Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Pentachloronitro benzene

MRID #: 440677-01

Matrix: Soil

Analysis: GC/MS

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abc Laboratories

Working for You *Columbia, Missouri*

ABC METHOD 42126/42127/42128-Unified Rev 1.0

Determination of Pentachloronitrobenzene and Metabolites/Manufacturing By-Products Pentachlorobenzene, Hexachlorobenzene, Pentachloroaniline, and Pentachlorothioanisol in Bareground Soil, Turfgrass Soil, and Turfgrass Samples

ABC Study Numbers: 42126, 42127, and 42128

Sponsor: Amvac Chemical Corporation
4100 East Washington Boulevard
Los Angeles, California 90023

Test Substance: PCNB 10G (Bareground Soil)
PCNB 75W (Turfgrass Soil and Turfgrass)

Reference Substances: Pentachloronitrobenzene, Pentachlorobenzene, Hexachlorobenzene, Pentachloroaniline, Pentachlorothioanisol

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1.0 OBJECTIVES

The purpose of this method is to quantitate the residues of Pentachloronitrobenzene (PCNB) and metabolites/manufacturing by-products Pentachlorobenzene (PeCB), Hexachlorobenzene (HeCB), Pentachloroaniline (PCA), and Pentachlorothioanisol (PCTA) in bareground and turfgrass soils, and on turfgrass samples.

2.0 EXPERIMENTAL DESIGN

2.1 Summary

Soil Samples

One hundred grams of homogenized soil is extracted with 500 mL acetone and filtered into a separatory funnel. A 200-mL aliquot of aqueous 10% sodium chloride solution is added to the filtered acetone extract, and the sample extracted twice with 200-mL aliquots of petroleum ether. The petroleum ether extracts are washed with 200 mL deionized water to remove excess water and coextracted acetone, then dried by draining the petroleum ether through a bed of sodium sulfate. Approximately 2 mL of toluene is added to the sample and the extract rotary evaporated to ~5 mL. The remaining solvent and residues are transferred to precalibrated centrifuge tubes with petroleum ether, and the samples concentrated to ~2 mL under a gentle stream of N_2. An additional ~2-mL aliquot of toluene is added to the sample and the sample is concentrated back to ~2 mL to complete the solvent exchange. Final volume adjustments and dilutions are prepared at this time, and the samples are analyzed by gas chromatography (GC) utilizing mass selective detection (MSD).

Turfgrass Samples

Ten grams of homogenized turfgrass is extracted with 250 mL acetone and filtered into a separatory funnel. A 100-mL aliquot of aqueous 10% sodium chloride solution is added to the filtered acetone extract, and the sample extracted twice with 100-mL aliquots of petroleum ether. The petroleum ether extracts are washed with 100 mL deionized water to remove excess water and coextracted acetone, then dried by draining the petroleum ether through a bed of sodium sulfate. Approximately 2 mL of toluene is added to the sample and the extract rotary evaporated to ~5 mL. The remaining solvent and residues are transferred to precalibrated centrifuge tubes with petroleum ether, and the samples concentrated to ~2 mL under a gentle stream of N_2. An additional ~2-mL aliquot of toluene is added to the sample and the sample is concentrated back to ~2 mL to complete the solvent exchange. Final volume adjustments and dilutions are prepared at this time, and the samples are analyzed by gas chromatography (GC) utilizing mass selective detection (MSD).

2.2 Sample Analysis

Each set of samples will include an unfortified control sample. For analysis sets that contain six or fewer samples, a minimum of one fortification sample will be generated concurrently with the sample set. For analysis sets that contain more than six samples, a minimum of two fortification samples will be generated concurrently with the sample set. The samples will be quantified against standard curves generated from the GC/MSD analysis of PCNB, PeCB, HeCB, PCA, and PCTA reference substances as outlined in Section 3.7 of this method. The recovery of all fortification experiments after correcting for interference/residue in the control matrix will be 70-120% for the set to be considered acceptable. In addition, the standard curves generated from the reference standards will have a linear correlation coefficient of 0.995 or greater, and the detector response observed from repeat injection of a standard level should fall within ± 15% (± 20% for the low level standard) for the curve to be considered valid.

2.3 Reference Substances

Analytical grade standard (reference substance) of PCNB and the metabolites/manufacturing by-products PeCB, HeCB, PCA, and PCTA were obtained from Amvac Chemical Corporation, Los Angeles, California. The certificate of analysis for the reference substances included the information listed in the table below. They were stored in a freezer at ~-20 °C when not in use.

Compound (CAS #)	Source	Ref. #	Purity	Exp. Date	Amount	Storage
PCNB (82-68-8)	Amvac	690201	99.8%	08/20/97	1.0 g	Cool, dark
PeCB (608-93-5)	Pfaltz & Bauer	PO2030	97%	03/10/96	0.5 g	Cool, dark
HeCB (118-74-1)	Amvac	312802	99.9+%	07/15/99	0.5 g	Cool, dark
PCA (527-20-8)	Alfa Products	072384	99.0+%	07/11/97	0.5 g	Cool, dark
PCTA (1825-19-0)	Lancaster Synthesis	AM22891	98.9%	03/11/96	0.5 g	Cool, dark

3.0 EXPERIMENTAL

3.1 Weighing and Fortifying of Samples

Weigh 100 g of homogenized soil, or 10 g of homogenized turfgrass, into a 1-L glass jar. Prepare fortifications as necessary at this point.

