martin TITLE: Staff Scientist
L. Mickley Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

SECONDARY REVIEW BY: Dana Spatz
TITLE: Chemist
ORG: EFGWB/EFED/OPP

SIGNATURE:

JUL 2 2 1991

CONCLUSIONS:

Degradation - Photodegradation in Water

1. This study cannot be used to fulfill the Photodegradation in Water data requirement.

2. The data are considered to be of uncertain value and should not be used to predict the photodegradation of PCNB in aqueous solution.

3. This study is unacceptable for the following reasons:

   a. The mass balances were incomplete in the irradiated samples; recoveries were as low as 44.9-64.6% of the applied at 7 days posttreatment for the sensitized and non-sensitized samples, respectively. Volatilization was neither measured nor controlled; the ampules were sealed during the exposure period and opened for analysis with no provision to trap volatiles.

b. Degradates present at up to approximately 80% of the radioactivity in the irradiated solutions were not characterized. The study author reports that "Virtually all of the degradation was seen as one peak from the reverse phase HPLC... This peak did not correspond to any of the degradate standards supplied by the sponsor." It was reported that the degradate(s) will be identified in a supplemental study.

c. The stability of PCNB under refrigeration was not addressed in this study. Samples were stored under refrigeration for approximately 2 months before analysis.

4. Since the material balances were incomplete and volatilization was neither controlled nor measured, the problems with this study cannot be resolved with the submission of additional data. Therefore, a new study must be conducted.

METHODOLOGY:

Uniformly ring-labeled [^{14}C]PCNB (radiochemical purity 97.6%, specific activity 46.4 mCi/mmole, Amvac) dissolved in acetonitrile was added, with sonication, at a nominal concentration of 0.3 μg/ml to sterile (autoclaved) pH 5.01 sodium acetate buffer solutions; the cosolvent concentration was 0.90%. Acetone was added at 0.99% by volume to one solution as a photosensitizer. Sensitized and nonsensitized test solutions were added to sterile glass ampules and heat sealed. Half of the ampules in each treatment were exposed to a xenon arc lamp at 23-26°C. The intensity of the artificial light source was equal to one-half that of sunlight. The remainder of the vials for each treatment were wrapped in aluminum foil and placed in a closed box in the photolysis chamber to serve as dark controls. One ampule of each irradiated or dark control solution was removed at 0, 1, 2, 3, 4, 5, 6, and 7 days posttreatment. At each sampling interval, the test solutions in the ampules were transferred to sealable culture tubes and stored under refrigeration for an unspecified period.

Duplicate aliquots of each sample were analyzed for total radioactivity by LSC. Additional aliquots were analyzed by HPLC (polygosil C_{18} column) with an acetonitrile:water gradient and radioactivity detection. Analytical standards of PCNB, pentachloroaniline, 2,3,4,5-tetrachloronitrobenzene, pentachlorothioanisole, hexachlorobenzene, pentachlorobenzene, and 2,3,5,6-tetrachloronitrobenzene were chromatographed as described above.

DATA SUMMARY:

[^{14}C]PCNB (radiochemical purity 97.6%) at a nominal concentration of 0.3 µg/mL photodegraded with registrant-calculated half-lives of 2.46 days in sensitized (approximately 1% acetone) and 2.50 days in nonsensitized sterile aqueous buffer solutions (pH 5) that were irradiated with an artificial light source (xenon lamp) at 23-26°C for 7 days. In the nonsensitized irradiated samples, [^{14}C]PCNB was 99.4% of the radioactivity remaining immediately posttreatment, 67.2% at day 2, 63.5% on day 3, and 19.9% on day 7 posttreatment. In the sensitized irradiated samples, [^{14}C]PCNB was 99.1% of the radioactivity remaining immediately posttreatment, 76.5% at day 2, 57.3% at day 3, and 34.5% at 7 days posttreatment (Table II). Uncharacterized radioactivity increased from 0.6-0.9% of the radioactivity immediately posttreatment to 65.5-80.1% of the radioactivity remaining at 7 days posttreatment. [^{14}C]PCNB was 97.5-100.6% of the radioactivity in the sensitized and nonsensitized dark control solutions.

The material balances were 44.9-100% in the irradiated solutions (sensitized and nonsensitized) and 93.2-112.3% in the dark control solutions (sensitized and nonsensitized).

COMMENTS:

1. The registrant states that the concentration of PCNB in the test solutions was 0.3 µg/mL. However, based on the Day 0 liquid scintillation analysis (Table I), the actual values are approximately 0.12 µg/mL in the sensitized solution and 0.140 µg/mL in the nonsensitized solution.

2. Since the xenon light source was reported as one-half the intensity of the sun, "24 hrs of continuous exposure is equal to approximately 12 hours of sunlight".

3. Additional studies were performed in an attempt to improve the mass balances by minimizing volatilization. In the first study, completely filled screw cap culture tubes were used; in the second study, foil lined caps with teflon liners were used. The mass balances were low in both studies and the data were not reported.

PCNB environmental fate review

Page _____ is not included in this copy.

Pages 24 through 27 are not included in this copy.

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 product 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

_x_ 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 2

CHEM 056502 PCNB §161-3

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41014201

Bowman, B. 1988. Determination of the photolysis rate of PCNB on the surface of soil. Laboratory project ID: ABC Final Report No. 36007. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by Uniroyal Chemical Co., Inc., Middlebury, CT.

REVIEWED BY: N. Shishkoff TITLE: Staff Scientist

EDITED BY: W. Martin TITLE: Staff Scientist
L. Parsons Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

SECONDARY REVIEW BY: Dana Spatz
TITLE: Chemist
ORG: EFGWB/EFED/OPP

SIGNATURE: JUL 2 2 1991

CONCLUSIONS:

Degradation - Photodegradation on Soil

1. This study cannot be used to fulfill the Photodegradation on Soil data requirement.

2. This study is unacceptable for the following reasons:

   a. The soils were dosed with 10.5 µg parent equivalents per gram of soil, however, after 30 days, only 6.84 µg/g were recovered. The photolysis half-life appears to be based upon loss due to poor recoveries rather than on photodegradation of PCNB.

   b. The day 0 sampling showed that only 90.8% of the extracted material was parent PCNB. The reason for this loss was not apparent.

c. Reference was made to confirming the identity of the residues using GC and GC/MS, however, the data verifying the identifications were not provided.

d. The complete set of spectra used in the analysis of residues was not provided for review and validation.

e. The dark control portion of the study was repeated because the initial dark control samples were inadvertently exposed to light for approximately 25 hours. The procedure in the dark sample repeat was different from the original procedure used for the exposed samples and the first set of dark controls (soil was air-dried at room temperature rather than in an oven at 120°C for 3 days, and different types of sealed vials were used). The results from the two dark control trials differed significantly. The registrant-calculated half-life was 367 days in the samples which were irradiated 1 day and 99.7 days in the repeat. No explanation was given by the registrant for why the repeat had a shorter half-life than the original dark control, especially when the original was mistakenly irradiated for 1 day. A concern about the sterility of the soils arises.

f. The r^2 value for the first-order degradation in the exposed system was an unacceptable 0.82. No explanation for this poor result was provided.

g. Material balance was only 80.6% on day 30 for the dark control.

