Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Quintozene (PCNB)

MRID #: 452286-01

Matrix: Water

Analysis: GC/ECD

This method is provided to you by the Environmental Protection Agency's (EPA) Environmental Chemistry Laboratory (ECL). This method *is not* an EPA method but one which was submitted to EPA by the pesticide manufacturer to support product registration. EPA recognizes that the methods may be of some utility to state, tribal, and local authorities, but makes no claim of validity by posting these methods. Although the Agency reviews *all* Environmental Chemistry Methods submitted in support of pesticide registration, the ECL evaluates only about 30% of the currently available methods. Most methods perform satisfactorily but some, particularly the older methods, have deficiencies. Moreover, the print quality of the methods varies considerably because the methods originate from different sources. Therefore, the methods offered represent the best available copies.

If you have difficulties in downloading the method, or further questions concerning the methods, you may contact Elizabeth Flynt at 228-688-2410 or via e-mail at flynt.elizabeth@epa.gov.

Volume 2

MRID# 45228601
QUINTOZENE

STUDY TITLE:

VALIDATION OF THE ANALYTICAL METHOD FOR THE DETERMINATION OF PENTACHLORONITROBENZENE (PCNB) AND PENTACHLOROANILINE (PCA) IN WATER

Data Requirement:
E.U. Commission Directive 96/46EC (16 July 1996)
U.S. EPA OPPTS 850.7100
And
U.S. EPA OPPTS 860.1340: Residue Analytical Methods

Study Authors:
Shari K. Long and Wadim Batorewicz
Study Completion Date: January 12, 2000

Sponsor and Performing Laboratory:
Uniroyal Chemical Company, Inc.
Crop Protection Division
Registration Chemistry Section
World Headquarters
Middlebury, CT 06749

Analytical Method No. AC-7003
Uniroyal Chemical Company Study No:
99198

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA Section 10(d)(1)(A), (B), or (C).

Company: Uniroyal Chemical Company, Inc.

Company Agent: _____ Date: 9-12-00
Signature

Willard F. Cummings
U.S. Registration Manager

These data are the property of the Uniroyal Chemical Company, Inc., and, as such, are considered to be confidential for all purposes other than compliance with FIFRA Section 10. Submission of these data in compliance with FIFRA does not constitute a waiver of any right to confidentiality which may exist under any statute or in any other country.

COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

This study was conducted in accordance with EPA Good Laboratory Practice Standards, 40 CFR 160 with the following exceptions:

Nonvalidated computer systems were used in the course of this study.

W. Batorewicz Date: 1/12/00

Wadim Batorewicz, Ph.D.
Study Director
Crop Protection R & D
Uniroyal Chemical Co., Inc.
Middlebury, CT 06749

Gerald J. Putterman Date: January 12, 2000

Gerald J. Putterman, Ph.D..
Sponsor Representative/Submitter/Applicant
Crop Protection R & D
Uniroyal Chemical Co., Inc.
Middlebury, CT 06749

Sponsor: Uniroyal Chemical Co., Inc.

QUALITY ASSURANCE STATEMENT

The study reported here, Analytical Method for Determining Pentachloronitrobenzene (PCNB) and Pentachloroaniline in Water, (Study No. 99198) was inspected/audited by the Quality Assurance Unit, and the findings were reported to the Study Director and Management on the following dates:

Date(s) Inspected/Audited	Study Phase	Date Reported to Management & Study Director
08/23/99	Protocol Review	08/23/99
09/29/99	0.1 ppb fortification of pond water,	10/04/99
12/17/99	Raw data/final report audit	12/17/99

Charles M. Ford

Date: 01/12/2000

Charles M. Ford, RQAP-GLP
Quality Assurance Auditor
Uniroyal Chemical Company, Inc.
Crop Protection R & D

CERTIFICATION OF AUTHENTICITY

I, the undersigned, hereby declare that this study was performed under my supervision according to the procedures described herein and that this report provides a true, unaltered, and accurate record of the results obtained.

W. Batorewicz

Date: 1/12/00

Wadim Batorewicz, Ph.D.
Study Director
Crop Protection R&D
Uniroyal Chemical Company, Inc.
Middlebury, CT 06749
203-573-3695

Approved By:

Gerald J. Putterman

Date: January 12, 2000

Gerald J. Putterman, Ph.D.
Sponsor Representative/Submitter/Applicant
Crop Protection R&D
Uniroyal Chemical Company, Inc.
Middlebury, CT 06749
203-573-3073

APPROVAL

Study No. 99198
Registration Chemistry Section
Crop Protection R&D
Uniroyal Chemical Company, Inc.

TITLE: Validation of the Analytical Method for the Determination of Pentachloronitrobenzene (PCNB) and Pentachloroaniline (PCA) in Water

STUDY INITIATION DATE: 8/30/99
EXPERIMENTAL START DATE: 8/30/99
EXPERIMENTAL TERMINATION DATE: 10/21/99
STUDY COMPLETION DATE: 01/12/00
NOTEBOOK: Loose leaf notebooks; three volumes

RAW DATA ARCHIVED AT AND MAY BE OBTAINED FROM CROP PROTECTION DIVISION ARCHIVES OF UNIROYAL CHEMICAL CO., INC., WORLD HEADQUARTERS, MIDDLEBURY, CT 06749, UPON REQUEST.

REPORT APPROVED BY:

W. Batorewicz DATE: 1/12/00
Wadim Batorewicz, Ph.D.
Study Director
Crop Protection R&D
Uniroyal Chemical Company, Inc.

Gerald J. Putterman DATE: January 12, 2000
Gerald J. Putterman, Ph.D.
Sponsor Representative/Submitter/Applicant
Crop Protection R&D
Uniroyal Chemical Company, Inc.

James B. Pierce DATE: Jan 12, 2000
James B. Pierce, Ph.D.
Manager, Registration Chemistry Section
Crop Protection R&D
Uniroyal Chemical Company, Inc.

PERSONNEL

Personnel participating in the conduct of this study are presented in the following list:

NAME	TITLE
Wadim Batorewicz, Ph.D.	Senior Research Scientist
Shari K. Long, M.S.	Development Chemist

TABLE OF CONTENTS

I. PRINCIPLE OF THE METHOD

The water sample is passed through a C-8 Speedisk™ under vacuum. The pentachloronitrobenzene (PCNB) and its metabolite pentachloroaniline (PCA) (which are retained on the disk) are eluted with toluene and concentrated under nitrogen. The analysis is by capillary GC with an electron capture detector. The quantitation is by external standard method, using a mixture of PCNB and PCA standards to generate the two calibration plots. The LOQ for the method is 0.1 ppb.

II. MATERIALS

A. EQUIPMENT

1. Analytical Evaporator, Model "N-Evap", Organomation Associates, Inc. The evaporator is connected to a house nitrogen line fitted with a filter and a pressure regulator, Balstone Filter Products
2. Balance, analytical, Model 1602MP, Sartorius
3. Vortex mixer, Fisher Scientific
4. Vacuum manifold for SPE disks, 12 port, Supelco
5. Vacuum pump, 1/6 HP, Model 0211-U45N-G8CX, Gast
6. Water purification system, Model Milli-Q, Millipore

Note: Equivalent equipment from other sources can be employed.

B. SUPPLIES

1. Assorted volumetric disposable pipettes, Fisher Scientific
2. Customary analytical laboratory glassware and supplies
3. Filtration suction flask, 0.5L, Fisher Scientific
4. Kolmer evaporation tubes, 10 mL graduated, Fisher Scientific
5. C-8, 50 mm, solid phase extraction disks and reservoirs, Bakerbond Speedisk™, J.T.Baker

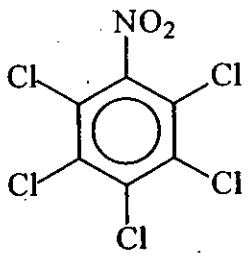
Note: Equivalent supplies from other sources can be employed. It is recommended, however, that SPE disks from J.T. Baker be used.

C. HPLC SOLVENTS

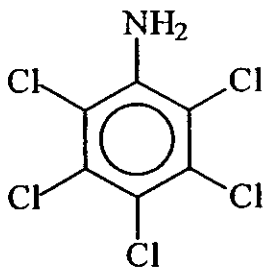
1. Methanol, Fisher Scientific
2. Toluene, Fisher Scientific
3. Water, Milli-Q

Note: HPLC solvents of equivalent purity from other sources can be used.

D. STANDARDS-TEST SUBSTANCES



Chemical name: Pentachloronitrobenzene
Common name: PCNB
CAS number: 82-68-8
Uniroyal Chemical Company Inc. Analytical Standard Code: P-01
Uniroyal Chemical Company Inc. Analytical Standard Lot No.: AC-1398-107
Chemical Purity: 99.8%
Molecular Weight: 295.33
Storage Conditions: Freezer



Chemical Name: Pentachloroaniline
Common Name: None
CAS number: 527-20-8
Uniroyal Chemical Company Inc. Analytical Standard Code: P-02
Uniroyal Chemical Company Inc. Analytical Standard Lot No.: AC-1398-93C
Chemical Purity: 98.7%
Molecular Weight: 265.35
Storage Conditions: Freezer

Standards are archived by the Analytical Services Group of the Crop Protection Division.

E. GC INSTRUMENTATION

E-1 Residue Analysis by GC/Electron Capture Detector

Gas chromatograph: Model HP 6890 equipped with an electron capture detector and an autosampler-injector, Model HP 6763. Instrument parameters and data capture are controlled by a computer with a HP 3365 ChemStation on Windows 95 platform.

Column: DB1701, 30 m X 0.25 mm, 0.05 µm film thickness, J & W Scientific
Carrier Gas: Helium
Makeup Gas: Nitrogen

E-2 Confirmatory Analysis by GC/MSD

Gas chromatograph: Model HP 5890A equipped with a HP Model 5971A mass selective detector and an autosampler-injector, Model HP 6890. Instrument parameters and data capture are controlled by HP Chemstation Software G-1701AA, Rev.A.03.00.

Column: Rtx 200, 30 m X 0.25 mm, 0.05 µm film thickness, Restek Corporation
Carrier Gas: Helium

F. SAFETY AND HEALTH

This method should be performed by trained chemical personnel. Hazards associated with the chemicals used in this analytical method are found in their respective Material Safety Data Sheets (MSDS).

III. ANALYTICAL METHOD

A. WATER SOURCE AND CHARACTERIZATION

This method should be applicable for water from various sources. Pond water from Preston Hill Pond, Middlebury, CT was collected for use in this study. The pond water is characterized by its dissolved organic carbon, pH, and total hardness. The levels of dissolved organic carbon, the pH, and the total hardness of the pond water are shown in Appendix A.

B. PREPARATION OF THE STANDARD SOLUTIONS

B-1a. PCNB Stock Solution

On an analytical balance, accurately weigh approximately 50 mg of PCNB standard into a 50 mL volumetric flask and dilute to volume with toluene. Sonicate the flask. Thus, the concentration of the stock solution is 1 mg/mL. Correct the concentration for the purity of the standard: mg/mL X (percent purity/100).

B-1b. PCA Stock Solution

On an analytical balance, accurately weigh approximately 50 mg of PCA standard into a 50 mL volumetric flask and dilute to volume with toluene. Sonicate the flask. Thus, the concentration of the stock solution is 1 mg/mL. Correct the concentration for the purity of the standard: mg/mL X (percent purity/100).

B-2a. PCNB Standard Working Solution

Pipette 0.5 mL of PCNB Stock solution B-1a into a 50 mL volumetric flask and dilute to volume with toluene. Thus, the concentration of the solution is 10 µg/mL.

B-2b. PCA Standard Working Solution

Pipette 0.5 mL of PCA Stock solution B-1b into a 50 mL volumetric flask and dilute to volume with toluene. Thus, the concentration of the solution is 10 µg/mL.

B-3a. PCNB Fortification Solution for 1 ppb Recoveries

Pipette 1.0 mL of PCNB working solution B-2a into a 10 mL volumetric flask and dilute to volume with methanol. Thus, the concentration of the solution is 1.0 µg/mL.

B-3b. PCA Fortification Solution for 1 ppb Recoveries

Pipette 1.0 mL of PCA working solution B-2b into a 10 mL volumetric flask and dilute to volume with methanol. Thus, the concentration of the solution is 1.0 µg/mL.