3.2 Soil or Turfgrass Extraction and Filtration

Add 500 mL of acetone to a soil sample, or 250 mL of acetone to a turfgrass sample, and close the jar using a piece of aluminum foil and a Teflon-lined screw cap. Place the samples on an oscillating shaker and shake for 30 min at a low setting. Assemble a vacuum filtration system consisting of the following: 1) a 12.5 cm i.d. Büchner funnel attached to an adapter inlet with a piece of solvent resistant plastic to form a seal (such as a cut out #24 stopper); 2) a 29/42 to 24/40 standard taper reducing adapter to bridge the gap between the adapter inlet and a 1000-mL separatory funnel; 3) tubing to attach to the adaptor inlet and a solvent trap/vacuum source capable of ~25 inches Hg vacuum (~5 inches Hg pressure). Decant the sample through a Büchner funnel containing GF/A filter paper directly into a 1000-mL separatory funnel under vacuum. Add an additional 25 mL of acetone to the sample jar, shake, and transfer to the Büchner funnel. Upon completion of the filtration, remove the separatory funnel from the vacuum filtration apparatus and discard the filter cake.

3.3 Petroleum Ether Extraction and Drying

Add 200 mL of a 10% NaCl aqueous solution to a soil sample, or 100 mL of a 10% NaCl aqueous solution to a turfgrass sample, and swirl to mix. Extract a soil sample with 200 mL of petroleum ether, and a turfgrass sample with 100 mL of petroleum ether, by vigorously shaking for ~1 min. Allow the phases to separate, and drain the lower aqueous phase into a 1000-mL flat-bottomed boiling flask and set aside. Drain the upper petroleum ether phase into a second 1000-mL flat-bottomed boiling flask and set aside. Transfer the aqueous phase back to the first 1000-mL separatory funnel, and extract the aqueous sample again using the appropriate aliquot volume of petroleum ether as previously described. Allow the phases to separate, and discard the lower aqueous phase. Transfer the first petroleum ether extract to the 1000-mL separatory funnel, thus combining the petroleum ether extracts. Rinse the second 1000-mL flat-bottomed boiling flask with an additional 25 mL of petroleum ether and add to the combined extracts. Wash combined soil extracts with 200 mL of DI water, and combined turfgrass extracts with 100 mL of DI water, shaking vigorously for ~1 min. Allow the phases to separate, and discard the lower aqueous phase. Drain the petroleum ether extract through a powder funnel containing ~120 g (~80 cc of 12-60 mesh) anhydrous Na_2SO_4 into a 1000-mL flat-bottomed boiling flask. This drying step should be performed slowly to maximize efficiency of the water removal. The glass wool plug should be tightly inserted into the powder funnel to control

the rate at which the petroleum ether passes through the anhydrous Na_2SO_4. Rinse the anhydrous Na_2SO_4 with an additional 25 mL aliquot of petroleum ether, and add ~2 mL of toluene to the sample.

3.4 Concentration, Solvent Exchange, and Volume Adjustment

Rotary evaporate the sample in a water bath at <20 °C (~16 °C ± 3 °C) until only ~5 mL of toluene/petroleum ether remains. Transfer the sample to precalibrated 13-mL centrifuge tubes with petroleum ether, and continue concentration of the sample to ~2 mL under a gentle stream of N_2. Add an additional ~2 mL of toluene to each sample, and continue concentration to ~2 mL under a gentle stream of N_2 in a 25-35 °C water bath. Adjust the sample to the appropriate final volume and prepare dilutions as necessary with toluene.

3.5 Instrument Conditions

Instrument: Hewlett-Packard 5890 Series II Gas Chromatograph equipped with a 7673B Autosampler, a 5972A Mass Selective Detector, and a Vectra 486/66U computer operating with HP G1034C MS Chemstation Software

Injector Configuration: On-Column injection

Column: HP-5MS, 30 m x 0.25 mm x 0.25 μm film thickness connected to 5 m x 0.53 mm deactivated retention gap at inlet end (minimum of 1 m required). The columns are joined with a stainless steel column connector (e.g. J&W Scientific Part #5001153; Scientific Products Catalog #C4586-339). The 0.25 mm analytical column end is inserted ~4 cm into the 0.53 mm retention gap, and the connector forms a zero dead volume seal.

Carrier Gas: Helium, operating in constant flow mode (0.70 mL/min), Vacuum Compensation On

Injection Volume: 1.0 μL

Injector Temperature: Oven Track Mode (3 °C above oven temperature)

Detector Temperature: 280 °C

Oven Temperatures: (PCNB Analysis)

Initial: 90 °C, hold for 0 min
Ramp 1: 20 °C/min to 130 °C, hold for 4.00 min
Ramp 2: 15 °C/min to 280 °C, hold for 10.00 min

Oven Temperatures: (PeCB, HeCB, PCA, and PCTA Analysis)

Initial: 90 °C, hold for 0 min
Ramp 1: 25 °C/min to 140 °C, hold for 3.00 min
Ramp 2: 5 °C/min to 210 °C, hold for 0.00 min
Ramp 3: 25 °C/min to 280 °C, hold for 8.00 min

SIM Values

Because of the large differences in the levels of PCNB (ppm range) relative to the metabolites/manufacturing by-products (ppb range), the samples are analyzed for PCNB and metabolites/manufacturing by-products using separate injections, and often with different dilution factors.

PCNB Analysis: m/z=237, Retention time ~11-12 min

PeCB, HeCB, PCA, and PCTA Analysis:

PeCB:	m/z=250, Retention time ~7.5-10 min
HeCB:	m/z=284, Retention time ~11.3-12.3 min
PCA:	m/z=265, Retention time ~12.9-15.1 min
PCTA:	m/z=296, Retention time ~13.5-17.0 min

The retention times shown will vary with length and age of analytical column, modifications to optimize the chromatography and integration, etc. These values are given only as approximations based on the oven temperature parameters shown in this section. The elution order shown is based on use of the analytical column described in this section. Equivalent equipment may be used. Operating parameters may be modified as necessary to optimize chromatography.

3.6 Standards

Primary standard solutions of the various reference substances are prepared as described in Section 5.0 of this method. All fortifications are prepared with the reference substances with acetone as the solvent, and all quantitation standards are prepared with toluene as the solvent.