2. Uniformly ring-labeled [^{14}C]PCNB (radiochemical purity 98.3%) degraded with a half-life of 22.2 days on sandy loam soil irradiated with artificial light (xenon arc lamp) at 25 ±2°C for 30 days. The only degradate identified in the irradiated soil was pentachloroaniline (PCA). No degradates were identified in the dark controls.

METHODOLOGY:

Sandy loam soil (64% sand, 20% silt, 16% clay, 1.6% organic matter, pH 6.2, 9.5 Meq/100 g CEC) was placed in borosilicate glass vials to a depth of approximately 1 mm, wetted with water, and dried in an oven at 120°C for 3 days. The vials were cooled in an acetone/dry ice bath and treated with uniformly ring-labeled [^{14}C]PCNB (radiochemical purity 98.3%, specific activity 8.9 mCi/mmole, Uniroyal Chemical Co.), dissolved in acetonitrile, at 10.5 ppm. The vials were flame-sealed; two vials were used as time zero samples, and the remainder were placed 32 inches from a xenon lamp (Atlas, 6500 W) and maintained at 25 ±2°C. The intensity of the artificial light source was approximately half that of sunlight. The initial set of dark control vials were inadvertently exposed to the xenon light source for 1 day. This set of dark control vials was

subsequently placed in the dark (not further described). Duplicate vials were removed at 0, 1.05, 3.13, 7.10, 14.0, and 30.1 days posttreatment.

At each sampling interval, the vials were cooled in a dry ice/acetone bath in order to reduce volatilization, then the vials were opened and the soil was flooded with 4 mL of acetone. The soil:acetone slurry was transferred to 15 mL culture tubes and extracted twice more with acetone. Duplicate aliquots of the pooled acetone extracts were analyzed by LSC. Additional aliquots were analyzed by TLC on silica gel plates developed in hexane:toluene:acetone (7:3:1). Unlabeled reference standards of PCNB, 2,3,4,5-tetrachloronitrobenzene, pentachloroaniline, pentachlorothioanisole, 2,3,5,6-tetrachloronitrobenzene, hexachlorobenzene, and pentachlorobenzene, as well as an aliquot of the stock solution, were cochromatographed with the extracts. Unlabeled standards were visualized by UV light, radioactive areas were located and quantified by radioscanning. Additional soil samples were analyzed by LSC following combustion; the combustion efficiency was 90.3-93.6%.

Additional dark controls were prepared by placing soil in square glass containers, moistening the soil with deionized water, air-drying at room temperature and then treating the soil with uniformly ring-labeled [^{14}C]PCNB dissolved in acetonitrile at 10.5 ppm. The containers were flame-sealed, wrapped in aluminum foil, and placed in a box in a stainless steel water-jacketed photolysis chamber maintained at 25 ±1°C. Containers were removed at 0, 1, 3, 7, 15, and 30 days and analyzed as previously described.

DATA SUMMARY:

Uniformly ring-labeled [^{14}C]PCNB (radiochemical purity 98.3%) at 10.5 ppm degraded with a registrant-calculated half-life of 22.2 days in sandy loam soil irradiated with a artificial light source (xenon lamp) for 30 days at 25 ±2°C. The PCNB concentration was 9.26 µg/g immediately posttreatment, 5.64 µg/g at 7.1 days, 4.04 µg/g at 14 days and 3.69 µg/g at 30.1 days posttreatment. The only degradate identified was

- pentachloroaniline (PCA)

which was a maximum of 34.7% of the extracted radioactivity activity at 30 days. At 30.1 days posttreatment PCNB was 54% of the extracted radioactivity and 4.4% of the radioactivity remained at the origin.

In the first set of dark controls (exposed to light for one day), [^{14}C]PCNB degraded with a registrant-calculated half-life of 367 days. In the second set of dark controls (maintained in the dark for 30 days), [^{14}C]PCNB degraded with a registrant-calculated half-life of 99.7 days. The [^{14}C]PCNB concentrations were 8.22-9.26 µg/g in the first set of dark controls and 6.79-8.48 µg/g in the second set

of dark controls. No degradates were identified in the dark controls.

The material balances were 87.1%-104% for the irradiated samples, 94.9-102% for samples irradiated 1 day, and 80.6-106% for rerun dark samples.

COMMENTS:

1. Samples were tested for storage stability by placing dosed soil in the refrigerator or freezer for one day. The study author stated that "if storage was necessary, the samples should be stored in a freezer". No data were presented for review.

2. Preliminary investigations were performed to determine approximate degradation rate, sampling intervals, and evaluation of proposed methodology. The material balances were poor in these experiments, probably due to volatilization of PCNB.

3. It was difficult to determine which methods from the preliminary experiments were carried over to the definitive experiment. The reviewer assumed that the methodology was the same.

PCNB environmental fate review

Page _____ is not included in this copy.

Pages 32 through 38 are not included in this copy.

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 product 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

_X_ FIFRA registration data

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DATA EVALUATION RECORD

STUDY 3

CHEM 056502 PCNB §162-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41713202

Cranor, W. 1990. Supplemental report to: aerobic soil metabolism of ^{14}C-pentachloronitrobenzene, MRID 41384501. Laboratory Project ID: ABC Final Supplemental Report No. 368241. Performed by Analytical Bio-Chemistry Laboratories, Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA.