B-3c. PCNB Fortification Solution for 0.1 ppb Recoveries

Pipette 1.0 mL of PCNB working solution B-2a into a 10 mL volumetric flask and dilute to volume with methanol. Pipette 1 mL of this 1.0 µg/mL

solution into a 10 mL volumetric flask and dilute to volume with methanol. Thus, the concentration of the final solution is 0.1 µg/mL.

B-3d. PCA Fortification Solution for 0.1 ppb Recoveries

Pipette 1.0 mL of PCA working solution B-2b into a 10 mL volumetric flask and dilute to volume with methanol. Pipette 1 mL of the 1.0 µg/mL solution into a 10 mL volumetric flask and dilute to volume with methanol. Thus, the concentration of the final solution is 0.1 µg/ml.

B-4a. Calibration Plot for PCNB and PCA in the 1.0 ppb Range

Into five 10 mL volumetric flasks, pipette 1.0, 0.75, 0.5, 0.25, and 0.1 mL of PCNB working solution B-2a and PCA working solution C-2b. Dilute to volume with toluene. Thus, the concentration of the resulting standard solutions are 1.0, 0.75, 0.5, 0.25, and 0.1 µg/mL for PCNB and PCA.

B-4b. Calibration Plot for PCNB and PCA in the 0.1 ppb Range

Pipette 1 mL of PCNB standard working solution B-2a into a 10 mL flask and bring to volume with toluene. Pipette 1 mL of PCA standard working solution B-2b into a 10 mL flask and bring to volume with toluene. Into five 10 mL volumetric flasks, pipette 1.0, 0.75, 0.5, 0.25 and 0.1 mL of each of the above PCNB and PCA solutions. Thus, the concentration of the resulting standard solutions are 0.1, 0.075, 0.05, 0.025, and 0.01 µg/mL for PCNB and PCA..

C. FORTIFICATION OF WATER SAMPLES

C-1 Fortification with PCNB and PCA at the 1.0 ppb level

Measure 500 mL of pond water using a 500 mL graduated cylinder. Decant the sample into a l L glass bottle. Using a 500 µL syringe, add 500 µL each of fortification solutions B-3a and B-3b. Cap the bottle and mix well by shaking.

C-2 Fortification with PCNB and PCA at the 0.1 ppb level

Measure 500 mL of pond water using a 500 mL graduated cylinder. Decant the sample into a 1 L glass bottle. Using a 500 µL syringe, add 500 µL each of fortification solutions B-3c and B-3d.

D. EXTRACTION PROCEDURE

1. Place a C-8 Speedisk™ onto a 500 mL vacuum flask attached to a vacuum pump. Pass 10 mL of toluene through the disk under vacuum and continue to pull the vacuum until the disk is dry.
2. Condition the disk with 10 mL of methanol. Allow the methanol to flow through slowly by gravity. Do not allow the disk to dry.
3. When 3-5 mL of methanol remains on the disk, add 10 ml of HPLC grade water.
4. When 3-5 mL of water remains on the disk, begin adding the 500 mL water sample under full vacuum (15 mm Hg). Leave the disk on full vacuum for 5 minutes after the water has passed through the disk. Then release the vacuum.
5. Transfer the disk to a solid phase extraction manifold and continue drying under full vacuum (15 mm Hg) for an additional 30 minutes.
6. With the manifold port now closed, place a 10 mL Kolmer evaporation tube under the port. Add 10 mL of toluene to the disk. Allow the toluene to soak into the disk for 10 minutes, then open the manifold port so the toluene can be eluted by gravity. Apply slight vacuum to elute any remaining analyte.
7. Concentrate the eluant to 1 mL under a gentle stream of nitrogen at about 50°C . Transfer to an autosampler vial for GC-ECD analysis.

E. CONDITIONS FOR GC-ECD ANALYSIS

Injector Temperature:	250°C
Injector Mode:	Splitless
Detector Temperature:	325°C

Oven Temperature Program:

Initial Temperature:	100°C	
Initial Time:	2.0 minutes	
Ramp: (°C/min)	Final Temp (°C)	Final Time (min.)
30.0	225	5.0
15.0	280	1.0

Injection Volume:	1.0 µL
Retention times:	PCA 7.1 min., PCNB 6.5 min.
Run Time:	15.8 minutes
Combined gas flow:	68.3 mL/min. for injector

Injection Sequence:

a. Start the sequence by injecting the solvent blank (toluene), then the standards, starting with the lowest.
b. Inject a solvent blank then a reagent blank followed by another solvent blank.
c. Inject pond water controls, then a solvent blank.
d. Inject the fortified pond water samples, then a solvent blank.
e. Complete by injecting the standards, starting with the lowest.

Note 1: Reagent blank is 500 mL distilled water which is brought through the entire extraction and concentration procedure identically to the samples.
Note 2: The injection sequence was modified after analyzing the 1 ppb pond water samples. PCA and PCNB standards were combined to reduce the total number of injections.

F. CONFIRMATORY TECHNIQUE

F-1 Method of Confirmation

The method for the confirmation of PCNB and PCA was by GC/MSD. Confirmation was made by comparison of the retention times and mass spectra of the peaks in the extracts from both 1.0 ppb and 0.1 ppb fortified pond water samples with the spectra of the standards.

F-2 Conditions for GC/MSD Analysis

Column head pressure:	10 psi
Injector Temperature:	250°C
Injection Volume:	1.0 µL
Oven Temperature Program:	
Initial Temperature:	100°C
Initial Time:	1.0 min.

Ramp: (°C/min.)	Final Temp.(°C)	Final Time(min.)
12.0	280	6.0

Run Time:	22.0 min.
Equilibration Time:	3.0 min.
MSD Operation Parameters:	
Transfer Line:	280°C
Electron Multiplier Range:	~2000 mV
+eV above Autotune:	0
SIM Settings:	
Group III, PCA	m/z 265
Group II, PCNB	m/z 237, 295
Resolution:	Low
Dwell Time :	50
Solvent Delay (min.):	4.0

G. TIME REQUIRED FOR ANALYSIS

A sample set consists of a reagent blank, two control pond water samples, and five fortified pond water samples. The time required to complete the extraction and concentration of a mixture of PCNB and PCA from pond water is about six

hours. The GC/ECD analysis is carried out overnight using an autosampler injector. The data are processed using Microsoft Excel, version 97, software.

H. CALCULATIONS

H-1 Calibration Plot

Peak areas of the standards (PCA or PCNB) are the dependent variables and the concentrations of the standard solutions, expressed as µg/mL, are the independent variables. They are used to generate a linear regression equation to determine the intercept, the slope, and the linearity of the detector response (coefficient of determination) R^2.

$$Peak\ Area = Intercept + Slope\ X\ (\mu g / mL)_{Std.}$$

H-2 Calculate the amount of PCNB or PCA in the toluene extract

Using the peak area of PCNB or PCA found in the extract, determine the concentration using the following equation:

$$PCNB(\mu g / mL) = \frac{(Peak\ Area_{\ sample} - Intercept)}{Slope}$$

H-3 Calculate the amount (µg) of PCNB and PCA found in the sample

Divide the amount of analyte calculated in section H-2 by the analyte volume, expressed in mL. The analyte volume used in this validation is 1 mL.

$$\mu g = \mu g / mL\ X\ Analyte\ Volume(mL)$$

H-4 Calculate the % Recovery of PCA and PCNB from fortified pond water

Divide the value calculated in section H-3, which is the amount of analyte found in the extract, by the amount of analyte added to the fortified pond water and multiply this value by 100.

$$\%\,Recovery = \frac{\mu g\ Found}{\mu g\ Added} X\,100$$

Example of the calculation method:

A water sample was fortified at the 0.1 ppb level with PCNB and PCA and analyzed on 11/01/99, sample PW#3, sample ID sl110115 (Appendix B).

0.05 µg of PCA and 0.051 µg PCNB were added to a 500 mL sample. PW3 peak area was: 11705 for PCA.

From the PCA linear regression analysis: Intercept =-17.2561 and Slope=326799.2

The µg/mL PCA in the analyte was calculated using the equation:

$$Found, \mu g / mL = \frac{(Peak\ Area - Intercept)}{Slope}$$

{11705-(-17.2561)}/326799.2=.03587 µg/mL

The amount (µg) of PCA in the analyte was calculated as:

Volume of the analyte is 1 mL, therefore:

0.03587 µg/mL X 1 mL=0.03587 µg

The amount (µg) of PCA expected in the 1 mL of analyte:

0.05 µg of PCA was added to a 500 mL sample with a final volume of 1 mL, therefore the expected amount is 0.05 µg.

% Recovery:

$$\frac{\mu g\ found}{\mu g\ added} X\ 100 = \%\,Rec.$$

(0.03587/0.05) X 100=71.74% Recovery

III. RESULTS AND DISCUSSION

A. RESULTS OF METHOD VALIDATION

The study was conducted according to the protocol given in Appendix C. The analytical method for determining PCNB and PCA was validated at 1.0 ppb and 0.1 ppb levels for accuracy, precision, linearity, and specificity. Five samples of Preston Hill Pond water were fortified at each level, extracted and analyzed by GC-ECD. The experiment was repeated on a subsequent day for each level. Mean recoveries were all within the 70-110% range, the relative standard

deviations (RSD) were ≤20%. Linearity of detector response was determined for each level using a five point calibration curve to obtain a coefficient of determination (R^2) for PCNB and PCA. The coefficients of determination were ≥ 0.99.

B. ACCURACY/RECOVERY

The accuracy of the method is expressed as % recovery of PCNB and PCA added to pond water. Mean recoveries for each set of samples were 70 – 110%, with relative standard deviation ≤20%. Specifically, the mean recovery of PCA at 1.0 ppb was 87.5% with a RSTD of ±5.68 % (n = 10) The mean recovery of PCA at 0.1 ppb was 71.2% with a RSTD of ±6.43 % (n =10). The mean recovery of PCNB at 1.0 ppb was 81.7% with a RSTD of ±8.58% (n =10), and the mean recovery of PCNB at 0.1 ppb was 75.4% with a RSTD of ±9.47% (n = 10). Data for the 1.0 ppb and 0.1 ppb fortification levels for PCA are summarized in Tables I and II respectively. Data for the 1.0 ppb and 0.1 ppb fortification levels for PCNB are summarized in Tables III and IV respectively.

Typical calibration plots for quantitation of pond water samples fortified at 0.1 ppb for PCA and PCNB are shown in Figures 1 and 2, respectively. Representative chromatograms of PCA and PCNB calibration standards for the lowest point (0.01 µg/mL for both), the mid point (0.05 µg/mL and 0.051 µg/mL, respectively), and the highest point (0.1 µg/mL and 0.102 µg/mL, respectively), are shown in Figures 3,4, and 5, respectively.

Typical calibration plots for quantitation of pond water samples fortified at 1 ppb for PCA and PCNB are shown in Figures 6 and 7, respectively. Representative chromatograms of PCA and PCNB calibration standards for the lowest point (0.1 µg/mL and 0.102 µg/mL, respectively), the mid-point (0.5 µg/mL and 0.51 µg/mL, respectively), and the highest point (1.0 µg/mL and 1.02 µg/mL, respectively) are shown in Figures 8,9,10,11,12, and 13, respectively.

Representative chromatograms of a reagent blank, pond water control, and pond water sample spiked at 0.1 ppb with PCA and PCNB are shown in Figures 14,15, and 16, respectively. Representative chromatograms of a reagent blank, pond water control, and pond water sample spiked at 1.0 ppb with PCA and PCNB are shown in Figures 17,18, and 19, respectively.

C. PRECISION

The precision of the method was determined from ten consecutive injections of a fortified sample of pond water. Precision was expressed as the RSTD of the observed set of measurements. The RSTD was ±3.0% for PCNB and ±3.3% for PCA. The data are summarized in Table V.

D. LIMIT OF QUANTITATION (LOQ) AND LIMIT OF DETECTION (LOD)

The LOQ was targeted at 0.1 ppb, which was the lowest fortification level employed. The method LOD is set at one third of the LOQ (0.1 ppb), which is 0.03 ppb (*Anal. Chem.* 1999, 2672-2677).

E. SELECTIVITY

The selectivity of the method is shown by baseline to baseline resolution of PCNB (retention time 6.5 minutes) and PCA (retention time 7.1 minutes), using GC-ECD.