3.7 Quantitation

A standard curve is generated to bracket the anticipated levels of PCNB or metabolites/manufacturing by-products in the samples. Standards are injected before and after the samples in a set, with a standard injected after approximately every third sample in a set. The linear correlation coefficient of the peak areas is required to be >0.995 for a set to be considered valid, and the detector response observed from repeat injection of a standard level

is required to fall within ± 15% (± 20% for the low level standard) for the curve to be accepted.

4.0 EQUIPMENT AND REAGENTS

Assorted class A volumetric flasks (e.g.-Kimax #28015P-50 and #28015P-100)
Assorted class A pipets (e.g.-Kimax #37004-1 and #37004-5)
Assorted Hamilton Syringes (e.g.-Hamilton #710 and #750)
Assorted graduated cylinders (e.g.-Kimax #20025-100 and #20025-500)
Disposable glass transfer pipets (Pasteur pipets, 9")
Balances (analytical balance capable of 0.1 mg resolution for weighing of standards, and laboratory balance capable of 0.01 g resolution for weighing sample matrix)
Whatman GF/A filter paper (12.5 cm, #1820 125)
1-Liter (32 oz) glass jars with 89/400 Teflon lined screw caps (e.g.-Scientific Products #B7465-50)
Büchner funnel, 12.5 cm i.d., porcelain (e.g.-Fisher Scientific #10-356F)
Reducing glass adapter, Bushing type with 29/42 to 24/40 joints (e.g-Kimble #44865-2942)
Inlet adapter for vacuum attachment, 24/40 joints (e.g.-Kontes #205000-2440)
1000-mL separatory funnels w/PTFE plug stopcocks and polyethylene stopper (e.g.-Kimble #29049F-1000)
1000-mL flat-bottomed boiling flasks (e.g.-Kimble #25055-1000)
Glass powder funnels (e.g.-Kimble #29020-80)
Glass wool for powder funnel plug (e.g.-Fisher Scientific #11-388)
13 mL centrifuge tubes (Fisher Scientific #05-538-40A)
Reagent grade sodium sulfate (rinsed prior to use with petroleum ether) (e.g.-J.T. Baker #3375-07, ACS Reagent Grade, Anhydrous, Granular (12-60 mesh), 12 kg)
Reagent grade sodium chloride (e.g.-J.T. Baker, #3624-07, ACS Reagent Grade, Crystal, 12 kg)
Water baths for rotary evaporation and N_2 concentration
Pesticide grade acetone (e.g.-Burdick and Jackson)
Pesticide grade petroleum ether (e.g.-Burdick and Jackson)
Pesticide grade toluene (e.g.-Burdick and Jackson)
Deionized water (e.g.-Labconco Water Pro PS System, 18 MΩ)
Vacuum rotary evaporation system

Equivalent materials may be substituted. Glassware may be silanized if necessary. The silanizing agent recommended is 5% dichlorodimethylsilane in toluene. Allow clean glassware to be exposed to the silanizing reagent for ~15 sec, and then thoroughly rinse the surface with three aliquots of toluene followed by three aliquots of methanol. For this method, the glassware must be washed again after silanization due to a series of interfering m/z=237 peaks in the SIM analysis of PCNB.

5.0 PREPARATION OF INJECTION AND FORTIFICATION STANDARDS

Quantitation standards are prepared from analytical grade reference substances listed in Section 2.3 of this method. Stock solutions are prepared containing ~1.0 mg/mL of a reference substance in pesticide grade toluene, and dilutions are prepared in the range of ~200 ng/mL to ~2000 ng/mL for PCNB. An example chromatogram for a PCNB quantitation standard is shown in Figure 1. Mixed standards are prepared for the metabolites (PCNB is not present in the mixed metabolite standards) in the range of ~20 ng/mL to ~800 ng/mL for PeCB and PCTA, and from ~100 ng/mL to ~3200 ng/mL for PCA and HeCB. An example chromatogram for a mixed metabolite quantitation standard is shown in Figure 2.

Fortification solutions are prepared from the same analytical grade reference substances listed in Section 2.3 of this method. Stock solutions are prepared containing ~1.0 mg/mL reference substance in pesticide grade acetone, and dilutions are prepared at ~ 50 µg/mL and ~1.0 µg/mL for PCNB in acetone. Stock solutions can be prepared in a more concentrated form (~25 mg/mL) for high level fortifications if necessary. Mixed fortification solutions are prepared for the metabolites/manufacturing by-products in acetone (PCNB is not present in the mixed metabolite solutions). Fortifications are prepared by adding a known volume of the appropriate standards in acetone to a known weight of control soil matrix.

6.0 VALIDATION RANGES AND DISCUSSION OF METHOD DIFFICULTIES

The methods described were validated on various matrices including bareground soil, turfgrass soil, and turfgrass. The method validation sets included duplicate controls, and fortifications of control matrix in duplicate in ranges intended to be representative of the levels anticipated in study samples. The relatively high levels of PCNB to the low levels of metabolites required validation of the metabolites as a function of the level of PCNB in the fortification, and required the selectivity of GC/MSD.

Bareground Soils Validated #1

Soil Horizon (inches)	ppm PCNB	ppm PeCB	ppm HeCB	ppm PCA	ppm PCTA
0-6"	250-0.0499	2.0-0.0050	2.00-0.0250	2.00-0.0500	2.00-0.00500
LOQ 6-12" and below	0.00998	0.0050	0.00500	0.00500	0.00500

It should be noted that in the presence of ~10 ppm PCNB, as may be found in the 0-6" soil depth, HeCB and PCA LOQ values are a minimum of 0.025 ppm and 0.050 ppm, respectively. A summary of the method validation results for the most difficult soil depth, 0-6", is presented in Table 1. Example chromatograms from the bareground soil validation are presented in Figures 3-4*.