REVIEWED BY: N. Shishkoff TITLE: Staff Scientist

EDITED BY: W. Martin TITLE: Staff Scientist
L. Parsons Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

SECONDARY REVIEW BY: Dana Spatz
TITLE: Chemist
ORG: EFGWB/EFED/OPP

SIGNATURE: JUL 2 2 1991

CONCLUSIONS:

Metabolism - Aerobic Soil

This document was submitted by the registrant to supplement "Aerobic Soil Metabolism of [^{14}C]Pentachloronitrobenzene" (MRID 41384501), an experiment previously reviewed by EFGWB in a report dated January 16, 1991. That study, while scientifically sound, did not fulfill the aerobic soil metabolism data requirement for the following reasons:

a. Residue identification was incomplete. Unidentified extractable ^{14}C-residues were observed at the TLC origin throughout the study. These residues increased from an average day-0 level of 0.16 ppm in soil to a maximum of 0.64 ppm at 122 days posttreatment.

b. The analytical methodology employed to separate and identify residues was insufficient. In this study, one-dimensional TLC with either one of two solvent systems was used to identify residues. Because TLC does not give confirmatory residue identification, HPLC was also employed, however, this only tentatively confirmed the presence of parent PCNB. HPLC did not give confirmatory identification of any degradates.

   Although GC-MS is preferred whenever feasible, HPLC and then 2-dimensional TLC may prove to be acceptable if the HPLC and TLC systems employ different mobile phases and the separations achieved are unequivocal.

c. The soil extracts were stored frozen prior to TLC and HPLC analyses. Examination of HPLC data in Appendix I revealed that the analyses were performed 5-9 months after the sampling date. Freezer storage stability data for PCNB and its degradates in soil extracts were not provided.

In order to provide the needed information samples from the original study were used. A soil extract that had been frozen for approximately 2 years was re-analyzed and a soil sample from the same sampling interval was extracted and the extract was analyzed. The data from the analyses of the frozen extract and soil were compared to the original data for the corresponding sampling interval.

The frozen soil sample was extracted by a method "similar to the procedure employed in the original study". The soil was extracted three times with methanol:1 N acetic acid (80:20). Aliquots of this soil extract and the original frozen extract were analyzed by TLC on silica gel plates developed in hexane:toluene:acetone (7:3:1, v:v:v). Reverse-phase HPLC had been used in the original study to confirm TLC results. There was poor correlation for quantification between the two methods (TLC and reverse-phase HPLC). To resolve this problem, in the supplemental document, the extracts were partitioned with hexane and analyzed by normal-phase HPLC on a silica gel column eluted with a hexane:chloroform gradient with UV (254 nm) detection. Fractions were collected and analyzed by LSC. The recovery of radioactivity by the HPLC procedure was 76.7-95.7% (Table IV).

In the original analysis of the 30-day posttreatment soil extract, PCNB was 82.4% and 96.2% of the extracted radioactivity by TLC and HPLC, respectively, and the degradate

pentachloroaniline (PCA)

was 8.7% by TLC and not detected by HPLC. In the reanalysis of this extract after 2 years of freezer storage, parent PCNB was 90.6% and 91.2% of the extracted radioactivity by TLC and HPLC, respectively; PCA was 6.5% and 3.9% by TLC and HPLC, respectively, and an unidentified degradate(s) was 4.9% as determined by HPLC (this degradate was not detected by TLC; Table II).

In the extract of the frozen soil sample, parent PCNB was 86.1% and 91.3% of the extracted radioactivity by TLC and HPLC, respectively; PCA was 8.6% and 4.7% by TLC and HPLC, respectively, and an unidentified degradate(s) was 4.0% as determined by HPLC. This degradate was not detected by TLC (Table II). In the supplemental document, uncharacterized residues remaining in the aqueous phase of both extracts after the hexane partitioning step were present at up to 9% of the extracted radioactivity.

This supplement does adequately address the deficiencies in the original study. These studies fulfill the aerobic soil metabolism data requirement.

Pentachloronitrobenzene (PCNB) dissipated with a half-life of 77 days from sandy loam soil that was incubated in the dark at approximately 25°C. The major degradate was pentachloroaniline (PCA), and the degradate pentachlorothioanisole (PCTA) was also isolated. Volatilized residues included PCNB, PCA, and carbon dioxide.

PCNB environmental fate review

Page _____ is not included in this copy.

Pages 42 through 43 are not included in this copy.

The material not included contains the following type of information:

___ Identity of product inert ingredients

___ Identity of product impurities

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DATA EVALUATION RECORD

STUDY 4

CHEM 056502 PCNB §162-2

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41384301
Cranor, W., and C. Patterson. 1989. Anaerobic soil metabolism of ^{14}C-pentachloronitrobenzene. Laboratory Project ID: ABC Final Report No. 36825. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA.

STUDY ID 41686001
Cranor, W., and C. Patterson. 1990. Amended final report to: anaerobic soil metabolism of ^{14}C-pentachloronitrobenzene, MRID 41384301. Laboratory Project ID: ABC Amended Final Report No. 36825. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA.

STUDY ID 41713203
Cranor, W. 1990. Supplemental report to: amended final report to: anaerobic soil metabolism of ^{14}C-pentachloronitrobenzene, original final report MRID 41384301, amended final report MRID to be determined. Laboratory Project ID: ABC Final Supplemental Report No. 368251. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and submitted by AMVAC Chemical Corporation, Los Angeles, CA.

DIRECT REVIEW TIME = 20

REVIEWED BY: N. Shishkoff TITLE: Staff Scientist

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L. Parsons Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

SECONDARY REVIEW BY: Dana Spatz
TITLE: Chemist
ORG: EFGWB/EFED/OPP

SIGNATURE: JUL 22 1991

CONCLUSIONS:

Metabolism - Anaerobic soil

1. This study cannot be used to fulfill the Anaerobic Soil Metabolism data requirement.

2. [^{14}C]PCNB degraded with a half-life of 9.0 days in sandy loam soil anaerobically incubated in the dark at 25.0-26.9°C following 30 days of aerobic incubation. The degradates identified were pentachloroaniline (PCA) and pentachlorothioanisole (PCTA).

3. This study is scientifically sound, but does not meet Subdivision N guidelines for the following reason:

   Degradates present in the soil extracts (found at origin of TLC plate) at up to 7% of total soluble residues, were not identified.

4. In order for this study to fulfill the anaerobic soil metabolism data requirement, the registrant must identify all degradates present at ≥0.01 ppm.

METHODOLOGY:

[^{14}C]PCNB (radiochemical purity 99%, specific activity 46.4 mCi/mmol, Chemsyn) dissolved in hexane was added at 10.47 ppm to sandy loam soil (54% sand, 36% silt, 10% clay, 0.8% organic matter, CEC 4.7 meq/100 g, pH 6.5) that had been air-dried and sieved (2-mm mesh). The hexane was evaporated under nitrogen, and the soil was mixed to obtain a uniform distribution of PCNB. Portions of soil (10 g dry weight) were placed in tared tubes and adjusted to approximately 75% of field capacity with deionized water. Deionized water was added as needed at weekly intervals to maintain this moisture level. The tubes were placed in a metabolism vessel in an environmental chamber and incubated at 25.0-26.9°C for 30 days. Humidified, carbon dioxide-free air was drawn through the vessel and exhausted through ethylene glycol, H_2SO_4, and KOH volatile trapping solutions. Single tubes were removed for analysis at 0, 1, 3, 7, 14, and 30 days posttreatment.