F. SPECIFICITY

The specificity of the method for each analyte was determined by GC/MSD using selected ions monitoring method. Extracts of previously analyzed water samples fortified at 1.0 ppb and 0.1 ppb were analyzed directly on the system described in section III F. Figure 20 shows a mass spectrum of PCNB. The molecular ion m/z 295 and a base ion m/z 237 were used for selected ion monitoring. Figure 21 shows a mass spectrum of PCA. Only the molecular ion m/z 265 was used for selected ion monitoring because there were no other ions of sufficient abundance to be useful for identification of PCA. Selected ion chromatograms and mass spectra of the pond water sample containing PCNB and PCA (figures 22 and 23, respectively) correspond to the standard chromatogram and mass spectra of PCNB and PCA.

IV CONCLUSIONS

This analytical method is applicable for the analysis of PCNB and PCA in all types of water samples in the 0.1 ppb to 1.0 ppb range. The method meets all of the requirements of the EPA and EU guidelines for analytical methods for water analysis.

TABLE I. PCA Recovery at 1.0 ppb Fortification Level

Sample ID	Fortification, ppb	Sample Name	% Recovery
Sl091008 & 31	None	Reagent blank	None
Sl091010 & 33	None	Control#1	None
Sl091011 & 34	None	Control#2	None
Sl091013 & 36	1.0	*PW#1	89.9
Sl091014 & 37	1.0	*PW#2	85.7
Sl091015 & 38	1.0	*PW#3	86.9
Sl091016 & 39	1.0	*PW#4	82.0
Sl091017 & 40	1.0	*PW#5	81.0
Sl092208 & 31	None	Reagent blank	None
Sl092210 & 33	None	Control#1	None
Sl092211 & 34	None	Control#2	None
Sl092213 & 36	1.0	*PW#1	92.2
Sl092214 & 37	1.0	*PW#2	90.8
Sl092215 & 38	1.0	*PW#3	83.9
Sl092216 & 39	1.0	*PW#4	85.4
Sl092217 & 40	1.0	*PW#5	97.0
		Average=	87.48
		STD=	4.971653
		RSTD%=	5.683188

TABLE II. PCA Recovery at 0.1 ppb Fortification Level

Sample ID	Fortification, ppb	Sample Name	% Recovery
Sl110108	None	Reagent Blank	None
Sl110110	None	Control#1	None
Sl110111	None	Control#2	None
Sl110113	0.1	*PW#1	68.0
Sl110114	0.1	*PW#2	68.3
Sl110115	0.1	*PW#3	71.7
Sl110116	0.1	*PW#4	71.1
Sl110117	0.1	*PW#5	71.1
Sl110808	None	Reagent blank	None
Sl110810	None	Control#1	None
Sl110811	None	Control#2	None
Sl110813	0.1	*PW#1	73.4
Sl110814	0.1	*PW#2	77.0
Sl110815	0.1	*PW#3	77.5
Sl110816	0.1	*PW#4	61.7
Sl110817	0.1	*PW#5	72.3
		Average=	71.21
		STD=	4.577105
		RSTD%=	6.427615

TABLE III. PCNB Recovery at 1.0 ppb Fortification Level

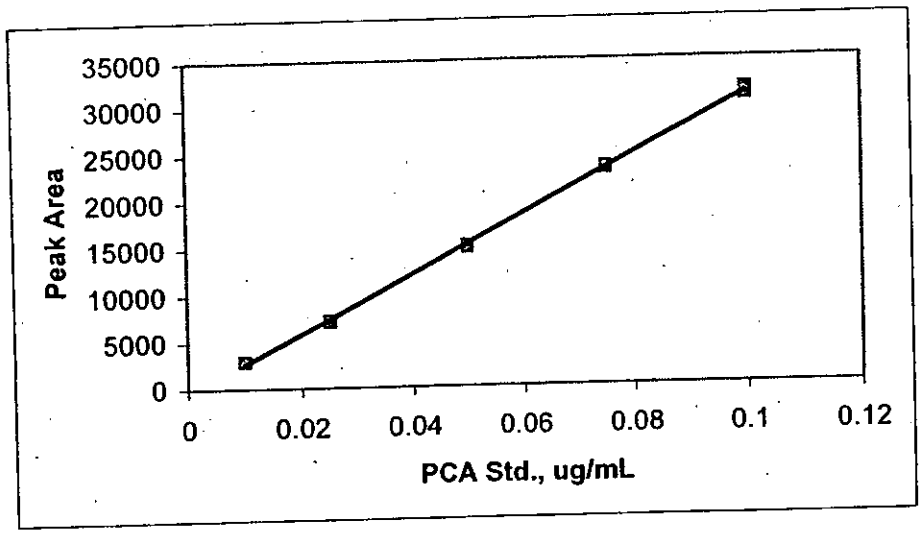
Sample ID	Fortification, ppb	Sample Name	% Recovery
Sl091008 & 31	None	Reagent Blank	None
Sl091010 & 33	None	Control#1	None
Sl091011 & 34	None	Control#2	None
Sl091013 & 36	1.0	*PW#1	80.4
Sl091014 & 37	1.0	*PW#2	78.5
Sl091015 & 38	1.0	*PW#3	77.8
Sl091016 & 39	1.0	*PW#4	72.6
Sl091017 & 40	1.0	*PW#5	72.5
Sl092208 & 31	None	Reagent Blank	None
Sl092210 & 33	None	Control#1	None
Sl092211 & 34	None	Control#2	None
Sl092213 & 36	1.0	*PW#1	88.6
Sl092214 & 37	1.0	*PW#2	88.1
Sl092215 & 38	1.0	*PW#3	81.8
Sl092216 & 39	1.0	*PW#4	82.8
Sl092217 & 40	1.0	*PW#5	94.3
		Average =	81.74
		STD=	7.016837
		RSTD%=	8.584337

TABLE IV. PCNB Recovery at 0.1 ppb Fortification Level

Sample ID	Fortification, ppb	Sample Name	% Recovery
Sl110108	None	Reagent Blank	None
Sl110110	None	Control#1	None
Sl110111	None	Control#2	None
Sl110113	0.1	*PW#1	70.2
Sl110114	0.1	*PW#2	71.7
Sl110115	0.1	*PW#3	71.0
Sl110116	0.1	*PW#4	73.0
Sl110117	0.1	*PW#5	70.2
Sl110108	None	Reagent Blank	None
Sl110810	None	Control#1	None
Sl110811	None	Control#2	None
Sl110813	0.1	*PW#1	72.7
Sl110814	0.1	*PW#2	84.4
Sl110815	0.1	*PW#3	91.2
Sl110816	0.1	*PW#4	71.2
Sl110817	0.1	*PW#4	78.7
		Average =	75.43
		STD =	7.144081
		RSTD=	9.47114

TABLE V. PRECISION: Ten Consecutive Injections of a Fortified Sample of Pond Water

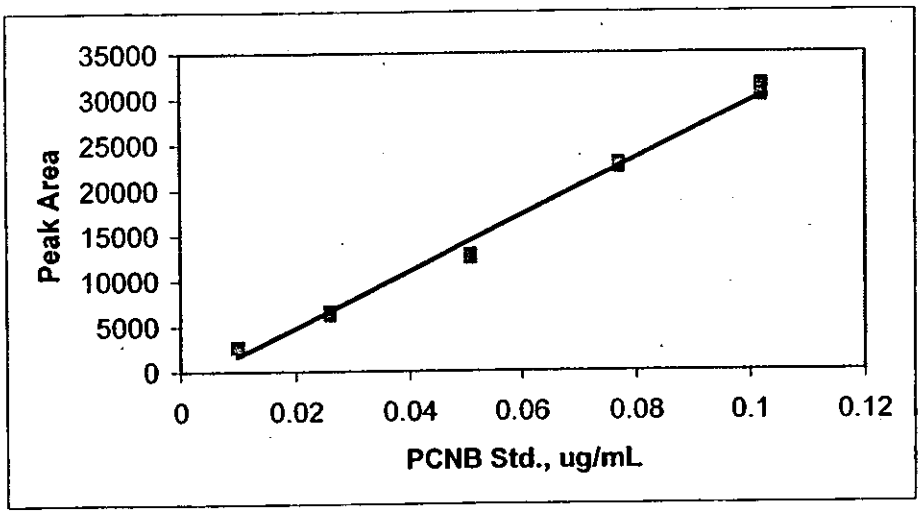
Run #	PCNB Area Counts	PCA Area Counts
1	11219	10759
2	11804	10747
3	11816	10274
4	11530	11238
5	11023	10585
6	11909	10488
7	11025	10493
8	11722	10052
9	11202	10224
10	11197	10244
Ave.=	11444.7	**Ave.=** 10510.4
STD=	348.3109	**STD=** 345.2153
RSTD=	3.043425	**RSTD=** 3.284511



PCA ST ug/mL	Pk.Area
0.01	2914
0.01	2894
0.025	7273
0.025	7115
0.05	15102
0.05	14945
0.075	23420
0.075	23226
0.1	31609
0.1	30961

Slope = 317421.4
Int. = -560.012
RSQ = 0.999362

Figure 1. Representative Calibration Plot for PCA Employed for Pond Water Samples Fortified at 0.1 ppb.

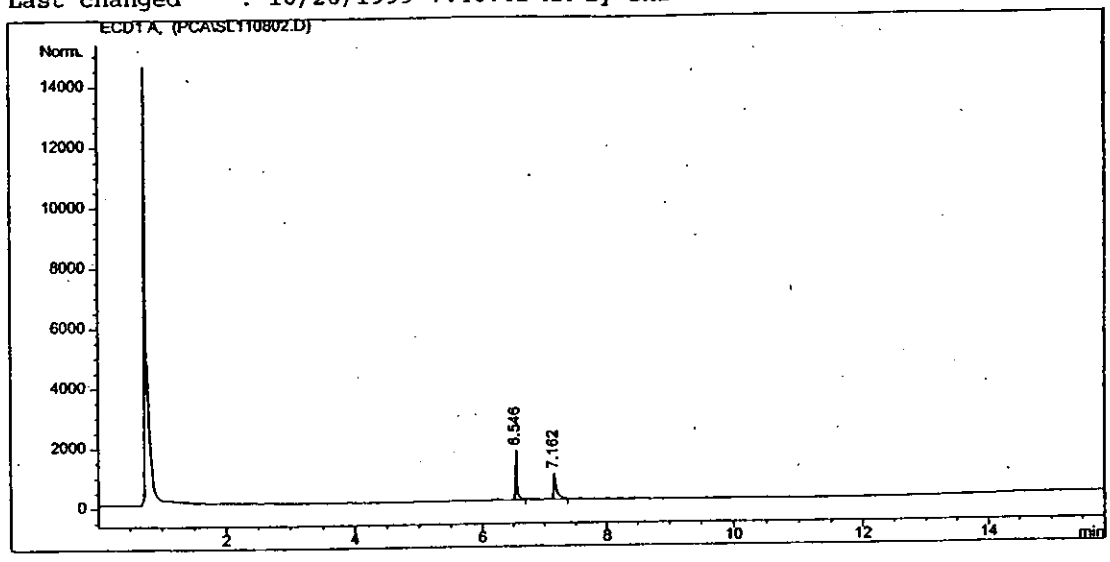


PCNB ST ug/mL	Pk.Area
0.01	2707
0.01	2739
0.026	6655
0.026	6309
0.051	12908
0.051	12517
0.077	22914
0.077	22436
0.102	31488
0.102	30226

Slope = 310062.4
Int. = -1405.42
RSQ = 0.990857

Figure 2. Representative Calibration Plot for PCNB Employed for Pond Water Samples Fortified at 0.1 ppb.