Turfgrass Soils Validated #2

Soil Horizon (inches)	ppm PCNB	ppm PeCB	ppm HeCB	ppm PCA	ppm PCTA
0-6"	9.97-0.00998	0.50-0.0050	0.500-0.0500	0.500-0.250	0.500-0.00500
LOQ 6-12" and below	0.00998	0.0050	0.00500	0.00500	0.00500

It should be noted that in the presence of ~10 ppm PCNB, as may be found in the 0-6" soil depth, HeCB and PCA LOQ values are a minimum of 0.050 ppm and 0.25 ppm, respectively. A summary of the method validation results for the most difficult soil depth, 0-6", is presented in Table 2. Example chromatograms from the turfgrass soil validation are presented in Figures 5-6*.

Turfgrass Matrix Validated #3

	ppm PCNB	ppm PeCB	ppm HeCB	ppm PCA	ppm PCTA
Turfgrass	3750-0.998	10-0.10	100-0.100	100-0.100	10.0-0.100

It should be noted that in the presence of 3750 ppm PCNB, HeCB and PCA LOQ values are a minimum of 100 ppm. A summary of the method validation results for the turfgrass is presented in Table 3. Example chromatograms from the turfgrass validation are presented in Figures 7-8*.

*These example chromatograms are reduced in size from the original chromatograms; they are 74% the size of the original documents.

The method described is very rugged for the analysis of PCNB and metabolites/manufacturing by-products with the exception of a single, very consistent phenomenon. The LOQ's for HeCB and PCA are a function of the level of PCNB present in the sample, and a function of the amount of coextracted material from the matrix. It

appears that the HeCB is a manufacturing by-product in the PCNB formulations, and thus its levels are directly related to the level of PCNB present in a sample. The PCA appears to be influenced by the level of PCNB present in a sample and the amount of coextracted material present in the final concentrate injected onto the chromatographic system. Even though a thermally-gentle on-column system is used, reduction of PCNB to PCA appears to occur on the chromatographic column when matrix coextractants are dense. These factors limit the LOQ for both HeCB and PCA in the presence of high levels of PCNB (> 10 ppm).

Due to the high levels of coextracted materials (high grams matrix extracted relative to the final extract volume), deterioration of chromatographic performance can be expected if adequate maintenance is not carried out. Generally, after injecting a set of samples, including standards, blanks, and samples, the inlet end of the retention gap should be clipped off ~30 cm. This routine may be carried out until only ~1 m of the original 5 m retention gap remains. At this point, it is necessary to install a new retention gap, and it is recommended that the analytical column be clipped off ~30 cm prior to connecting the new retention gap and the analytical column.

7.0 SIGNATURES

This unified method supersedes methods previously used for analysis of PCNB and metabolites/manufacturing by-products. These include the following:

ABC Method Number 42126/42127-Soil Rev. 1.0, entitled, "Determination of Pentachloronitrobenzene and Metabolites Pentachlorobenzene, Hexachlorobenzene, Pentachloroaniline, and Pentachlorothioanisol in California and Georgia Bareground Soil," effective date February 14, 1995.

ABC Method Number 42128-Soil Rev. 2.0, entitled, "Determination of Pentachloronitrobenzene and Metabolites Pentachlorobenzene, Hexachlorobenzene, Pentachloroaniline, and Pentachlorothioanisol in California Turfgrass Soil, " effective date February 14, 1995.

It was necessary to revise the methods including the details of the instrumental analysis, and to include the validated turfgrass method prior to initiating the analysis of the turfgrass samples. Whereas the description of the method for the soil analysis has increased in this unified method, the content and practice of the method has not changed in any way from the above mentioned methods. Thus, the methods used to date in the analysis of soils are valid and have not changed with this revision. This method will be used beginning April 17, 1995, in the analysis of soil samples in ABC Study #42126. SARS Protocol # SARS-94-75, "Pentachlorobenzene: Terrestrial Field Dissipation of PCNB 10G on Bareground Plot in California," ABC Study #42127, SARS Protocol # SARS-94-76,

"Pentachlorobenzene: Terrestrial Field Dissipation of PCNB 10G on Bareground Plot in Georgia," and soil and turf samples in ABC Study #42128, SARS Protocol # SARS-94-77, "Pentachlorobenzene: Terrestrial Field Dissipation of PCNB 75W on Turfgrass Plot in California."

Loren C. Schrier 4/28/95

Loren C. Schrier Date
Principal Analytical Investigator
ABC Laboratories, Inc.

Bruce C. Leppert 4/28/95

Bruce C. Leppert Date
Study Director
STEWART Agricultural Research Services, Inc.

Table 1. Summary of 0-6" Bareground Soil Method Validation Data for PCNB and Metabolites/Manufacturing By-Products

Sample ID	Sample Description	% PCNB Rec.	% PeCB Rec.	% HeCB Rec.	% PCA Rec.	% PCTA Rec.
42126-003	9.97 ppm PCNB + 0.50 ppm Metabolites	97.4	94	100	105	96.8
42126-004	9.97 ppm PCNB + 0.50 ppm Metabolites	93.6	94	99.0	101	93.6
42126-005	0.00998 ppm PCNB + 0.0050 ppm PeCB & PCTA + 0.025 ppm HeCB + 0.050 ppm PCA	Interference in Control	86	84.0	82.7	88.0
42126-006	0.00998 ppm PCNB + 0.0050 ppm PeCB & PCTA + 0.025 ppm HeCB + 0.050 ppm PCA	Interference in Control	94	92.4	89.9	95.6
42126-007	9.97 ppm PCNB + 0.0050 ppm PeCB & PCTA + 0.025 ppm HeCB + 0.050 ppm PCA	97.8	78	92.8	93.5	85.2
42126-008	9.97 ppm PCNB + 0.0050 ppm PeCB & PCTA + 0.025 ppm HeCB + 0.050 ppm PCA	94.8	96	112	112	102
42126-019	0.0998 ppm PCNB	91.1	-	-	-	-
42126-020	0.0998 ppm PCNB	91.0	-	-	-	-