After 30 days of aerobic incubation, the remaining samples were flooded with 20 mL of deionized water and 0.1 g of glucose was added to facilitate microbial activity. The air flow was discontinued and a flow of nitrogen was established to insure anaerobic conditions. Additional tubes were removed for analysis at 60 and 90 days posttreatment (30 and 60 days after flooding).

The soil was extracted three times with methanol:1 N acetic acid (80:20) and triplicate aliquots of the extract were analyzed by LSC. Prior to the extraction of the soil in the anaerobic tubes, the

floodwater was pipetted from the soil. The volume of the floodwater was measured and triplicate aliquots were analyzed by LSC. Triplicate subsamples of the extracted soil were analyzed by LSC following combustion. Soil extracts and floodwater samples were stored in the freezer for an unspecified time until analysis by TLC and HPLC. Aliquots of the soil extracts and floodwater were analyzed by TLC on silica gel plates developed in "a solvent system of hexane". Reference standards of PCNB, pentachloroaniline, pentachlorothioanisole, pentachlorobenzene, 2,3,4,5-TCNB, 2,3,5,6-TCNB, and hexachlorobenzene were cochromatographed with the samples. The radioactive areas were located by radioscanning and autoradiography; the radioactive areas were scraped off the plates and quantified by LSC. The identity of degradates detected by TLC was confirmed using reverse-phase HPLC (C_{18} column) eluted with an acetonitrile:water gradient with UV (254 nm) detection. Fractions were collected and analyzed by LSC. Subsamples of the extracted soil were analyzed by LSC following combustion.

Triplicate aliquots of each trapping solution were analyzed using LSC. The ethylene glycol trapping solution was extracted with methylene chloride, the extract was evaporated to dryness, the residues were redissolved in methanol and triplicate aliquots were analyzed using LSC. The remaining extract was concentrated under a stream of nitrogen and aliquots were analyzed by TLC on silica gel plates developed in hexane or hexane:toluene:acetone (7:3:1, v:v:v). Aliquots of the extracts from the anaerobic incubation period were also analyzed by HPLC as previously described.

The remaining extracts from the 60- and 90-day sampling intervals were combined and concentrated. The concentrated extract was analyzed by TLC on silica gel plates developed in hexane:toluene:acetone (7:3:1, v:v:v), the area corresponding to PCA was scraped off the plate, eluted from the silica gel with 5% methanol in methylene chloride and analyzed by GC/MS.

SUPPLEMENTAL STUDY:

In order to provide additional information confirming the identity of residues and information on storage stability, samples from the original study were used. A soil extract that had been frozen for approximately 2 years was re-analyzed and a soil sample from the same sampling interval was extracted and the extract was analyzed. The data from the analyses of the frozen extract and soil were compared to the original data for the corresponding sampling interval.

The frozen soil sample was extracted by a method "similar to the procedure employed in the original study". The soil was extracted three times with methanol:1 N acetic acid (80:20). Aliquots of this soil extract and the original frozen extract were analyzed by TLC on silica gel plates developed in hexane:toluene:acetone (7:3:1, v:v:v). Reverse-phase HPLC had been used in the original study to confirm TLC results. There was poor correlation for quantification between the

two methods (TLC and reverse-phase HPLC). To resolve this problem, in the supplemental document, the extracts were partitioned with hexane and analyzed by normal-phase HPLC on a silica gel column eluted with a hexane:chloroform gradient with UV (254 nm) detection. Fractions were collected and analyzed by LSC.

In the original analysis of the 30-day posttreatment soil extract, PCNB was 82.4% and 96.2% of the extracted radioactivity by TLC and HPLC, respectively, and the degradate

pentachloroaniline (PCA)

was 8.7% by TLC and not detected by HPLC. In the reanalysis of this extract after 2 years of freezer storage, parent PCNB was 90.6% and 91.2% of the extracted radioactivity by TLC and HPLC, respectively; PCA was 6.5% and 3.9% by TLC and HPLC, respectively, and an unidentified degradate(s) was 4.9% as determined by HPLC (this degradate was not detected by TLC.

In the extract of the frozen soil sample, parent PCNB was 86.1% and 91.3% of the extracted radioactivity by TLC and HPLC, respectively; PCA was 8.6% and 4.7% by TLC and HPLC, respectively, and an unidentified degradate(s) was 4.0% as determined by HPLC. This degradate was not detected by TLC. In the supplemental document, uncharacterized residues remaining in the aqueous phase of both extracts after the hexane partitioning step were present at up to 9% of the extracted radioactivity.

DATA SUMMARY:

[^{14}C]PCNB (radiochemical purity 99%), at 10.47 ppm, degraded with a registrant-calculated half-life of 9.0 days in sandy loam soil incubated in the dark at 25.0-26.9°C under anaerobic conditions (flooding plus nitrogen atmosphere) for 60 days following a 30-day aerobic incubation period. The registrant-calculated half-life under aerobic conditions was 81.5 days. Parent PCNB was 94.9% of the applied radioactivity immediately posttreatment, 71.4% at 30 days, 1.3% at 60 days (30 days of anaerobic incubation) and 0.70% at 90 days posttreatment (60 days of anaerobic incubation (Table VII).

The degradates identified in the extracts were

pentachloroaniline (PCA)

at a maximum of 70.3% of applied at day 60 (30 days of anaerobic incubation); and

pentachlorothioanisole (PCTA)

at a maximum of 1.9% at day 30.

Unidentified residues remaining at the TLC origin were a maximum of 7.0% of the total soluble residues at 60 days posttreatment (30 days of anaerobic incubation). Unextracted residues increased from 0.1% of the applied immediately posttreatment to 1.5% of the applied at 30 days posttreatment, and were 9.7-10.4% from 60-90 days posttreatment.