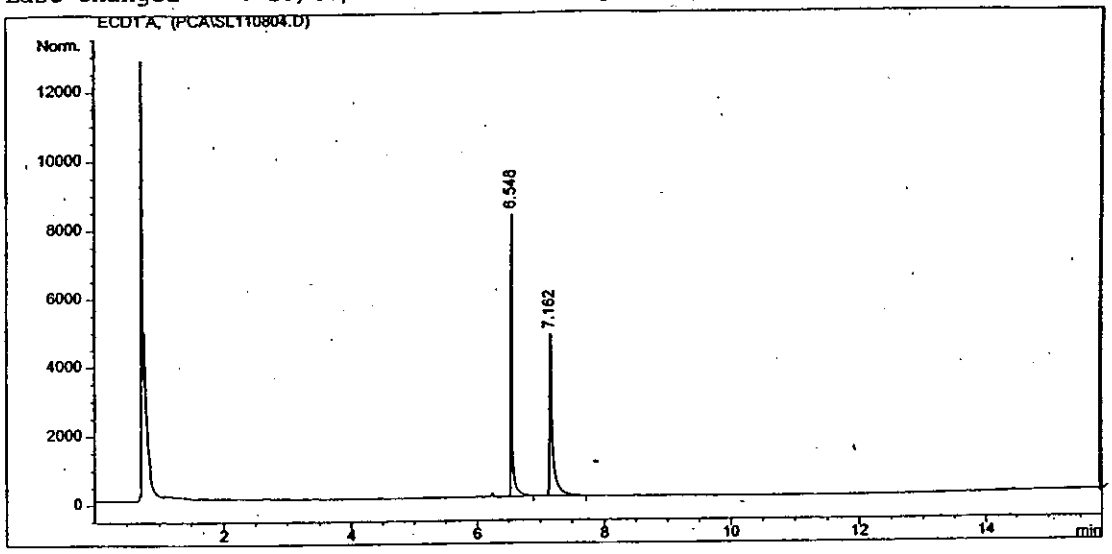
```
Batch Run # 2 of 12
Data File C:\HPCHEM\2\DATA\PCA\sl110802.D                    Sample Name: std#1

   pca 0.01ug/ml
   pcnb 0.0102ug/ml

================================================================================
Injection Date  : 11/8/1999 1:30:12 PM          Seq. Line :   2
Sample Name     : std#1                              Vial :   2
Acq. Operator   : skl                                 Inj :   1
                                                Inj Volume : 1 µl

Method          : C:\HPCHEM\2\METHODS\PCA.M
Last changed    : 10/26/1999 7:46:41 AM by skl
```




```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: ECD1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [Hz*s]     [Hz]       %
----|-------|----|-------|----------|----------|--------|
   1   6.546 VV    0.0234 2707.50537 1645.27332 48.16073
   2   7.162 BV    0.0456 2914.30591  854.18463 51.83927
```

Figure 3. Representative Chromatogram of PCA and PCNB standards at the 0.01 µg/mL level (lowest point). The retention time of PCA is 7.1 minutes, and the retention time of PCNB is 6.5 minutes.

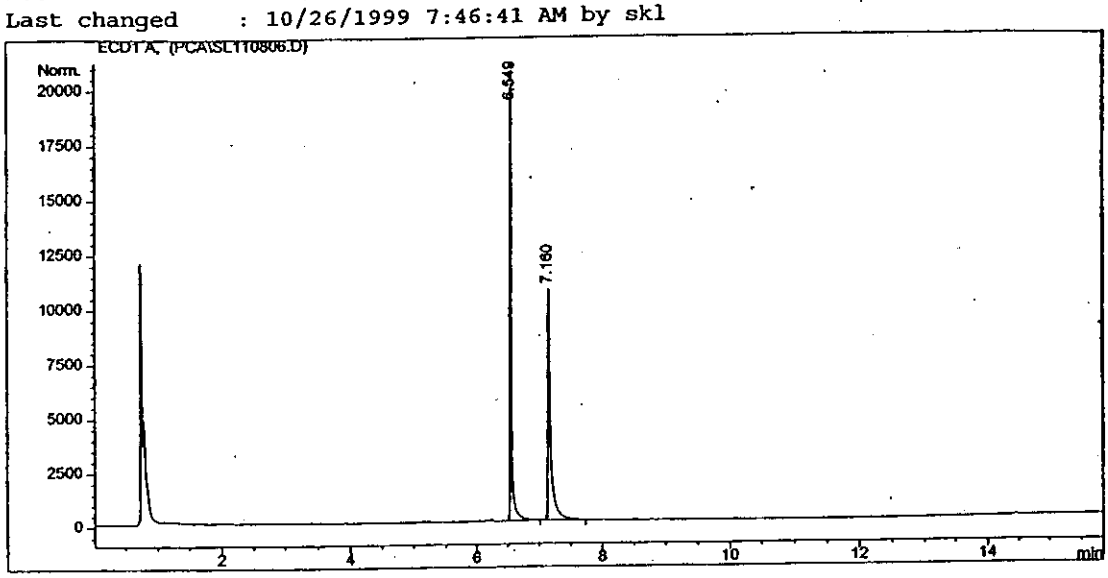
```
Batch Run # 4 of 12
Data File C:\HPCHEM\2\DATA\PCA\sl110804.D                    Sample Name: std#3

    pca 0.05ug/ml
    pcnb 0.051ug/ml

================================================================================
Injection Date  : 11/8/1999 2:16:39 PM          Seq. Line :   4
Sample Name     : std#3                            Vial :   4
Acq. Operator   : skl                               Inj :   1
                                                Inj Volume : 1 µl

Method          : C:\HPCHEM\2\METHODS\PCA.M
Last changed    : 10/26/1999 7:46:41 AM by skl
```




```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: ECD1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]    [Hz*s]      [Hz]       %
----|-------|----|-------|----------|----------|--------|
   1   6.548 VB S  0.0228 1.29083e4  8224.30859 46.08378
   2   7.162 PB S  0.0434 1.51022e4  4713.39355 53.91622
```

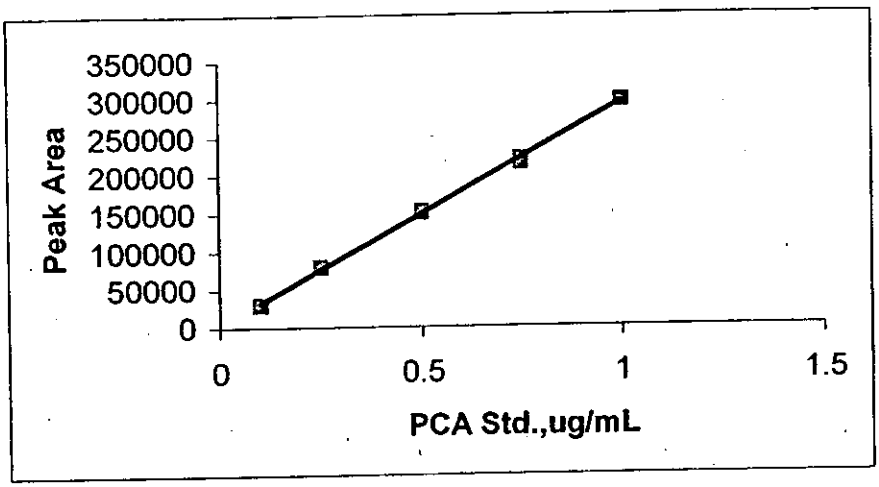
Figure 4. Representative Chromatogram of PCA and PCNB standards at 0.05 and 0.051 µg/mL, respectively. The retention time of PCA is 7.1 minutes and the retention time of PCNB is 6.5 minutes.

```
Batch Run # 6 of 12
Data File C:\HPCHEM\2\DATA\PCA\sl110806.D                    Sample Name: std#5

    pca 0.1ug/ml
    pcnb 0.102ug/ml

================================================================================
Injection Date  : 11/8/1999 3:03:03 PM          Seq. Line :   6
Sample Name     : std#5                             Vial :   6
Acq. Operator   : skl                               Inj :   1
                                                Inj Volume : 1 µl

Method          : C:\HPCHEM\2\METHODS\PCA.M
Last changed    : 10/26/1999 7:46:41 AM by skl
```




```
================================================================================
                              Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: ECD1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [Hz*s]      [Hz]       %
----|-------|----|-------|----------|----------|--------|
   1   6.549 VB S  0.0225 3.14885e4  2.01073e4  49.90450
   2   7.160 VB S  0.0403 3.16090e4  1.06640e4  50.09550
```

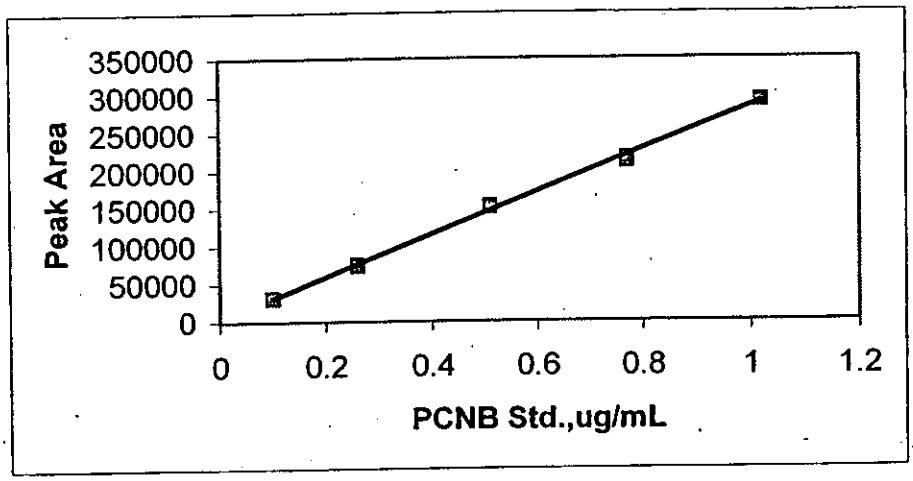
Figure 5. Representative Chromatogram of PCA and PCNB standards at 0.1 and 0.102 µg/mL respectively (highest point). The retention time of PCA is 7.1 minutes and the retention time of PCNB is 6.5 minutes.



PCA ST ug/mL	Pk.Area
0.1	30535
0.1	29731
0.25	79978
0.25	79132
0.5	152037
0.5	150420
0.75	215874
0.75	221144
1	297079
1	297556

Slope = 292324.9
Int. = 3339.634
RSQ = 0.998989

Figure 6. Representative Calibration Plot for PCA Employed for Pond Water Samples Fortified at 1.0 ppb.



PCNB ST ug/mL	Pk.Area
0.1	31921
0.1	31463
0.26	75270
0.26	73636
0.51	152201
0.51	152446
0.77	211646
0.77	217264
1.02	292389
1.02	292878

Slope = 281629.4
Int. = 3284.55
RSQ = 0.998214

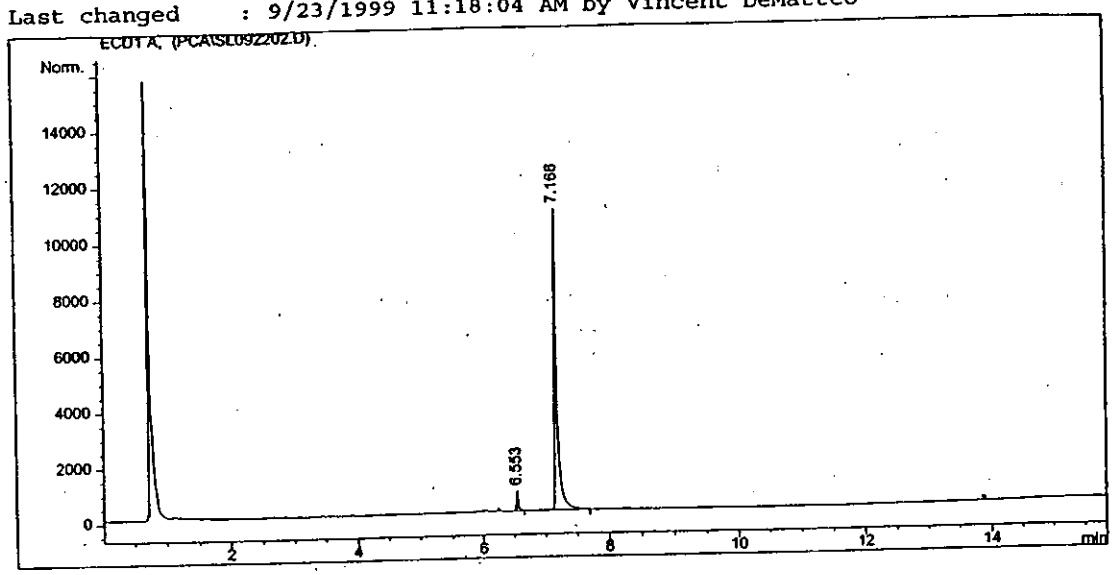
Figure 7. Representative Calibration Plot for PCNB Employed for Pond Water Samples Fortified at 1.0 ppb.