Table 2. Summary of 0-6" Turfgrass Soil Method Validation Data for PCNB and Metabolites/Manufacturing By-Products

Sample	Sample Description	% PCNB Rec.	% PeCB Rec.	% HeCB Rec.	% PCA Rec.	% PCTA Rec.
42128-021	9.97 ppm PCNB + 0.50 ppm Metabolites	93.4	88	90.6	115	93.4
42128-022	9.97 ppm PCNB + 0.50 ppm Metabolites	96.5	92	96.6	120	95.2
42128-023	0.00998 ppm PCNB +0.50 ppm Metabolites	81.3	94	96.4	110	100
42128-024	0.00998 ppm PCNB +0.50 ppm Metabolites	78.5	84	85.6	100	89.6
42128-025	9.97 ppm PCNB +0.0050 ppm Metabolites	101	98	> 120	> 120	109
42128-008	9.97 ppm PCNB +0.0050 ppm Metabolites	112	100	> 120	> 120	112
42128-040	9.97 ppm PCNB +0.050 ppm HeCB + 0.100 ppm PCA	109	-	114	> 120	-
42128-041	9.97 ppm PCNB +0.050 ppm HeCB + 0.100 ppm PCA	110	-	115	> 120	-
42128-059	9.97 ppm PCNB +0.025 ppm PeCB & PCTA + 0.125 ppm HeCB +0.25 ppm PCA	108	104	112	116	106
42128-060	9.97 ppm PCNB +0.025 ppm PeCB & PCTA + 0.125 ppm HeCB +0.25 ppm PCA	107	104	113	115	106

Table 3. Summary of Turfgrass Method Validation Data for PCNB and Metabolites/Manufacturing By-Products

Sample	Sample Description	% PCNB Rec.	% PeCB Rec.	% HeCB Rec.	% PCA Rec.	% PCTA Rec.
42128-098	3750 ppm PCNB + 10 ppm Metabolites	118	110	> 120	> 120	110
42128-099	3750 ppm PCNB + 10 ppm Metabolites	108	100	> 120	> 120	105
42128-100	3750 ppm PCNB + 0.10 ppm Metabolites	108	110	> 120	> 120	103
42128-101	3750 ppm PCNB + 0.10 ppm Metabolites	107	110	> 120	> 120	104
42128-102	0.998 ppm PCNB + 0.10 ppm Metabolites	118	100	100	110	101
42128-103	0.998 ppm PCNB + 0.10 ppm Metabolites	112	100	101	109	102
42128-140	3750 ppm PCNB +100 ppm HeCB & PCA	101	-	100	99.3	-
42128-141	3750 ppm PCNB +100 ppm HeCB & PCA	96.0	-	94.8	93.5	-

Figure 1. Example Chromatogram of a 600 ng/mL PCNB Quantitation Standard

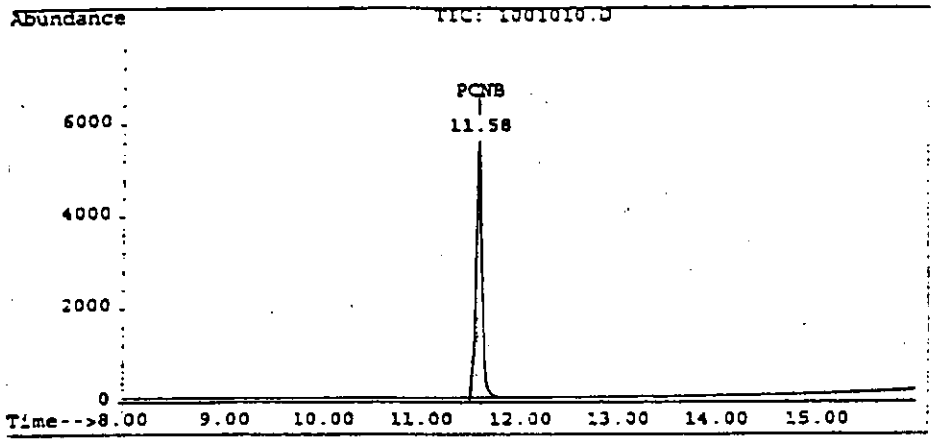


Figure 2. Example Chromatogram of a Mixed Metabolite Quantitation Standard Containing 125 ng/mL PeCB and PCTA and 500 ng/mL HeCB and PCA