Volatile residues increased from 0.1% at 3 days posttreatment to 5.8% at 90 days (Table V). Parent PCNB was 69.2-82.4% of the volatile radioactivity in the ethylene glycol trap from 3 to 30 days posttreatment and 6.7-14.4% at 60-90 days posttreatment; PCA was 1.5-2.5% at 30 days posttreatment and 69.6-80.1% at 60-90 days posttreatment. An unidentified residue, detected in one TLC system, was 4.8-7.4% of the radioactivity in the ethylene glycol trap at 3-30 days posttreatment (Table VI). The mass balances were 88.1% to 100% of applied (Table V).

PCNB environmental fate review

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Pages 49 through 59 are not included in this copy.

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 product 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

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DATA EVALUATION RECORD

STUDY 5

CHEM 056502 PCNB §163-1

FORMULATION--00--ACTIVE INGREDIENT

STUDY ID 41648201

Dykes, J. 1990. Soil adsorption/desorption with ^{14}C-PCNB. Laboratory project ID: ABC Final Report No. 38405. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc. Columbia, MO., and submitted by AMVAC Chemical Corporation, Los Angeles, CA.

REVIEWED BY: N. Shishkoff TITLE: Staff Scientist

EDITED BY: L. Parsons TITLE: Staff Scientist
W. Martin Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

SECONDARY REVIEW BY: Dana Spatz
TITLE: Chemist
ORG: EFGWB/EFED/OPP

SIGNATURE: JUL 2 2 1991

CONCLUSIONS:

Mobility- Adsorption/desorption studies

1. This study is acceptable and partially fulfills the Leaching-Adsorption/Desorption data requirement by providing information on the mobility (batch equilibrium) of unaged PCNB in silt loam, clay, sandy loam, and sand soils.

2. [^{14}C]PCNB was slightly mobile to immobile in silt loam, clay, sandy loam, and sand soils. Freundlich K_{ads} values were 7.25 for the sand soil, 15.61 for the sandy loam soil, 18.25 for the silt loam soil, and 51.91 for the clay soil.

3. No additional data on the mobility of unaged PCNB in soil are required at this time. However, data are needed on the mobility of aged PCNB residues in soil.

METHODOLOGY:

Silt loam (#21), clay (#36), sandy loam (#79), and sand soils (#92) were air-dried and sieved (2 mm). Based on preliminary batch equilibrium experiments, an equilibration time of 4 hours and a soil:solution ratio of 1:20 (g/mL) were chosen for the definitive study. Additional control samples containing no soil were used to determine if PCNB adsorbs onto glassware.

Uniformly ring-labeled [^{14}C]PCNB (radiochemical purity 99.4%, specific activity 67.8 mCi/mmole, Chemsyn Science Laboratories) was added to sterile 0.01 M $CaCl_2$ solutions at 0.172, 0.133, 0.065, 0.032, and 0.006 μg/mL. The definitive study was repeated with the sandy loam and sand soils with [^{14}C]PCNB concentrations of 0.124, 0.076, 0.024, and 0.006 μg/mL. Duplicate soil samples (1 g) were weighed into sterile culture tubes, and mixed with test solutions (20 mL). Tubes were sealed with teflon lined caps, vortexed, wrapped with aluminum foil, and shaken for 4 hours at 25 $\pm$1°C in the dark. After the equilibration period, the soil:solution slurries were centrifuged, the supernatants were decanted, and the volumes recorded. Duplicate aliquots of the supernatants were analyzed by LSC.

To determine desorption, an equivalent volume of pesticide-free 0.01 M $CaCl_2$ was added to the tubes and shaken as previously described for 24 hours. After shaking, the soil:solution slurries were centrifuged, the supernatants were decanted, volumes measured, and duplicate aliquots analyzed by LSC. Triplicate subsamples of the desorbed soil were analyzed by LSC following combustion.

Portions of the desorbed soils were extracted with methanol:acetic acid (4:1) by shaking overnight and centrifuging; the extracted soils were vortexed and centrifuged with methanol:acetone twice and the extracts were combined. Aliquots of the combined extracts were analyzed by LSC. The extracted soils were air-dried and analyzed by LSC following combustion.

To determine the stability of PCNB, the supernatant from the adsorption phase of the test solution with the highest concentration was analyzed by HPLC (Polygosil C_{18} column) eluted with a water:acetonitrile gradient with UV (254 nm) detection.

DATA SUMMARY:

Based on batch equilibrium studies, [^{14}C]PCNB (radiochemical purity 99.4%) was slightly mobile in silt loam, clay, sandy loam, and sand soil:calcium chloride slurries (1:20) containing concentrations of PCNB ranging from 0.172 to 0.006 μg/mL that were equilibrated for 4 hours at 25°C. Freundlich K_{ads} values were 7.25 for the sand soil, 15.61 for the sandy loam soil, 18.25 for the silt loam soil, and 51.91 for the clay soil; respective K_{oc} values were 2900, 3903, 1521,

and 4326. K_{des} values were 8.19 for the sand soil, 7.53 for the sandy loam soil, 16.69 for the silt loam soil, and 41.85 for the clay soil; respective K_{oc} values were 3276, 1883, 1391, and 3488. Adsorption of PCNB ranged from 25.00% to 90.77% and desorption ranged from 6.31 to 94%. The mass balances for the silt loam and clay soils were 77.95-119.53% and 74.10-121.35% for the sandy loam and sand soils (excluding samples not used for calculations).

After 4 hours of equilibration, PCNB degraded in three of the soils. Parent material ranged from 83.5-89% for the clay, silt loam, and sandy loam soils. Individual degradates were ≤10% of the total radioactivity, and were not identified. PCNB did not degrade in the sand soil (98% of the radioactivity was parent).

COMMENTS:

1. The definitive study was done with all four soils at [^{14}C]PCNB concentrations of 0.172, 0.133, 0.065, 0.032, and 0.006 μg/mL in $CaCl_2$. However, due to poor mass balances, the study was repeated for the sandy loam and sand soils at [^{14}C]PCNB concentrations of 0.340, 0.124, 0.076, 0.024, and 0.006 μg/mL.

2. The study author stated that, in the repeat of the sandy loam (#79) and sand (#92) soils, mass balance for the highest concentration (0.34 ppm) samples was poor (55-58% recovery) compared to mass balances at the other test concentrations (75-121%). Therefore, the data from the highest concentration were not used to calculate the Freundlich K_{ads} values. The study author stated that the poor mass balance was due to the highest concentration exceeding the solubility of PCNB in $CaCl_2$ solution.

3. The study author stated that, at 5 ppm nominal concentration in the preliminary studies, PCNB apparently adsorbed to both silanized and non-silanized glass; however it was later discovered that the solubility of PCNB in $CaCl_2$ solution was <5 ppm. The registrant did not adjust the results of the definitive study for PCNB adsorption to glass.