Batch Run # 2 of 46
Data File C:\HPCHEM\2\DATA\PCA\sl092202.D Sample Name: pca 0.1 ug/ml

Injection Date : 9/22/1999 3:16:35 PM
Sample Name : pca 0.1 ug/ml
Acq. Operator : skl

Seq. Line : 2
Vial : 2
Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed : 9/21/1999 7:14:59 AM by MHG
Analysis Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed : 9/23/1999 11:18:04 AM by Vincent DeMatteo



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 A,

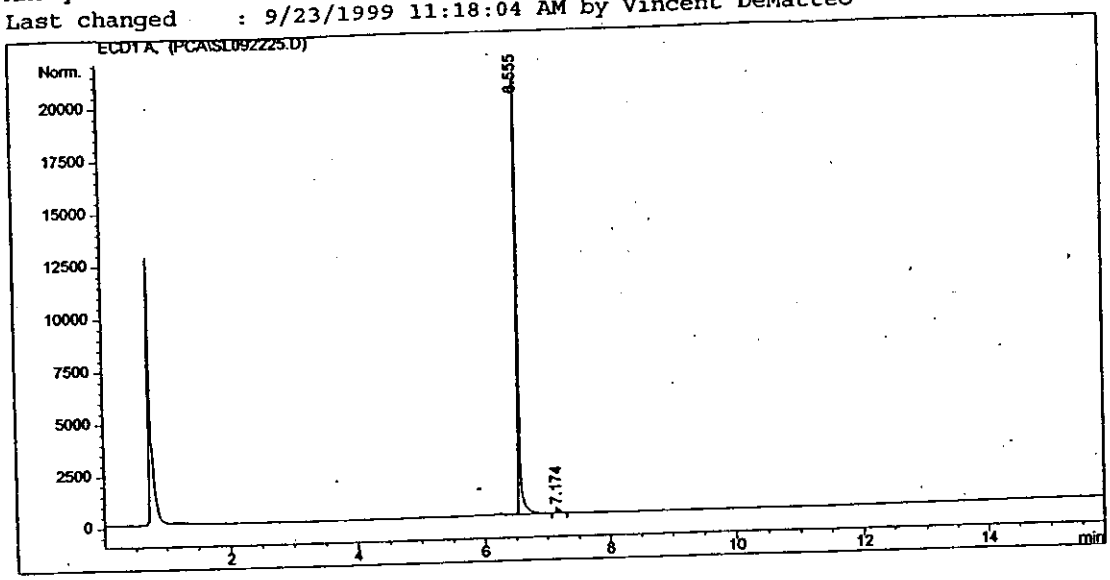
Peak #	RetTime [min]	Type	Width [min]	Area [Hz*s]	Height [Hz]	Area %
1	6.553	VV	0.0227	1148.40503	745.90564	3.62453
2	7.166	BB S	0.0385	3.05358e4	1.07989e4	96.37547
Totals :				3.16842e4	1.15448e4	

cal 99198
9.23.99
23
3 of 83

Figure 8. Representative Chromatogram of PCA Standard, 0.1 µg/mL (lowest point)

```
Batch Run # 25 of 46                                  Sample Name: pcnb 0.1ug/ml
Data File C:\HPCHEM\2\DATA\PCA\sl092225.D
```

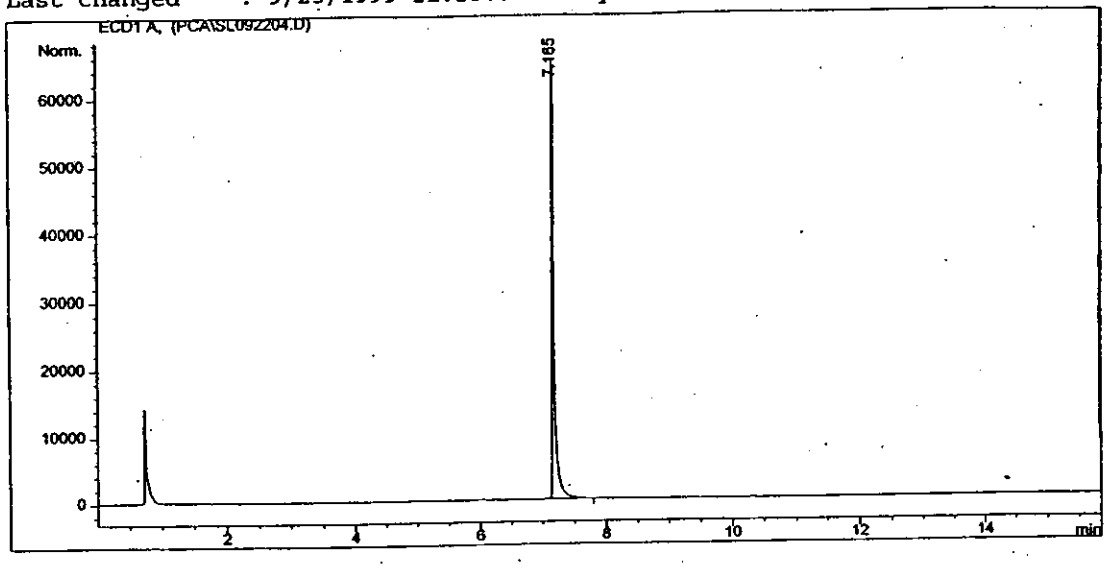
```
Injection Date  : 9/23/1999 12:10:26 AM          Seq. Line :  25
Sample Name     : pcnb 0.1ug/ml                       Vial :  19
Acq. Operator   : skl                                 Inj :   1
                                                 Inj Volume : 1 µl

Acq. Method     : C:\HPCHEM\2\METHODS\PCA.M
Last changed    : 9/21/1999 7:14:59 AM by MHG
Analysis Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed    : 9/23/1999 11:18:04 AM by Vincent DeMatteo
```




```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: ECD1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [Hz*s]      [Hz]       %
----|-------|----|-------|----------|----------|--------|
   1   6.555 VB S  0.0221 3.19210e4  2.08862e4  98.29888
   2   7.174 VV    0.0371  552.41156  181.15538   1.70112

Totals :                   3.24734e4  2.10673e4
```

Figure 9. Representative Chromatogram of PCNB Standard, 0.102 µg/mL (lowest point)

```
Batch Run # 4 of 46
Data File C:\HPCHEM\2\DATA\PCA\sl092204.D                    Sample Name: pca 0.5ug/ml

==========================================================================
Injection Date  : 9/22/1999 4:03:00 PM          Seq. Line :   4
Sample Name     : pca 0.5ug/ml                      Vial :   4
Acq. Operator   : skl                               Inj :   1
                                                Inj Volume : 1 µl

Acq. Method     : C:\HPCHEM\2\METHODS\PCA.M
Last changed    : 9/21/1999 7:14:59 AM by MHG
Analysis Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed    : 9/23/1999 11:18:04 AM by Vincent DeMatteo
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: ECD1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [Hz*s]      [Hz]       %
----|-------|----|-------|----------|----------|--------|
   1   7.165 VB S  0.0325 1.52037e5  6.50000e4  1.000e2

Totals :                  1.52037e5  6.50000e4
```

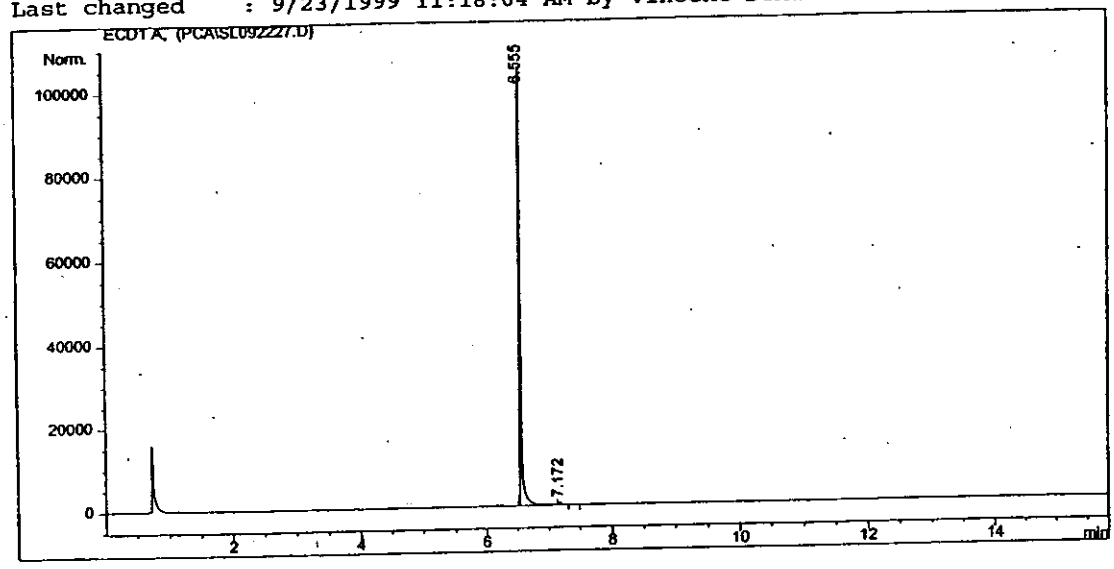
sl 99198 9-23-99 7 of 83

Figure 10. Representative Chromatogram of PCA, 0.5 µg/mL.

Batch Run # 27 of 46
Data File C:\HPCHEM\2\DATA\PCA\sl092227.D Sample Name: pcnb 0.51ug/ml

Injection Date : 9/23/1999 12:56:47 AM Seq. Line : 27
Sample Name : pcnb 0.51ug/ml Vial : 21
Acq. Operator : skl Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed : 9/21/1999 7:14:59 AM by MHG
Analysis Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed : 9/23/1999 11:18:04 AM by Vincent DeMatteo



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [Hz*s]	Height [Hz]	Area %
1	6.555	VB S	0.0212	1.52201e5	1.04949e5	98.85071
2	7.172	VB X	0.0397	1769.56775	595.40063	1.14929
Totals :				1.53971e5	1.05545e5	

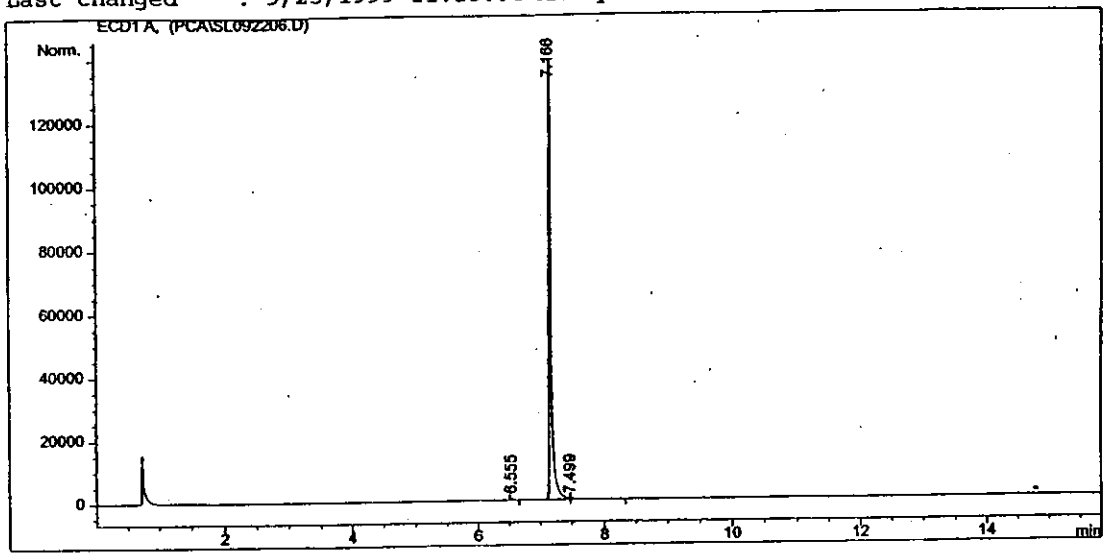
Figure 11. Representative Chromatogram of PCNB, 0.51 µg/mL.