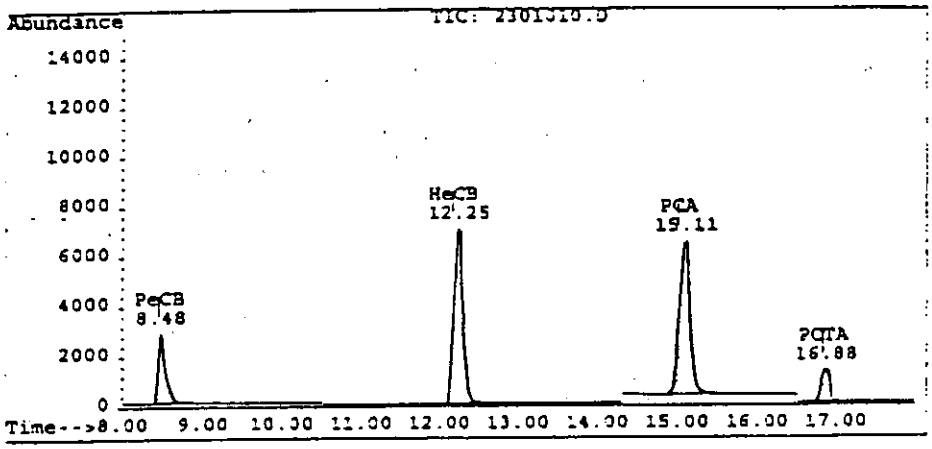
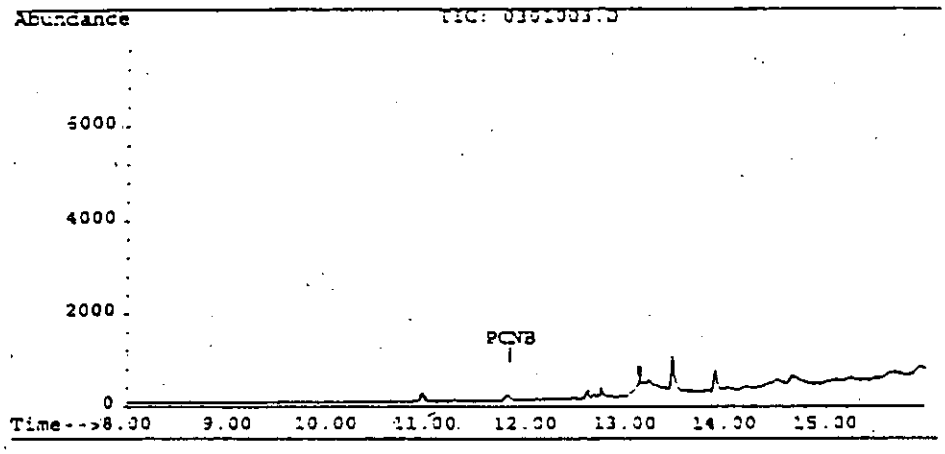
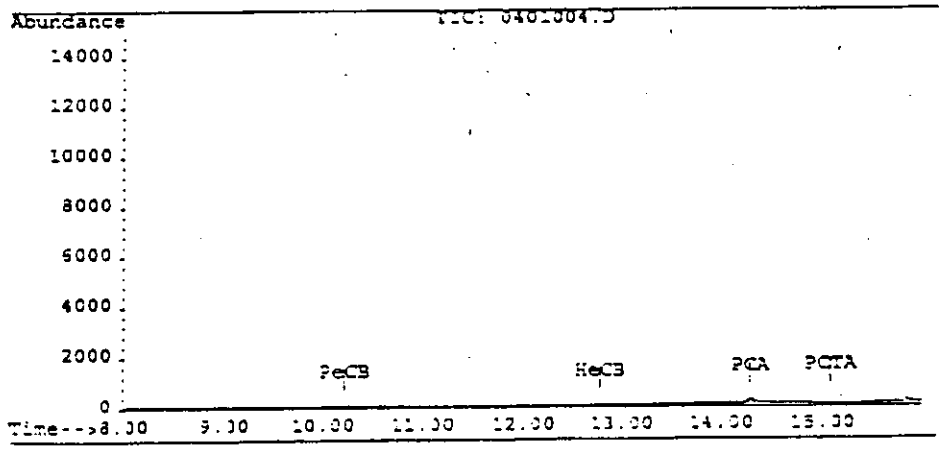


Figure 3. Example Chromatograms of Bareground Soil Control

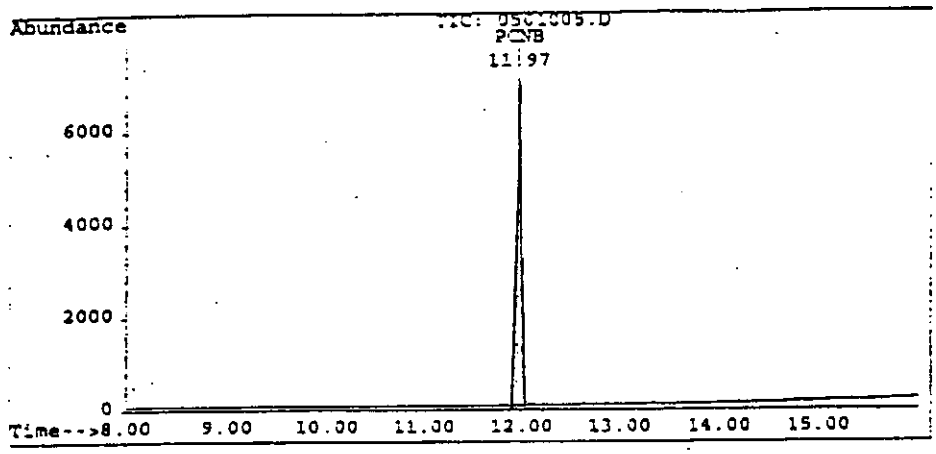


PCNB Analysis

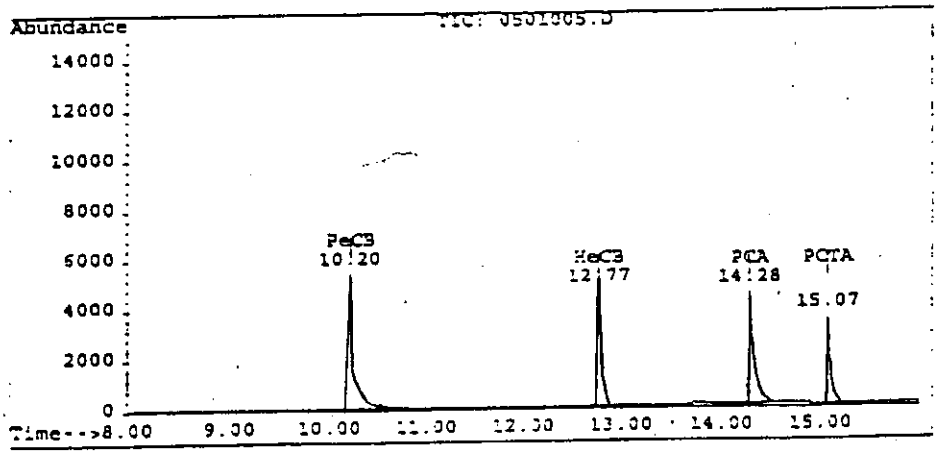


Metabolite Analysis

Figure 4. Example Chromatograms of Bareground Soil Fortifications at 9.97 ppm PCNB and 0.50 ppm Metabolites/Manufacturing By-Products