4. There is an apparent typographical error in Table III; "μg adsorbed onto soil" in sample 21A should be 0.11 instead of 0.24.

PCNB environmental fate review

Page _____ is not included in this copy.

Pages 63 through 87 are not included in this copy.

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___ Identity of product inert ingredients

___ Identity of product impurities

___ Description of the product manufacturing process

___ Description of product 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

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DATA EVALUATION RECORD

STUDY 6

CHEM 056502 PCNB §164-1

FORMULATION--15--EMULSIFIABLE CONCENTRATE

STUDY ID 41721401
Judy, D., B. Jacobson, and W. Guyton. 1990. Terrestrial field dissipation for PCNB in potatoes. Laboratory Project ID: ABC Final Report No. 36971. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and Agri-Growth Research, Inc., Hollandale, MN, and submitted by AMVAC Chemical Corporation, Los Angeles, CA.

DIRECT REVIEW TIME = 16

REVIEWED BY: N. Shishkoff TITLE: Staff Scientist

EDITED BY: K. Ferguson TITLE: Task Leader
W. Martin Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

SECONDARY REVIEW BY: Dana Spatz
TITLE: Chemist
ORG: EFGWB/EFED/OPP

SIGNATURE: JUL 2 2 1991

CONCLUSIONS:

Field Dissipation - Terrestrial

1. This study is acceptable and can be used towards satisfying the Soil Field Dissipation data requirement.

2. PCNB (2-EC, 2 lb/gallon EC), applied at 25 lb ai/A, dissipated very slowly with a registrant-calculated half-life of 324 days from loam soil (0-6" layer) in Minnesota that was treated in July 1988. The pesticide was incorporated (4- to 6-inch depth) immediately after application, and the plot was planted to potatoes. Total PCNB residues [PCNB, pentachloroaniline (PCA), and pentachlorothioanisole (PCTA)] dissipated with a half-life of 436 days. Two degradates were identified: pentachloroaniline (PCA), at a maximum of 1.1 ppm at 515 days, and pentachlorothioanisole (PCTA), at a maximum of 0.25 ppm at

125 and 295 days. PCNB and its degradates were not detected below the 12-inch soil depth.

3. This study may be used to support registration of PCNB applied at or below 25 lb ai/A; additional data are required for use sites at which the registered application rate is greater. For complete fulfillment of the terrestrial field dissipation data requirement, acceptable data are required for each formulation type at two field sites.

METHODOLOGY:

PCNB (2-EC, 2 lb/gallon EC) was broadcast at a nominal rate of 25 lb ai/A onto a plot (60 x 90 feet) of Fieldon loam soil (44% sand, 38% silt, 18% clay, 3.3% organic matter, pH 7.7, CEC 14.3 meq/100 g) using a tractor-mounted spray applicator. The plot, located near Hollandale, Minnesota, was treated on June 23, 1988. A second plot (30 x 60 feet) located 110 feet south of the treated plot served as an untreated control. Immediately after treatment, the pesticide was incorporated into the soil with an S-tine field cultivator to a depth of 4- to 6-inches; potatoes were planted and maintained using normal agricultural practices. For the purpose of sampling, the treated and control plots were divided into 300 and 100 subplots, respectively. Soil cores from fifteen treated subplots and five control subplots were collected prior to and after application, and at 1, 7, 14, 28, 56, 84, 126, 166, 295, 316, 365, 456, and 515 days posttreatment. Through day 28, samples from the 0- to 6-inch soil depth were collected using a hand-operated zero-contamination sampler, then samples from the 6- to 30-inch depth were collected using a zero-contamination hydraulically operated probe; after day 28, 0- to 30-inch cores were collected using the hydraulically operated probe. After sampling, the core holes were filled with untreated soil and marked to prevent resampling. Soil cores were frozen upon arrival at the subcontracting laboratory, and were shipped frozen on dry ice to the analytical laboratory. At the analytical laboratory, the soil cores were grouped into sets of five cores each, divided into 6-inch segments, and composited by depth. The frozen soil was passed three times through a grist mill with enough dry ice to keep the sample frozen, then placed in a polyurethane bottle. The dry ice was allowed to sublimate, and the ground soil was stored at -20°C for up to 1 year until analysis.

Portions of each soil were extracted with acetone by shaking for 30 minutes. An aliquot of the acetone extract was added to aqueous sodium chloride in a separatory funnel. This solution was partitioned against petroleum ether for 1 minute, the phases were allowed to separate, and the organic phase was drained into a flask through anhydrous sodium sulfate. The aqueous phase was repartitioned with petroleum ether, then discarded. The combined organic phases were mixed with a small amount of toluene, and the sample was evaporated until only toluene remained in the flask. The extract was then diluted with toluene and analyzed using GLC with

electron capture detection and GC/MS. Reference standards of PCNB, pentachloroaniline (PCA), pentachlorothioanisole (PCTA), and the impurity hexachlorobenzene (HCB) were used for comparison. Recovery efficiencies from soil samples fortified with PCNB, PCA, PCTA, and HCB ranged from 71 to 112% of the applied, and averaged 91-95% with a standard deviation of 5-7%. The method detection limit was 0.01 ppm.

Ancillary Study - Freezer Storage Stability

Bulk soil samples (uncharacterized) were fortified with 0.10 ppm of PCNB, PCTA, PCA, and HCB, and homogenized. The soils were transferred to polyethylene bottles and stored frozen at -20°C for 12 months; portions of the soil were sampled immediately posttreatment and at 1, 2, 3, 6, 9, and 12 months. The soil samples were analyzed as previously described.

DATA SUMMARY:

PCNB (2-EC, 2 lb/gallon EC), applied at 25 lb ai/A, dissipated with a registrant-calculated half-life of 324 days from loam soil in Minnesota that was treated in July, 1988. The pesticide was incorporated (4- to 6-inch depth) immediately after application, and the plot was planted to potatoes. In the 0- to 6-inch soil depth, parent PCNB averaged 9.1 ppm immediately after treatment, 4.4-4.5 ppm at 316 and 365 days, 1.4 ppm at 456 days, and was 3.3 ppm at 515 days. In the 6- to 12-inch soil depth, PCNB was 0.04-0.28 ppm at 126 days, ≤0.01-0.07 ppm at 166 days, and ≤0.02 ppm at all other sampling intervals. PCNB was not detected in any soil sample below the 12-inch depth, except for 0.021 ppm in the 12- to 18-inch depth at 126 days posttreatment (Table 8).

Total PCNB residues [PCNB, pentachloroaniline (PCA), and pentachlorothioanisole (PCTA)] dissipated with a half-life of 436 days in the surface 6 inches of soil. PCNB residues totaled 9.2 ppm at day 0, 4.4 ppm at day 316, and 3.3 ppm at day 515. The degradate

pentachloroaniline (PCA)

averaged 0.08 ppm in the 0- to 6-inch soil layer immediately posttreatment and increased to a maximum 1.1 ppm at 515 days. PCA was detected in the 6- to 12-inch layer at 0.019-0.023 ppm on days 126 and 456; it was <0.01 ppm in samples deeper than 12 inches at all sampling intervals.

Pentachlorothioanisole (PCTA)

was first detected at 7 days posttreatment at an average 0.03 ppm, was a maximum 0.25 ppm at 126 and 295 days, and decreased to 0.17 ppm at 515 days. PCTA was <0.01 ppm in samples deeper than 6 inches at all sampling intervals.

a contaminant of PCNB, was not detected in any soil sample.

During the study, average high and low temperatures were 57 and 36°F, respectively. Total precipitation plus irrigation for the entire 515-day period was 44.57 inches.

Ancillary Study - Freezer Storage Stability

PCNB was stable in soil samples treated with PCNB at 0.10 ppm and stored frozen at -20°C for 9 months; PCNB ranged from 90 to 104% of the applied with no discernable pattern during this period (Table 15). PCNB was 83 and 88% of the applied in duplicate frozen soil samples after 12 months of storage.

PCTA, PCA, and HCB were stable in soil samples treated with the respective compound at 0.10 ppm and stored frozen at -20°C for 9 months (Table 15). PCTA ranged from 91 to 115% of the applied, PCA ranged from 63 to 83%, and HCB ranged from 87 to 99%, all with no discernable pattern during this period. At 12 months posttreatment, PCTA was 61 and 88% of the applied, PCA was 62%, and HCB was 79 and 87%.

COMMENTS:

1. The actual application rate appeared to be approximately 75% of the nominal rate of 25 lb ai/A (which is equivalent to 12.5 ppm in the 0- to 6-inch soil depth). Filter paper discs, spread on the test soil before the application of PCNB, contained only 62-80% of the expected concentration (average 74 ± 8%), and the time 0 soil sample contained only 9.2 ppm of PCNB residues.

2. Samples from the untreated control soil contained low concentrations (<0.04 ppm) of PCTA. The study authors suggested that the source of the PCTA was either sample contamination or previous chemical use.

3. The treated plots were at an elevation of 1210 feet, with a slope of less than 1%. There was no subsurface drainage, and the seasonal average depth to the water table was >4 feet. In 1985, the test plot was planted to corn and treated with alachlor and cyanazine at 2.5 and 2 lbs ai/A, respectively; in 1986, the test plot was planted to soybeans and treated with fluchloralin and metribuzin at rates of 1 lb and 0.38 ai/A, respectively; in 1987, the test plot was planted to soybeans and treated with alachlor and metribuzin at 2.5 and 0.38 lbs ai/A, respectively.

Ancillary Study - Freezer Storage Stability

Following 9 months of relative stability in the frozen soil, the concentrations of all four compounds decreased at the 12-month sampling interval. This suggests that the observed decrease may have been due to poor extraction techniques or other types of errors, rather than to degradation of the compounds.

PCNB environmental fate review

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___ Identity of product impurities

___ Description of the product manufacturing process

___ Description of product 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

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DATA EVALUATION RECORD

STUDY 7

CHEM 056502 PCNB §164-1

FORMULATION--06--WETTABLE POWDER

STUDY ID 41747001

Rice, F., B. Jacobson, and B. Guyton. 1990. Terrestrial field dissipation for PCNB in broccoli. Laboratory Project ID: ABC Final Report No. 36972. Unpublished study performed by Analytical Bio-Chemistry Laboratories, Inc., Columbia, MO, and Plant Sciences, Inc., Watsonville, CA, and submitted by AMVAC Chemical Corporation, Los Angeles, CA.

DIRECT REVIEW TIME = 16

REVIEWED BY: N. Shishkoff TITLE: Staff Scientist

EDITED BY: C. Cooke TITLE: Staff Scientist
W. Martin Staff Scientist

APPROVED BY: W. Spangler TITLE: Project Manager

ORG: Dynamac Corporation
Rockville, MD

SECONDARY REVIEW BY: Dana Spatz
TITLE: Chemist
ORG: EFGWB/EFED/OPP

SIGNATURE: JUL 2 2 1991

CONCLUSIONS:

Field Dissipation - Terrestrial

1. This study cannot be used to fulfill the Soil Field Dissipation data requirement.

2. This study is unacceptable for the following reason:

The reported application rate of 30 lbs ai/A was not confirmed by the soil residue measurements taken immediately post-treatment. The actual application rate calculated from soil analysis was approximately 45% of the nominal rate of 30 lb ai/A (which is equivalent to 15.0 ppm in the 0- to 6-inch soil depth). Filter paper discs, placed on the test soil before the application of PCNB, contained only 12-55% of the expected

concentration (average 29 ± 13%), and the time 0 soil sample contained only 6.8 ppm of PCNB residues. The authors suggest that the volatility of PCNB may explain this loss. This explanation may account for some of the loss, however, a better explanation may be related to the fact that the concentration of PCNB in the spray solution was less than expected since it appears from the data that the test spray solution contained only 47% of the theoretical concentration of PCNB.

It should also be noted that the product label recommends soil incorporation to a depth of 4-6 inches immediately after application for effective control. No mention of soil incorporation was made in the report.

3. PCNB (75% WP), applied at 30 lb ai/A, dissipated very slowly with a registrant-calculated half-life of 305 days from sandy loam soil in California that was treated in September 1988. The plot was planted the next day to broccoli. Total PCNB residues [PCNB, pentachloroaniline (PCA), and pentachlorothioanisole (PCTA)] dissipated with a half-life of 401 days. In the 0- to 6-inch soil depth, parent PCNB averaged 6.7 ppm immediately after treatment. Two degradates were identified: pentachloroaniline (PCA), at a maximum of 0.39 ppm at 556 days, and pentachlorothioanisole (PCTA), at a maximum of 0.35 ppm at 275 days. PCNB and its degradates were not detected below the 12-inch soil depth.

METHODOLOGY:

PCNB (75% WP, source not specified) was broadcast at a nominal rate of 30 lb ai/A onto a plot (105 x 60 feet) of sandy loam soil (72% sand, 20% silt, 8% clay, 2.0% organic matter, pH 6.6, CEC 16.9 meq/100 g) using a tractor-mounted spray applicator. The plot, located in Santa Cruz County, California, was treated on September 27, 1988. A second plot (33 x 60 feet) located 140 feet northeast of the treated plot served as an untreated control. On September 28, 1988 broccoli was planted and maintained using normal agricultural practices. For the purpose of sampling, the treated and control plots were divided into 300 and 100 subplots, respectively. Soil cores from fifteen treated subplots and five control subplots were collected prior to and after application, and at 1, 7, 14, 28, 66, 84, 125, 191, 232, 275, 315, 373, 465, and 556 days posttreatment. Through day 28, samples from the 0- to 6-inch soil depth were collected using a hand-operated zero-contamination sampler, then samples from the 6- to 30-inch depth were collected using a zero-contamination hydraulically operated probe; after day 28, 0- to 30-inch cores were collected using the hydraulically operated probe. After sampling, the core holes were filled with untreated soil and marked to prevent resampling. Soil cores were frozen upon arrival at the subcontracting laboratory, and were shipped frozen on dry ice to the analytical laboratory. At the analytical laboratory, the soil cores were grouped into sets of five cores each, divided into 6-inch

segments, and composited by depth. The frozen soil was passed three times through a grist mill with enough dry ice to keep sample frozen, then placed in a polyurethane bottle. The dry ice was allowed to sublimate, and the ground soil was stored at -20°C for up to 1 year until analysis.

Portions of each soil were extracted with acetone by shaking for 30 minutes. An aliquot of the acetone extract was added to aqueous sodium chloride in a separatory funnel. This solution was partitioned against petroleum ether for 1 minute, the phases were allowed to separate, and the organic phase was drained into a flask through anhydrous sodium sulfate. The aqueous phase was repartitioned with petroleum ether, then discarded. The combined organic phases were mixed with a small amount of toluene, and the sample was evaporated until only toluene remained in the flask. The extract was then diluted with toluene and analyzed using GLC with electron capture detection and GC/MS. Reference standards of PCNB, pentachloroaniline (PCA), pentachlorothioanisole (PCTA), and the impurity hexachlorobenzene (HCB) were used for comparison. Recovery efficiencies from soil samples fortified with PCNB, PCA, PCTA, and HCB ranged from 70 to 119% of the applied, and averaged 90-94% with a standard deviation of 6-11%. The method detection limit was 0.01 ppm.

Ancillary Study - Freezer Storage Stability

Bulk soil samples (uncharacterized) were fortified with 0.10 ppm of PCNB, PCTA, PCA, and HCB, and homogenized. The soils were transferred to polyethylene bottles and stored frozen at -20°C for 12 months; portions of the soil were sampled immediately posttreatment and at 1, 2, 3, 6, 9, and 12 months. The soil samples were analyzed as previously described.

DATA SUMMARY:

PCNB (75% WP), applied at 30 lb ai/A, dissipated with a registrant-calculated half-life 305 days from sandy loam soil in California that was treated in September, 1988. The plot was planted the following day to broccoli. In the 0- to 6-inch soil depth, parent PCNB averaged 6.7 ppm immediately after treatment, 3.2 ppm at 315 days, 2.2 ppm at 465 days, and was 2.5 ppm at 556 days. In the 6- to 12-inch soil depth, PCNB was 0.15-0.22 ppm at 125 days, 0.06-1.6 ppm at 275 days, 0.03-0.32 ppm at 465 days, and 0.07-0.23 at 556 days. PCNB was not detected in any soil sample below the 12-inch depth, except for 0.012 and 0.015 ppm in the 12- to 18-inch depth at 0 and 1 days posttreatment. Total PCNB residues [PCNB, pentachloroaniline (PCA), and pentachlorothioanisole (PCTA)] dissipated with a half-life of 401 days in the surface 6 inches of soil. PCNB residues totaled 6.8 ppm at day 0, 3.9 ppm at day 315, 2.9 ppm at day 465, and 3.2 ppm at day 556. The degradate

pentachloroaniline (PCA)

averaged 0.07 ppm in the 0- to 6-inch soil layer immediately posttreatment and increased to a maximum 0.39 ppm at 556 days. PCA was detected in the 6- to 12-inch layer at a maximum level of 0.088 ppm at 373 days; it was <0.01 ppm in samples deeper than 12 inches at all sampling intervals.

Pentachlorothioanisole (PCTA)

was first detected at 7 days posttreatment at an average 0.04 ppm, was a maximum 0.35 ppm at 275 days, and decreased to 0.27 ppm at 556 days. PCTA was detected in the 6- to 12-inch layer at a maximum level of 0.06 ppm at days 232, and 373; it was <0.01 ppm in samples deeper than 12 inches at all sampling intervals.

Hexachlorobenzene (HCB)

a contaminant of PCNB, was not detected in any soil sample.

During the study, average high and low temperatures were 67 and 43°F, respectively. Total precipitation plus irrigation for the entire 556-day period was 50.09 inches.

Ancillary Study - Freezer Storage Stability

PCNB was stable in soil samples treated with PCNB at 0.10 ppm and stored frozen at -20°C for 12 months; PCNB ranged from 76 to 119% of the applied with no discernable pattern during this period.

PCTA, PCA, and HCB were stable in soil samples treated with the respective compound at 0.10 ppm and stored frozen at -20°C for 12 months. PCTA ranged from 88 to 98% of the applied, PCA ranged from 90 to 106%, and HCB ranged from 86 to 94%, all with no discernable pattern during this period.

COMMENTS:

1. The treated plots were at an elevation of 200 feet, with a slope of less than 1%. There was no subsurface drainage, and the seasonal average depth to the water table was >6 feet. In 1987, the test plot was planted to lettuce and cauliflower and treated with chlorthal dimethyl (1.70 lb ai/A), chlorpyrifos (1 lb ai/A), MSR (0.5 lb ai/A), mevinphos (3.25 lb ai/A), permethrin (0.4 ai/A), benefin (0.9 lb ai/A), isopropylin carbanilate (1 lb ai/A), disulfoton (2 lb ai/A), acephate (1.12 lb ai/A), isoproturon (1.33 lb ai/A), maneb (1.6 lb ai/A and 1.2 lb ai/A), methomyl (0.45 lb ai/A), metalaxyl (0.6 lb ai/A), and folpet (1 lb ai/A).

PCNB environmental fate review

Page _____ is not included in this copy.

Pages 120 through 137 are not included in this copy.

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 product 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

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