Batch Run # 6 of 46
Data File C:\HPCHEM\2\DATA\PCA\sl092206.D Sample Name: pca 1.0 ug/ml

Injection Date : 9/22/1999 4:49:28 PM
Sample Name : pca 1.0 ug/ml
Acq. Operator : skl

Seq. Line : 6
Vial : 6
Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed : 9/21/1999 7:14:59 AM by MHG
Analysis Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed : 9/23/1999 11:18:04 AM by Vincent DeMatteo



Area Percent Report

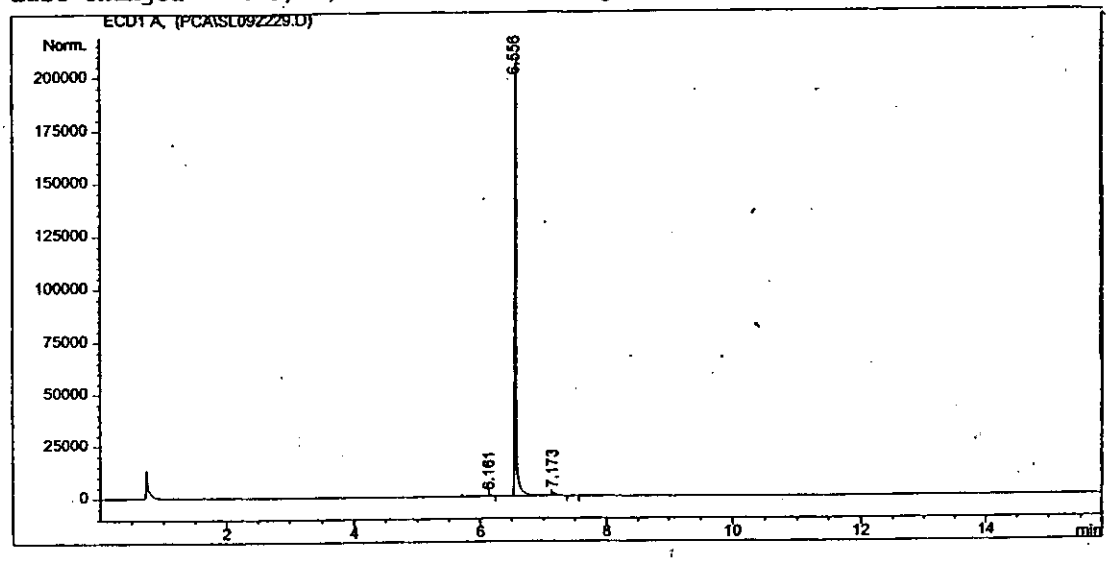
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [Hz*s]	Height [Hz]	Area %
1	6.555	VV	0.0222	929.17029	587.85602	0.30752
2	7.166	VV S	0.0305	2.97079e5	1.38887e5	98.32090
3	7.499	VB S	0.1027	4144.27734	488.42734	1.37159

Figure 12. Representative Chromatogram of PCA, 1.0 µg/mL (highest point).

```
Batch Run # 29 of 46
Data File C:\HPCHEM\2\DATA\PCA\sl092229.D              Sample Name: pcnb 1.02ug/ml

==========================================================================
Injection Date  : 9/23/1999 1:43:10 AM          Seq. Line :  29
Sample Name     : pcnb 1.02ug/ml                     Vial :  23
Acq. Operator   : skl                                Inj :   1
                                                Inj Volume : 1 µl

Acq. Method     : C:\HPCHEM\2\METHODS\PCA.M
Last changed    : 9/21/1999 7:14:59 AM by MHG
Analysis Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed    : 9/23/1999 11:18:04 AM by Vincent DeMatteo
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: ECD1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [Hz*s]     [Hz]       %
----|-------|----|-------|----------|----------|--------|
   1   6.161 VV    0.0258 1255.79407  672.89764  0.42157
   2   6.556 VB S  0.0209 2.92389e5  2.08406e5  98.15604
   3   7.173 VB X  0.0397 4237.03125 1476.03467  1.42239
```

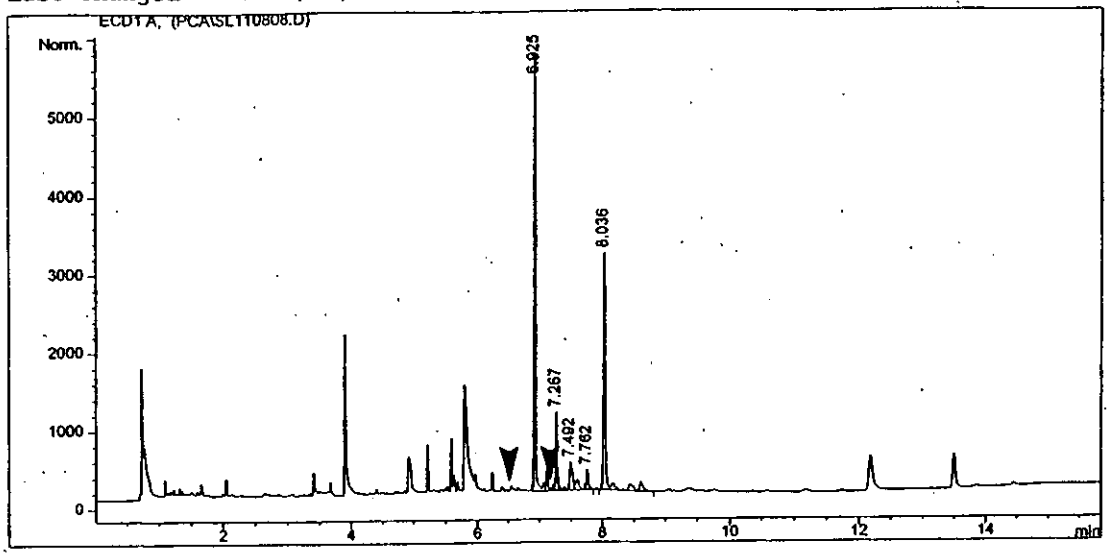
nl 99198 9/23/99 52 of 83

Figure 13. Representative Chromatogram of PCNB, 1.02 µg/mL (highest point).

Batch Run # 8 of 12
Data File C:\HPCHEM\2\DATA\PCA\sl110808.D Sample Name: Reagent blank

Injection Date : 11/8/1999 3:49:26 PM Seq. Line : 8
Sample Name : Reagent blank Vial : 8
Acq. Operator : skl Inj : 1
Inj Volume : 1 µl

Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed : 10/26/1999 7:46:41 AM by skl



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [Hz*s]	Height [Hz]	Area %
1	6.925	VB S	0.0241	9061.48828	5544.69629	47.31133
2	7.267	BV X	0.0200	1441.31934	1002.48401	7.52534
3	7.492	VV T	0.0408	1078.44678	361.52905	5.63073
4	7.762	BV T	0.0290	516.35779	259.61322	2.69598
5	8.036	VB S	0.0326	7055.27881	3032.36499	36.83663

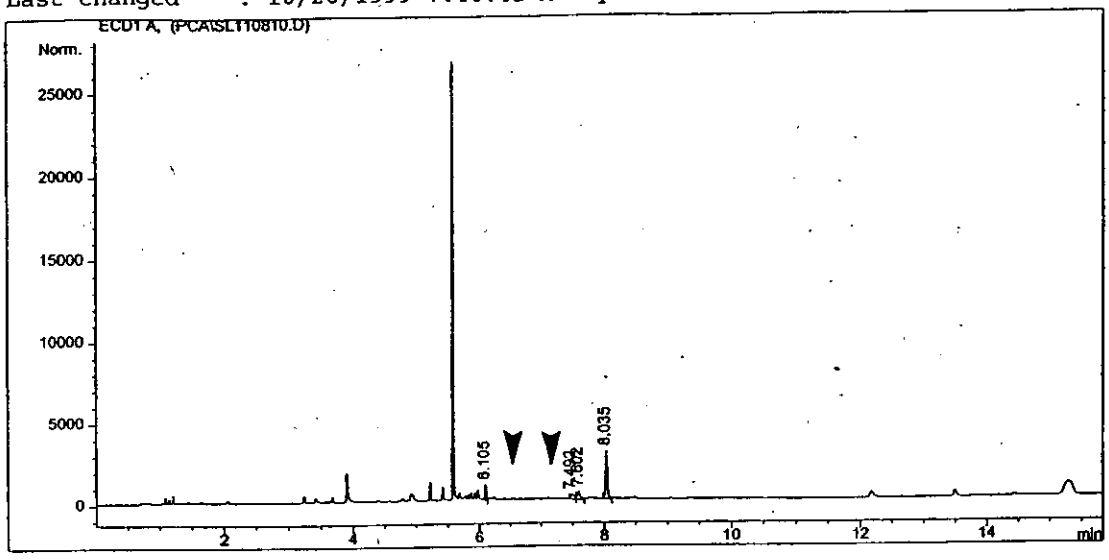
Figure 14. Representative Chromatogram of a Reagent Blank. The reagent blank is from a set of samples fortified at 0.1 ppb level with PCA and PCNB

Batch Run # 10 of 12
Data File C:\HPCHEM\2\DATA\PCA\sl110810.D Sample Name: control#1

Injection Date : 11/8/1999 4:35:55 PM
Sample Name : control#1
Acq. Operator : skl

Seq. Line : 10
Vial : 10
Inj : 1
Inj Volume : 1 µl

Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed : 10/26/1999 7:46:41 AM by skl



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 A,

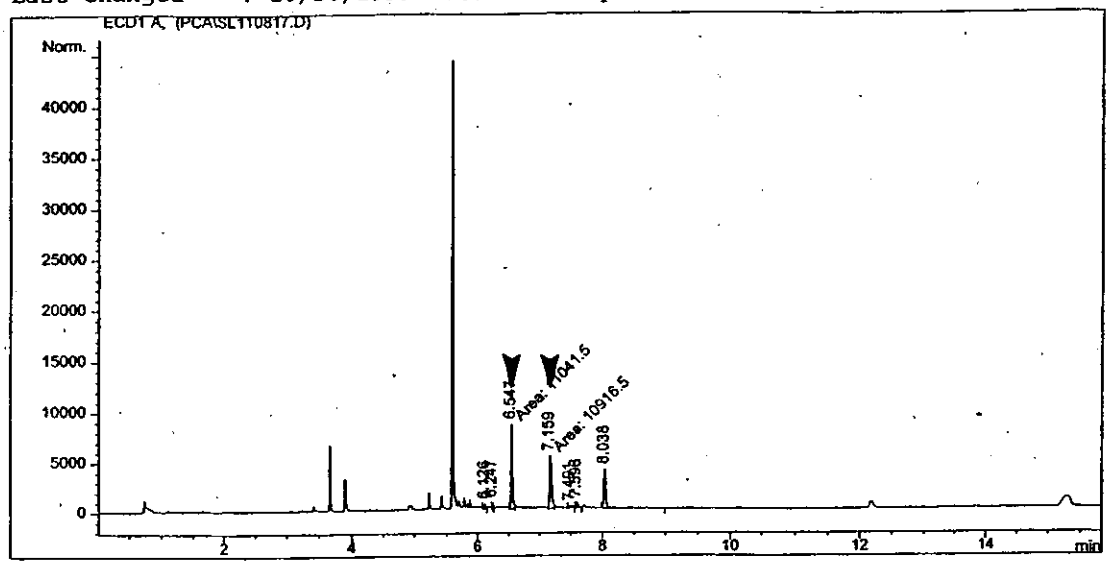
Peak #	RetTime [min]	Type	Width [min]	Area [Hz*s]	Height [Hz]	Area %
1	6.105	VV	0.0166	1113.95715	991.33594	11.69416
2	7.492	VV	0.0453	1143.76587	332.98712	12.00709
3	7.602	VV	0.0374	1442.63293	508.04306	15.14456
4	8.035	VV	0.0284	5825.39648	2967.89160	61.15419
Totals :				9525.75244	4800.25772	

Figure 15. Representative Chromatogram of Control Pond Water. The control is from a set of samples fortified at 0.1 ppb with PCA and PCNB.