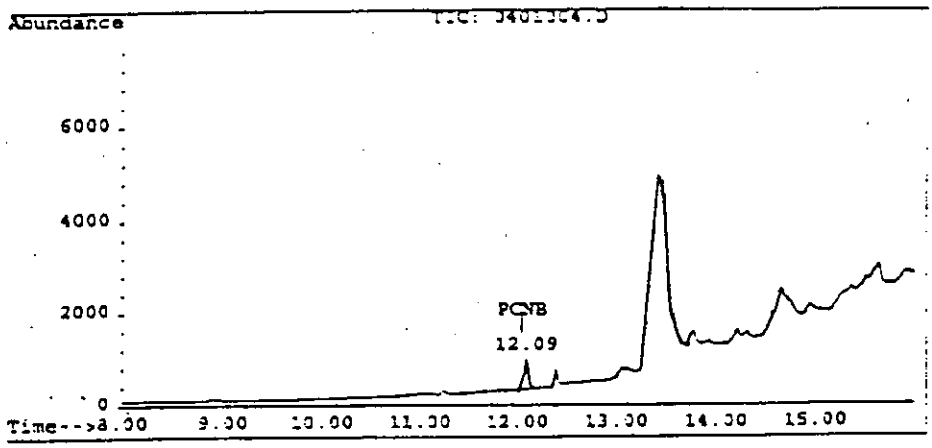


PCNB Analysis

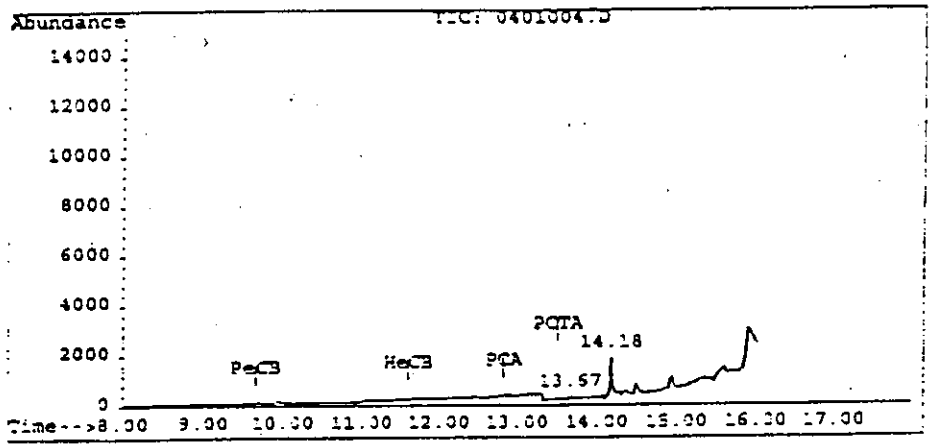


Metabolite Analysis

Figure 5. Example Chromatograms of Turfgrass Soil Control

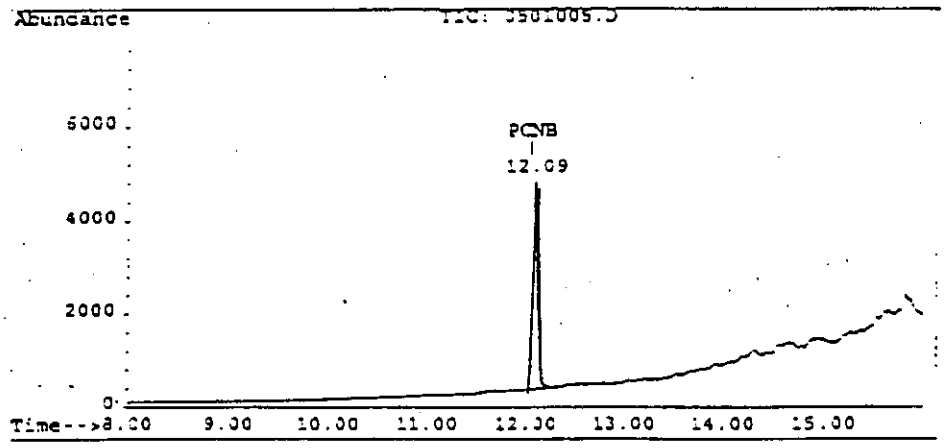


PCNB Analysis

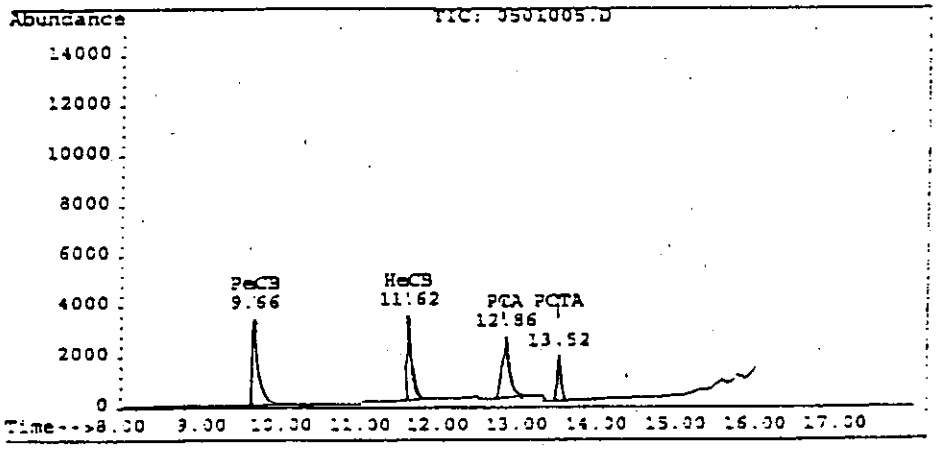


Metabolite Analysis

Figure 6. Example Chromatograms of Turfgrass Soil Fortifications at 9.97 ppm PCNB and 0.50 ppm Metabolites/Manufacturing By-Products

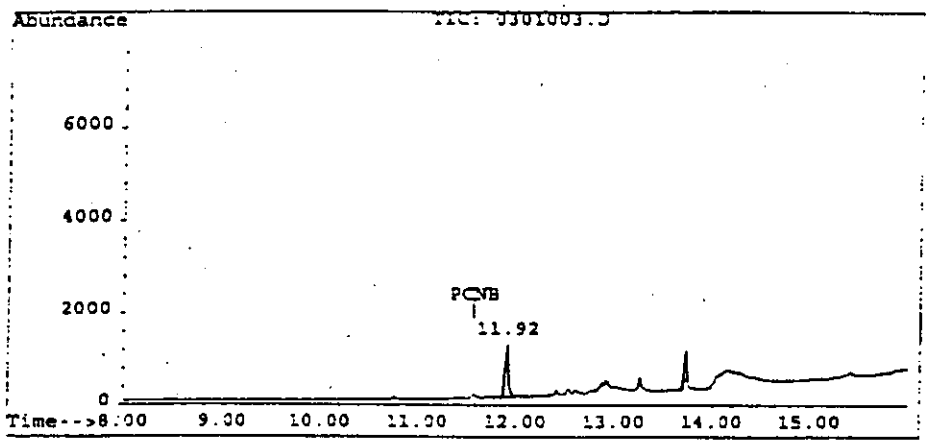


PCNB Analysis

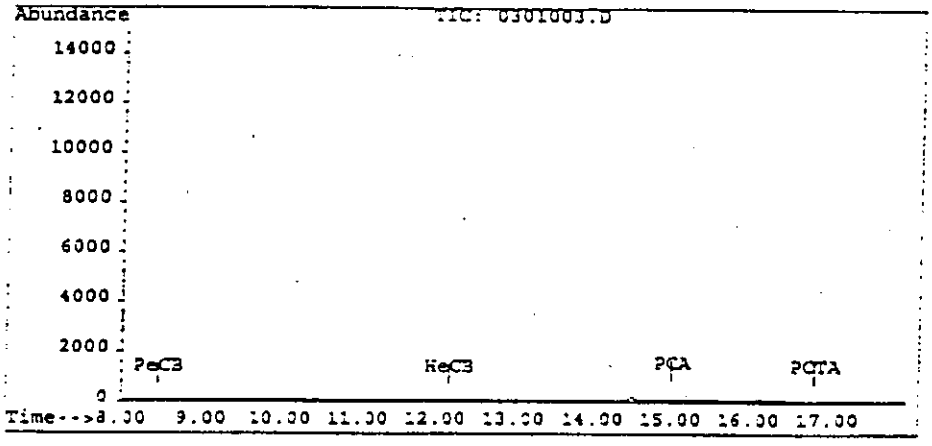


Metabolite Analysis

Figure 7. Example Chromatograms of Turfgrass Control

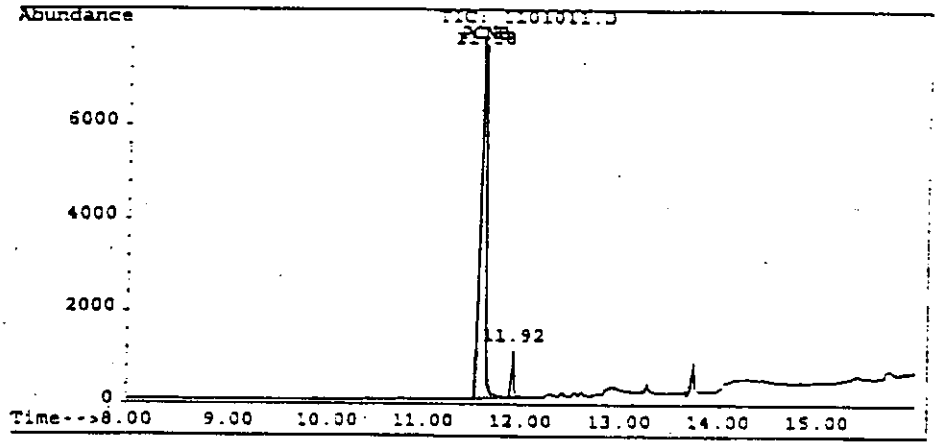


PCNB Analysis

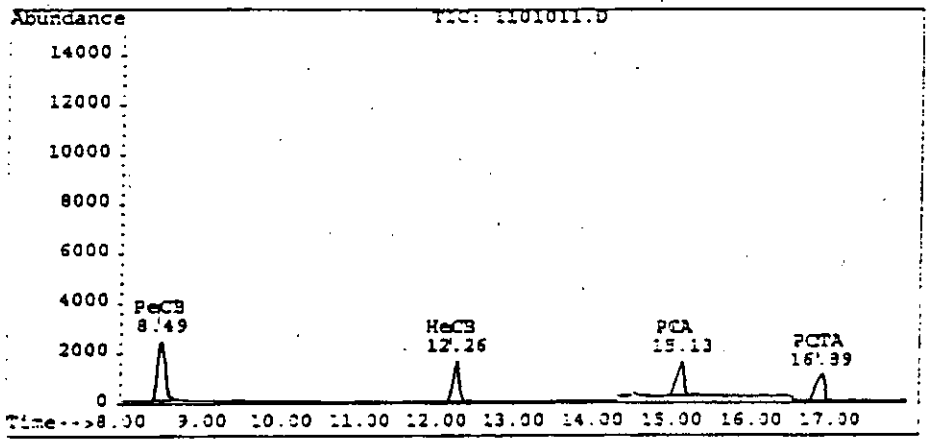


Metabolite Analysis

Figure 8. Example Chromatograms of Turfgrass Fortifications at 0.998 ppm PCNB and 0.10 ppm Metabolites/Manufacturing By-Products



PCNB Analysis



Metabolite Analysis