Data File C:\HPCHEM\2\DATA\PCA\SL110817.D Sample Name: water*#5

Injection Date : 11/8/1999 7:18:32 PM Seq. Line : 17
Sample Name : water*#5 Vial : 17
Acq. Operator : skl Inj : 1
Inj Volume : 1 µl

Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed : 10/26/1999 7:46:41 AM by skl



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

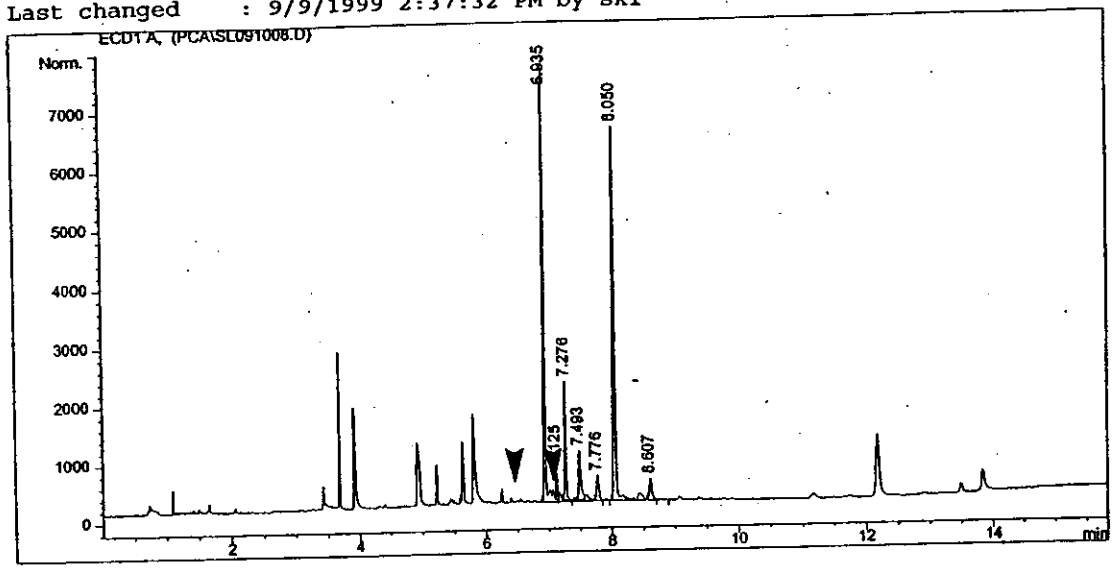
Signal 1: ECD1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [Hz*s]	Height [Hz]	Area %
1	6.126	VV	0.0267	1057.28943	531.54150	2.97424
2	6.247	VV	0.0189	872.60565	683.49194	2.45471
3	6.547	MM	0.0216	1.10415e4	8506.50879	31.06055
4	7.159	MM	0.0339	1.09165e4	5373.70361	30.70890
5	7.491	BV T	0.0416	819.31665	259.28174	2.30480
6	7.596	PV T	0.0300	1091.22119	564.60535	3.06969

Figure 16. Representative Chromatogram of Pond Water Fortified at the 0.1 ppb Level with PCA and PCNB.

```
Batch Run # 8 of 46                                   Sample Name: reagent blank
Data File C:\HPCHEM\2\DATA\PCA\sl091008.D
```

```
Injection Date   : 9/15/1999 4:38:09 PM          Seq. Line :   8
Sample Name      : reagent blank                      Vial :   8
Acq. Operator    : Vincent DeMatteo                   Inj :   1
                                                  Inj Volume : 1 µl

Method           : C:\HPCHEM\2\METHODS\PCA.M
Last changed     : 9/9/1999 2:37:32 PM by skl
```



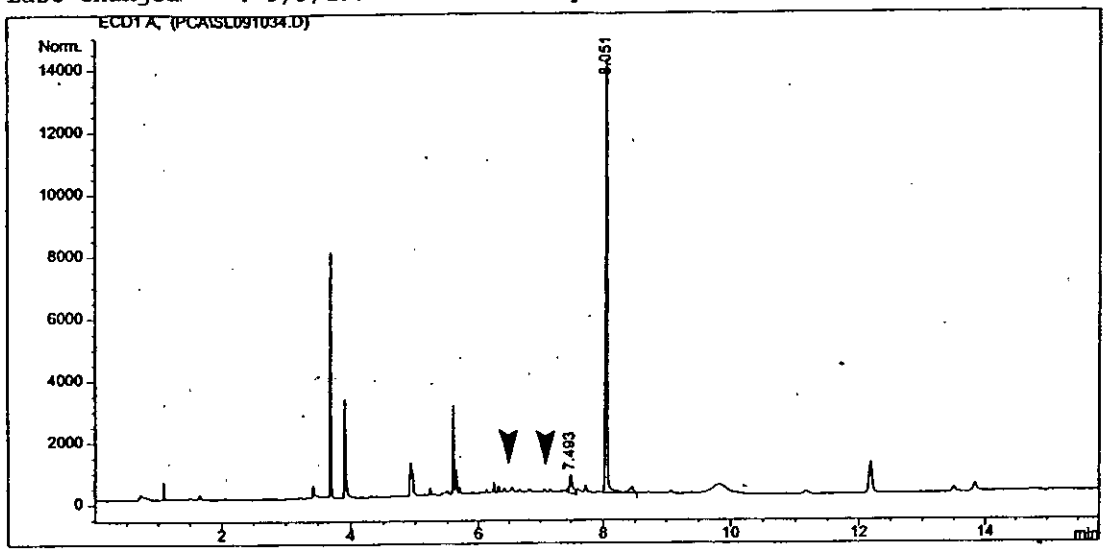

```
                        Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: ECD1 A,
```

Peak #	RetTime [min]	Type	Width [min]	Area [Hz*s]	Height [Hz]	Area %
1	6.935	VB S	0.0245	1.23997e4	7400.59766	37.77262
2	7.125	VV X	0.0199	690.70874	530.77618	2.10407
3	7.276	VV T	0.0228	3002.83984	2050.68237	9.14741
4	7.493	PV T	0.0357	2051.99658	850.10876	6.25090
5	7.776	BV T	0.0319	984.76184	431.41171	2.99983
6	8.050	VB S	0.0285	1.27054e4	6367.18213	38.70388

99198 pl
9-16-99
14 of 83

Figure 17. Representative Chromatogram of a Reagent Blank. The reagent blank is from a set of pond water samples fortified at 1.0 ppb with PCA and PCNB.

```
Batch Run # 34 of 46
Data File C:\HPCHEM\2\DATA\PCA\sl091034.D                    Sample Name: control#2

==========================================================================
Injection Date  : 9/16/1999 2:21:58 AM          Seq. Line :  34
Sample Name     : control#2                          Vial :  11
Acq. Operator   : Vincent DeMatteo                    Inj :   1
                                                Inj Volume : 1 µl

Method          : C:\HPCHEM\2\METHODS\PCA.M
Last changed    : 9/9/1999 2:37:32 PM by skl
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: ECD1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [Hz*s]      [Hz]       %
----|-------|----|-------|----------|----------|--------|
   1   7.493 VV    0.0411 1723.45459  581.67798  5.98807
   2   8.051 VB S  0.0293 2.70580e4  1.39831e4  94.01193

Totals :                   2.87815e4  1.45648e4

 Results obtained with enhanced integrator!
```

99198 µl
9-16-99
60 of 83

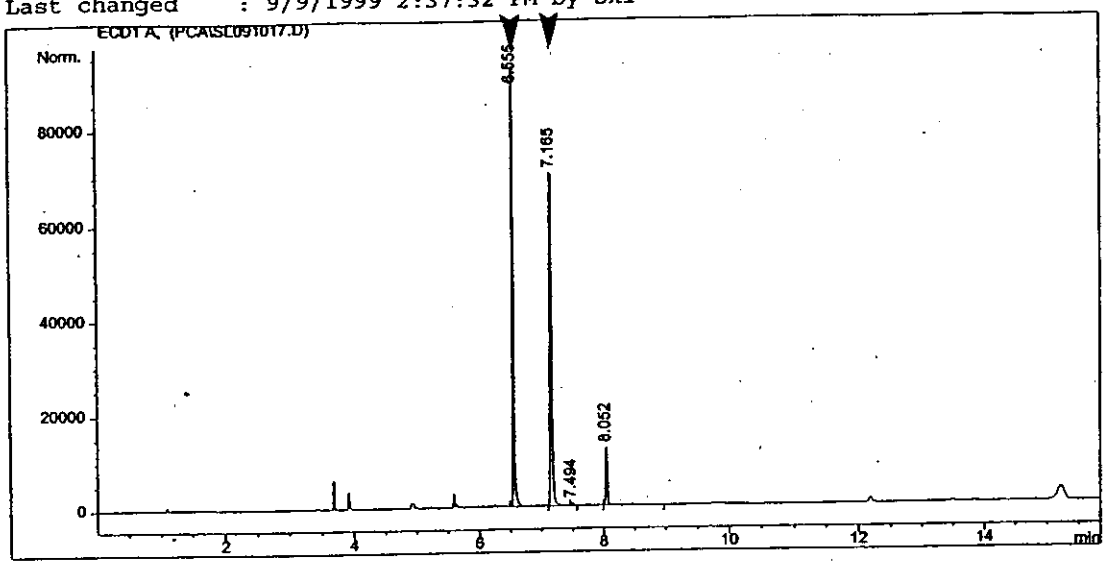
Figure 18. Representative Chromatogram of Control Pond Water. The control pond water is from a set of samples fortified at 1.0 ppb with PCA and PCNB.

Batch Run # 17 of 46
Data File C:\HPCHEM\2\DATA\PCA\s1091017.D Sample Name: water*5

Injection Date : 9/15/1999 8:00:16 PM
Sample Name : water*5
Acq. Operator : Vincent DeMatteo

Seq. Line : 17
Vial : 17
Inj : 1
Inj Volume : 1 µl

Method : C:\HPCHEM\2\METHODS\PCA.M
Last changed : 9/9/1999 2:37:32 PM by skl



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: ECD1 A,

Peak #	RetTime [min]	Type	Width [min]	Area [Hz*s]	Height [Hz]	Area %
1	6.555	VV S	0.0188	1.15368e5	9.26431e4	44.19005
2	7.165	VV S	0.0250	1.20446e5	7.02113e4	46.13520
3	7.494	BV T	0.0352	1527.20764	599.37421	0.58498
4	8.052	VPAS	0.0278	2.37308e4	1.22525e4	9.08978
Totals :				2.61072e5	1.75706e5	

99198.1
9-16-99
30 of 83

Figure 19. Representative Chromatogram of Pond Water Fortified at the 1.0 ppb Level with PCA and PCNB.

```
File       : C:\HPCHEM\1\DATA\101902.D
Operator   :
Acquired   : 19 Oct 1999  10:30     using AcqMethod DEFAULT
Instrument :    GC/MS Ins
Sample Name: pcnb, 1 mg/ml
Misc Info  :
Vial Number: 2
```

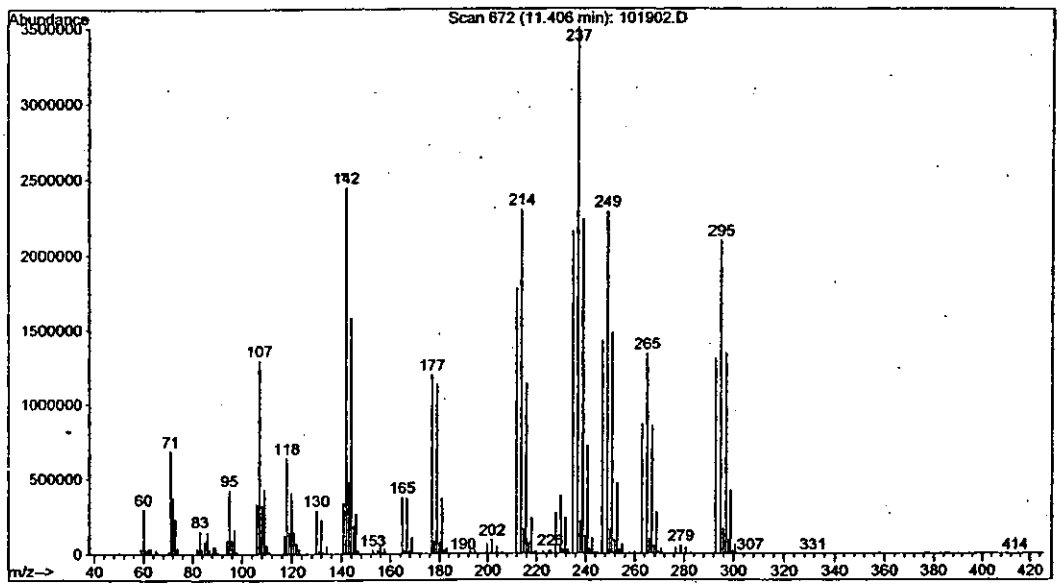


Figure 20. Mass Spectrum of PCNB

```
File        : C:\HPCHEM\1\DATA\101901.D
Operator    :
Acquired    : 19 Oct 1999  10:03     using AcqMethod DEFAULT
Instrument :    GC/MS Ins
Sample Name: pca, 1 mg/ml
Misc Info   :
Vial Number: 1
```

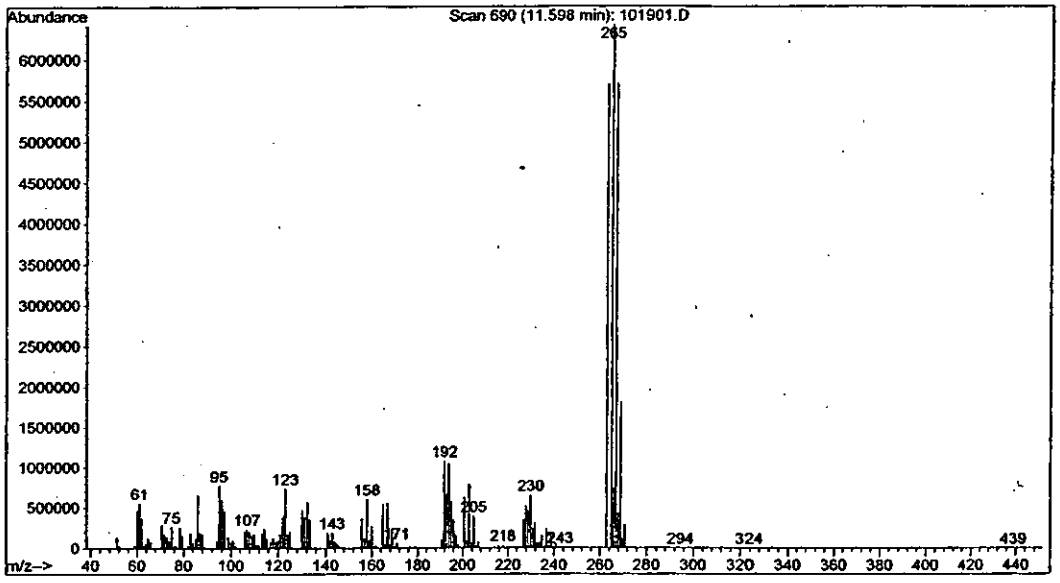


Figure 21. Mass Spectrum of PCA

```
File       : C:\HPCHEM\1\DATA\10219902.D
Operator   :
Acquired   : 21 Oct 1999  10:53     using AcqMethod PCA
Instrument :    GC/MS Ins
Sample Name: Spiked water extract#sl092215
Misc Info  : 1ppb level
Vial Number: 2
```

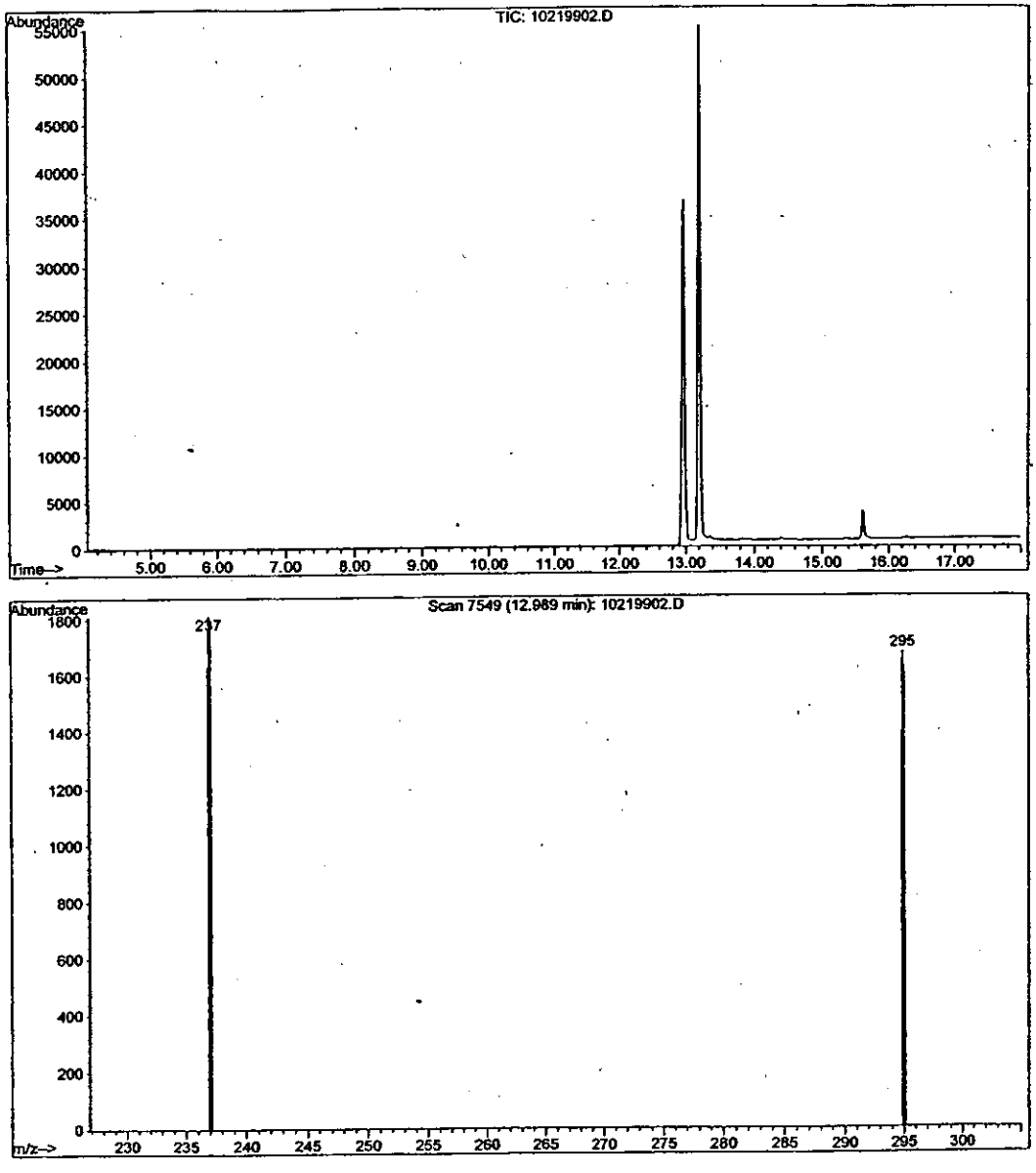


Figure 22. Selected Ion Chromatogram of Pond Water Fortified at 0.1 ppb with PCNB Showing the Selected Ions Used for Identification of PCNB.

```
File       : C:\HPCHEM\1\DATA\10219902.D
Operator   :
Acquired   : 21 Oct 1999  10:53     using AcqMethod PCA
Instrument :    GC/MS Ins
Sample Name: Spiked water extract#s1092215
Misc Info  : 1ppb level
Vial Number: 2
```

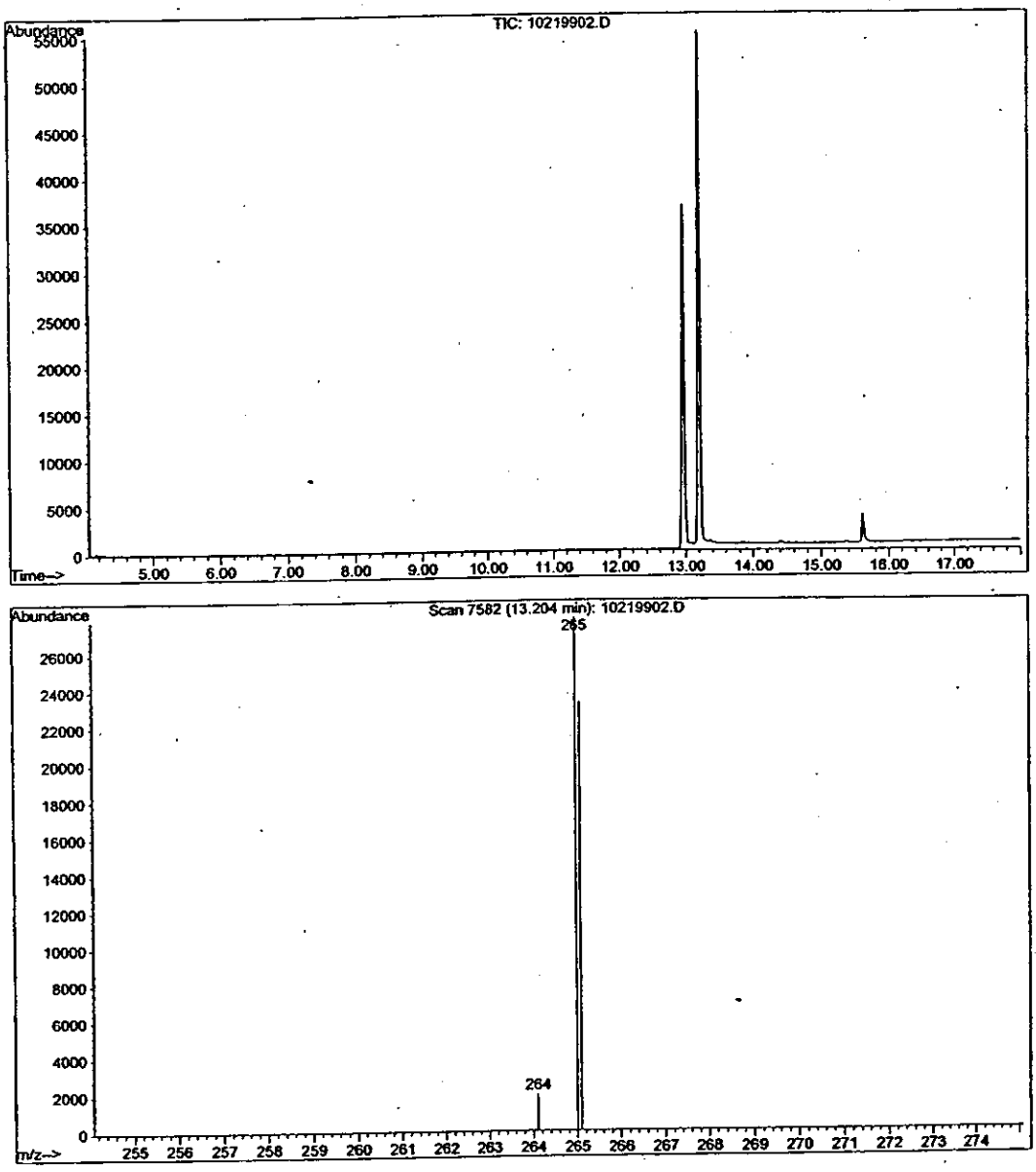


Figure 23. Selected Ion Chromatogram of Pond Water Fortified at 0.1 ppb With PCA Showing the Selected Ions Used for Identification of PCA.

APPENDIX A. CHARACTERIZATION OF POND WATER

Edglo
LABORATORIES, INC.
www.edglo.com
2121 E. Washington Blvd Fort Wayne, Indiana 46803
Phone:219-424 1622 (800) 891-8442 Fax: 219-424-9124
e-mail: pknott@edglo.com

Your Environmental Specialists
Since 1969

Shari K. Long

Uniroyal Chemical Company
Benson Road
Middlebury CT 06749

Laboratory #	Report Date	Date/Time Sampled
0924991099	9/30/99	From:
Date Received	Sample Matrix	To:
9/24/99	Water	
Comments		
Uniroyal Study #99198 - Preston Hill Pond		

Report of Analysis

Parameter	Method Code	MDL	Result	Units	Permit Limits	Date/Time	Analyst
Dissolved Organic Carbon	EPA 415.1	0.5	9.02	mg/L		9/29/99 9:00:00 AM	CI
pH	EPA 150.1		7.0	S.U.		9/24/99 3:00:00 PM	SB
Total Hardness	EPA 130.2	1	46	mg/L		9/27/99 8:50:00 AM	JCH

< Is Less than reported method detection limit

Certification Number: INC0203

Respectfully Submitted By: