Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Quintozene (PCNB)

MRID #:   446023-01

Matrix: Soil

Analysis: GC/ECD

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STUDY TITLE

Analytical Method for PCNB, and its degradates in soil

Data Requirement

Not Applicable

Author

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Study Completion Date

June 16, 1998

Performing Laboratory

Uniroyal Chemical Co.
Middlebury, CT 06749

Laboratory Project Identification

AC 6000

Related Reports

Uniroyal Research Project   MIRD
     92147
     9173   43061501
     RP-91051   (Appendix VII)

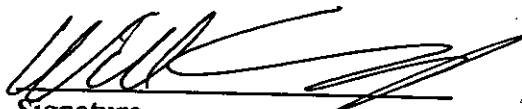### Compiled by:

J.B. Pierce

Key Words: Analytical method, pentachloronitrobenzene, pentachloroaniline, pentachlorophenol, pentachlorothioanisole, pentachlorobenzene, hexachlorobenzene, pentachlorothioanisole, sulfoxide, pentachlorothioanisole sulfone, tetrachlorothioanisole sulfone, soil

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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).

Sponsor: Uniroyal Chemical Company, Inc.

Company Agent: _____ _____7-6-98_____

                      Signature   Date

Willard F. Cummings

U.S. Registration Manager

STATEMENT OF ADHERENCE TO GLP's

This submission is not considered a "study" as defined by 40CFR 160 and as such falls outside the scope of GLP requirements. It consists of an analytical method which has been compiled and reformatted to conform more closely with data reporting guideline # 850.7100 (draft) and EU guidelines under commission directive 96/46/EC of 16 July 1996. Information for this report was taken from previously submitted GLP studies as indicated on the title page.

Certification

This analytical method was compiled from information in the following reports:

1)   Uniroyal project 92147
2)   Uniroyal project 9173
3)   Uniroyal project RP-91051

Signature: *J. B. Pierce*

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Date: June 16, 1998

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SUMMARY

PCNB and its degradates were extracted from soil. Before analysis one of the degradates, pentachlorophenol, was converted to the methyl ether using diazomethane. The combined extracts were analyzed by GC using an electron capture detector.

A)   MATERIALS

A.1   Equipment

Balance	Mettler PE 3000
Centrifuge	Damon/IEC
Centrifuge bottle, teflon 250 ml	Nalgene
Hobart Food Chopper	Hobart Mfg. Co.
Erlenmeyer Flask, 250 ml	Pyrex, Kimax
pH meter	Beckman
Rotary evaporator, Buchi Rotovap	Brinkman
Round bottom flasks, 500 ml	Pyrex, Kimax
Separatory funnel, 250 ml	Nalgene
Standard laboratory equipment:	
beakers, pipets, test tubes etc.	Pyrex, Kimex
TurboVap LV evaporator	Zymark

A.2 Reagents/Supplies

Acetone, residue grade	Baker
1-Decanol	Baker
Diazomethane in diethyl ether solution	Aldrich
Diethyl ether, residue grade	Baker
Dry Ice	Penn State University
n-Hexane, residue grade	Baker
HCB Analytical Standard AC-1194-38C	Uniroyal Chemical Co.
Nitrogen	MG Industries
PCA Analytical Standard AC-1234-1	Uniroyal Chemical Co.
PCB Analytical Standard AC-1166-14	Uniroyal Chemical Co.
PCNB Analytical Standard AC-1261-133	Uniroyal Chemical Co.
PCP Analytical Standard AC-1261-84	Uniroyal Chemical Co.
PCTA Analytical Standard AC-1166-16	Uniroyal Chemical Co.
PCTASO Analytical Standard AGD-1384-005	Uniroyal Chemical Co.
Sodium Hydroxide	Baker
Sulfuric Acid	Baker
TCTASOO Analytical Standard AGD-1384-024	Uniroyal Chemical Co.
Toluene, residue grade	Baker

A.3 Analytical Standards

The following standards are used to analyze for PCNB and its degradates. Standards should be stored at –5°C to -25°C until use. Standards can be obtained from Uniroyal Chemical Inc. Structures for these standards are shown in Figure I.

NAME	LOT NUMBER	PURITY
PCNB	AC-1261-133	99.8%
PCB	AC-1166-14	100.0%
HCB	AC-1194-38C	99.8%
PCA	AC-1234-1	97.0%
PCTA	AC-1166-16	99.1%
PCP	AC-1261-84	98.4%
TCTASOO	AGD-1384-024	99.4%
PCTASO	AGD-1384-005	96.7%

MSDS sheets for the above standards are found in Appendix I.

B. SAFETY AND HEALTH

This method should be performed by trained chemical personnel. Hazards associated with the chemicals used in this analytical method are shown in the MSDS sheets in Appendix I. Special precautions are needed during the use of diazomethane.

C. ANALYTICAL METHOD

C.1 Principle of the Method

Soil samples are homogenized and then extracted with acetone/hexane. Basic and neutral degradates (and PCNB) are partitioned into hexane after addition of pH 12 water. PCP is partitioned from the water phase into hexane after acidification and methylated with diazomethane. Both organic extracts are then

combined and the components analyzed by GC using an electron capture detector.

C.2 Types of Soils

This method is predicted to be applicable to most soil types. In Uniroyal Chemical Inc. project 92147 soils from a Texas USA location were used and the composition varied depending on soil depth from sandy loam (0-12 inch depth) to sandy clay loam (12-24 inch depth) to clay loam (24 to 48 inch depth).

C.3 Sample Processing

Frozen cores are normally received and are divided into smaller pieces with a cleaver and rubber mallet. The stones and debris are removed. A Hobart Food Chopper is pre-chilled with dry ice and the frozen soil pieces are put inside. The soil is chopped and homogenized with dry ice. The processed soil is placed in sample containers and stored in the freezer where the dry ice is allowed to sublime overnight at <-10°C. The sample containers are capped and kept under freezer conditions (-5°C to -25°C) until analysis.

C.4 Extraction Method

A flow diagram of the analysis procedure is shown in Figure II. Detailed explanations of each step are as follows:

Step 1   Extraction (10 g soil, wet weight)

Weigh 10 g of soil in a 250 ml teflon centrifuge bottle. Fortify the two spike samples used to determine extraction method recoveries for the set of samples.

Add 100 ml 50:50 v/v acetone: hexane and shake vigorously for 2 min. Centrifuge at 1500 RPM for 5 min. Pour the supernatant into a separatory funnel, leaving the soil in the bottle. Add another 50 ml of 50:50 v/v acetone: hexane to the soil, shake 1 min and centrifuge. Add supernatant to the separatory funnel.

Step 2   Partition

Add 50 ml distilled/deionized water (pH > 12, adjusted with 25% NaOH) to the separatory funnel and shake for 20 sec. Drain the water/acetone layer into a 250 ml beaker and collect the hexane layer in a 250 ml Erlenmeyer flask. Re-extract the water/acetone layer with 50 ml hexane, shaking for 1 min. Drain the water/acetone layer into the 250 ml beaker and add the remaining hexane layer to the flask. The extract done under basic conditions, in the Erlenmeyer flask, contains compounds PCB, HCB, PCNB, PCA, PCTA, TCTASOO, and PCTASO.

Step 3   Removal of Acidic Metabolite

Pour the water/acetone portion back into the separatory funnel and add 10 ml 10N H_2SO_4 to lower the pH < 1. Add 50 ml hexane, shake vigorously for 1 min and drain the water/acetone layer into the beaker. Pour the hexane layer into a 500 ml round bottom flask. Re-extract the water/acetone with another 50 ml hexane by shaking for 1 min. Drain the water/acetone layer into the beaker and add the remaining hexane layer to the round bottom flask. The extract done under acidic conditions, in the round bottom flask, contains the compound PCP.

Step 4   Methylation, Diazomethane Method

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Add 10 drops of decanol to the acidic extract to prevent the sample from going to dryness during evaporation and reduce the volume to about 5 ml using a rotary evaporator. Transfer this portion of the sample into a methylation vial, rinsing the round bottom flask with hexane. Further reduce the volume of the sample to 0.5 ml using a TurboVap LV evaporator under nitrogen. Add 0.5 ml diazomethane, or enough to turn the sample yellow. Let it stand under a hood for 10 min. Evaporate off the diazomethane using the TurboVap, reducing the volume again to 0.5 ml.

Step 5   Combine the Extracts and Adjust the Volume

Rinse a round bottom flask with acetone and transfer the basic extract (prepared in Step 2) from the Erlenmeyer flask to the round bottom flask. Reduce the volume to about 5 ml using the rotary evaporator, then add 10 ml toluene. Pour the methylated portion of the sample into the round bottom flask, rinsing the vial with 15 ml toluene. Reduce the volume of the combined extracts to about 5 ml with the rotary evaporator, then and bring the final volume up to 10 ml with toluene. The sample is now ready for GC analysis.

Step 6   Percent Soil Moisture Determination

Percent soil moisture is determined by weighing two aliquots of soil, before and again after oven drying for 16 hours at 100°C.

C.5   Gas Chromatography Method

The type of column used in the GC analysis of soil samples is a Restek Rtx-35, 30 meter in length, 0.53 mm internal diameter (ID), with 0.25 μm film thickness (DF). The Rtx-35 has a stationary phase made of 35% diphenyl-65%

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dimethyl polysiloxane. It is rated intermediate in polarity. The samples are delivered to the column by direct injection. The injector temperature is 270°C. The column housing oven is programmed to increase the temperature at a rate of 5°C/min from 100°C to 200°C, during which all of the compounds of interest passed through the column to the ECD detector. This is followed by a temperature increase at 20°C/min to 270°C to clean out any remaining impurities. The detector temperature is set at 300°C.

The GC run begins with the injection of a 1 µl aliquot of each of the four eight-component mixed standards in the range of 0.003µg/ml to 0.100 µg/ml. Standards and washes are run intermittently with the samples to help monitor the stability of the run, and to make sure the column is clean before the next sample is injected. The resulting standard peak areas are plotted versus concentration (µg/ml) of the corresponding standard to obtain standard calibration curves. Standard curves are generated for each analysis day, using all standards injected during the run.

A 1 µl aliquot of the sample is injected into the GC. If the compound peak area in the sample is greater than the peak area of the highest standard, the sample extract is diluted with toluene until the signal response falls within the standard curve range. The peak areas of the compounds are recorded and the concentration of each compound is determined relative to the standard curves generated for that day.

C.6 Preparation of Spiking and Standard Solutions

Analytical standards are used to prepare individual compound stock solutions from which working standard and method day spiking solutions are prepared. Stock solutions of each compound at a concentration of 1.0 mg/ml are made by

weighing out 10 mg of the analytical standard on an analytical balance and dissolving it in 10 ml of toluene. The amount of toluene added is determined considering the percent purity of the standard. For example, PCTA was 99.1% pure, so 10 mg of PCTA is weighed out and dissolved in 9.91 ml of toluene (10.0 x 0.991). The PCP stock solution is prepared using methanol. Methylated pentachlorophenol (PCP-OMe) is prepared in toluene and corrected considering the PCP equivalence. The molecular weight of PCP (266) is divided by the molecular weight of PCP-OMe (280) to yield a correction factor of 0.95. For example, if 10.0 mg of PCP-OMe is weighed out, then 9.50 ml of toluene is added to make 1 mg/ml solution (10.0 x 0.95 = 9.50).

A solution of seven compounds combined in toluene at a concentration of 100 µg/ml is made by adding 2 ml of each of the individual compound stock solutions at 1.0 mg/ml of PCB, HCB, PCNB, PCA, PCTA, TCTASOO and PCTASO, to 6 ml of toluene, so that the final volume is 20 ml. A solution of PCP at 100 µg/ml is made by diluting the 1.0 mg/ml stock solution of PCP 10-fold with methanol.

A method spiking solution of the combined seven compounds at a concentration of 10 µg/ml is made by a 10-fold dilution of the 100 µg/ml seven compound solution with toluene. Likewise, a PCP spiking solution at 10 µg/ml is made by a ten fold dilution with methanol of the 100 µg/ml PCP solution. Fortification of the method spike samples at a 0.1 ppm level is accomplished by adding 100 µl of the 10 µg/ml spiking solutions to 10 g. of control soil, and bringing the final volume of the extract to 10 ml. Fortification of the method spike samples at a 1.0 ppm level is done by adding 100 µl of the 100 µg/ml spiking solutions to 10 g of control soil and bringing the final volume of the extract to 10 ml.

A 10 µg/ml standard solution is made by adding 200 µl of each individual

compound stock solutions of PCB, HCB, PCP, PCNB, PCA, PCTA, TCTASOO and PCTASO at 1 mg/ml, and bringing the final volume to 20 ml with toluene. In this case the solution of PCP-OMe in toluene is used. A 1 µg/ml standard solution of the combined eight compounds is prepared by diluting the 10 µg/ml standard solution 10 fold with toluene. Dilutions of the 10 µg/ml and 1 µg/ml standard stock solutions are made to prepare working standards at 0.100 µg/ml, 0.050 µg/ml, 0.010 µg/ml, and 0.003 µg/ml.

C.7 Extraction Efficiency

Duplicate soil samples were spiked in the field with each of the analytes PCNB. PCA, PCP, PCTA, PCB, HCB, PCTASO and TCTASOO on two occasions, at the 120 day sampling (0-3 month) and at the 270 day sampling (6-12 month). The results of spiking at a level of 1.0 µg of analyte in 10 g of soil are shown in Table VI of Uniroyal Project 92147 (Figure III of this report). These results indicate that the analytes did not undergo significant breakdown under the conditions of handling and shipping. Examples of chromatograms and data calculation spreadsheets for the field spikes are presented in Appendix II.

C.8 Fortifications

Soil samples spiked in the laboratory which were extracted and analyzed along with the actual test samples showed recoveries in the range of 70 – 120% for all of the analytes. The laboratory spike results indicate that the analytical methodology provided reliable results during the course of study 92147. An example of chromatograms of control samples and spiked control samples are shown in Appendix III (taken from studies 92147 and RP 91051).

D. INSTRUMENTATION

The gas chromatograph and integrator models, column type, and operating conditions were as follows:

Instrument:	Hewlett Packard Model 5890 Series II Gas Chromatograph
Column:	Restek RTX-35, 30 m, 0.53 mm ID, 0.25 um df
Oven:	Initial temp. 100°C, Initial time 2 min
	Rate A: 5°C/min to 200°C, final time 1 min
	Rate B: 20°C/min to 270°C, final time 5 min
Detector:	Electron Capture Detector (ECD), temp. 300°C
Injector:	Direct Injection, temp. 270°C
Carrier Gas Flow:	Hydrogen, 10 ml/min
Make-up Flow:	Nitrogen, 35 ml/min
Integrator:	Shimadzu C-4RA Chromatopac

E. SAMPLE BRACKETING

The calibration was done by standard bracketing. A typical run involved running the standard curve, followed by a control, then two day spikes, 4 samples, two day spikes, and 4 more samples. Data from a typical run including the chromatographs are shown in Appendix IV.

F. POTENTIAL INTERFERENCES

This method could have interferences from other halogenated pesticides that might elute with similar retention times. One should consider the soil history in this respect and a confirmatory technique should be used if a problem is suspected.

G. CONFIRMATORY TECHNIQUES

The method of confirmation for the definite identification of PCB, HCB, PCNB, PCA, PCTA, TCTASOO and PCTASO was by GC/MS. It was completed using two standard solutions and sample 932265, which gave sharp peaks for all of the compounds of interest except PCP. The base peaks used in the spectra for selective ion monitoring of the compounds in the samples is as follows: compound-base peak, qualifying peak PCB-250, 215, HCB-284, 249, PCNB-237, 295, PCA-265, 263, PCTA-296, 246, TCTASOO-231, 215, PCTASO-297, 295. Comparing the total ion chromatograms of standard solutions containing all eight compounds at 0.100, 0.050 µg/ml the order of elution of the compounds is shown to be PCB, HCB, PCP, PCNB, PCA, PCTA, TCTASOO, PCTASO. Chromatograms showing these confirmations are shown in Appendix V.

The method of confirmation for the definite identification of PCP was done using an RXT-200 chromatographic column. The level of this compound found in the samples is usually too low for detection by GC/MS. Samples which show the greatest amount of this compound can be used for the confirmation. The RTX-200 column has a polarity selective for lone pair electrons and gives a sharp PCP peak. Confirmation of PCP can thus be done using the RTX-200 column rather than the RTX-35 column as a second chromatographic technique. Typical chromatograms of PCP and the other degradates using the RTX-200 column are shown in Appendix VI. Chromatography of a typical soil sample from study 92147 is also shown in Appendix VI.

H. TIME REQUIRED FOR ANALYSIS

The extraction of eight soil samples and the chromatography to develop the daily standard curve and run the eight samples and four day spikes and control can be done

in 24 hours.

J. MODIFICATION OR POTENTIAL PROBLEMS

None.

K. CALCULATIONS

The peak areas corresponding to the eight compounds (PCB, HCB, PCP, PCNB, PCA, PCTA, TCTASOO, and PCTASO) in the standards were obtained from the chromatograms and regressed versus the concentration of the compounds in the standards. Statistics were generated on a Swan Corporation 386/33 computer using an Axum program capable of performing quadratic regression (second order polynomial regression) on the peak areas versus their corresponding concentrations to generate standard curves. The following quadratic equation was used:

$$\text{Peak Area} = b_0 + b_1 * (\mu g/ml \text{ of standard}) + b_2 * (\mu g/ml \text{ of standard})^2$$

In a few cases, peaks were found in the control samples. If required, a corrected peak area value was determined using the following formula:

Peak area in sample corrected = Peak area in sample – Peak area in control

The corrected peak area of each sample was used to calculate the amount in ug/ml of each compound found in the samples analyzed relative to the generated standard curves. The square of the correlation coefficient (R^2) was used to evaluate the fit of the curve. The $\mu g/ml$ compound found value was then multiplied by the final volume of the sample to yield the μg compound found.

μg compound found = [$\mu g/ml$ compound found] x [final volume (ml)]

In fortified method spikes, the μg compound found values were converted to ppm

compound found values by dividing by the sample weights. The ppm compound found values were then divided by ppm compounds added to obtain the percent recoveries.

If the average percent recovery for the two spiked samples of the set was below 100%, the amount of compound found in the sample was divided by the average recovery of the spikes to give the corrected value. No correction was made for average recoveries above 100%

$$\mu g \text{ compound found corrected} = \mu g \text{ compound found} / \text{average spike recovery}$$

In field samples, the ppm compound found was calculated using the μg compound found corrected for percent recovery divided by sample weight.

$$\% \text{ moisture} = (\text{wet weight} - \text{dry weight} / \text{wet weight}) \times 100$$

The average % moisture for 2 aliquots was used to determine the % soil moisture by the following equation:

$$\text{ppm compound found} = \text{ppm compound found uncorrected}/[(100 - \% \text{ soil moisture})/ 100]$$

L.   COPIES OF CHROMATOGRAMS

Copies of chromatograms for a control and spiked samples are shown in Appendix III. Typical chromatograms for sample runs are shown in Appendix IV.

M.   METHOD VALIDATION

M.1   Accuracy (USA) / Recovery (EU)

A formal study of accuracy was done in Uniroyal report RP-91051 (Appendix VII) for PCNB and the metabolites PCB, HCB, PCP-OMe, PCA and PCTA. Figure IV summarizes the recovery data at five spiking levels. Mean percent

recoveries, standard deviations (SD), relative standard deviations (RSD), the range of recoveries, and the ± confidence limits for 95% confidence are shown for spiking levels of 0.005, 0.25, 1.0, 3.0 and 10 ppm of PCNB and the degradates mentioned above. Note that not all levels were tested on each day and that one value (the 0.005 ppm spike on day 12/12/91) was omitted from Figure IV because it was mistakenly done at 0.1 ppm (see Appendix VII). The data in Figure IV show that all recoveries are between 70 and 110% as required by the EU (70 – 120% as required by the USA).

In Figure IV the RSD was calculated as:

$$RSD = \frac{SD}{Average} \times 100\%$$

The 95% confidence limits (CL) were calculated as:

$$CL = \frac{t \times SD}{\sqrt{n}}$$

Where SD = standard deviation

n = the number of observations

t = the value t for n-1 degrees of freedom at 95% confidence as taken from table C.3 page 267 of Quality Assurance of Chemical Measurements, John K. Taylor, Lewis Publishers Inc. 1987.

The other two PCNB degradates TCTASOO and PCTASO were identified after report RP-91051 was done so no formal estimate of accuracy for these materials

was done. However information can be extracted from data for the day spikes used in Uniroyal GLP study 92147. Appendix IV contains the data from this report for the period 5/25/93 to 3/15/94. The data for the day spikes was extracted and is summarized in Figure V for PCNB and all its degradates including TCTASOO and PCTASO. All recoveries are between the limits required by the EU and USA regulations.

M.2 Precision

The USA requires a calculation of the relative standard deviation of recoveries (RSD's) at various concentration levels. These RSD's are shown in Figure IV and V and are less or equal to 20% as required by EPA.

The EU requires a repeatability study where the same sample is used at least 5 times on the same instrument with the same operator within a short time interval. This data is shown in Figure IV for PCNB, PCB, HCB, PCP-OMe, PCA and PCTA at 0.005, 0.25, and 1.0 ppm. Data is also shown for these compounds at 3.0 and 10.0 ppm but unfortunately is for 4 rather than the minimum 5 days. The confidence levels at 95% are shown in Figure IV for each level tested. The data from Figure IV indicate that this analytical method has good repeatability.

M.3 Limit of Quantitation (USA) / Limit of Determination (EU)

The lowest concentration tested as shown in Figure IV was 0.005 ppm. At this level the mean recoveries for PCNB and all its degradates were all between 70 and 110% and the relative standard deviations were all equal or less than 20%. Thus the limit of quantitation (LOQ) is 0.005 ppm.

M.4 Limit of Detection

No statistical estimate of the limit of detection (LOD) was made from the data
in Figure IV. However if we assume that the LOD is roughly one-third of the
LOQ the LOD would be about 0.002 ppm. In this connection the
chromatographic traces for two sets of control soils and for these controls spiked
at 0.05 µg (0.005 ppm), 1µg (0.10 ppm) and 10 µg (1.0 ppm) can be
considered (Appendix III).

M.5 Specificity

This is a gas chromatographic method and as demonstrated by typical
chromatograms of the spikes (see Appendix II) there is excellent separation of
the PCNB and its degradates. In the soils tested there were no interfering
compounds. However it is recommended that confirmatory identification of the
peaks be occasionally carried out as was done in study 92147 (see Appendix V
and VI).

M.6 Ruggedness

No ruggedness testing was done but the GC/ECD method is generally
considered a reliable method.

M.7 Limitations

None are known.

M.8   Independent Laboratory Validation (ILV) (USA)/ Reproducability (EU)

Reproducability (EU) is defined as an independent lab validation.
Reproducability is <u>not</u> required for soil samples according to EU directive
91/414/EEC, July 16, 1996. An ILV is suggested by the USA EPA. This has
not been done in a formal sense. However several field dissipation studies for
PCNB in different USA locations have been done at various times. Although
the same laboratory analyzed the samples, the fact that these analyses were done
successfully over a number of years suggests that the method in this report can
be considered as having been independently lab validated.

H.   <u>CONCLUSIONS</u>

The analytical method AC-6000 described in this report is applicable to the analysis of
PCNB and its degradates in a variety of soil types. The LOD is about 0.002 ppm and
the LOQ is 0.005 ppm. Recoveries and relative standard deviations are excellent and
well within the regulatory guidelines of both the EPA and EU.

FIGURES

Pentachloronitrobenzene
(PCNB)

Pentachloroaniline
(PCA)

Pentachlorothioanisole
(PCTA)

Pentachlorophenol
(PCP)

Pentachlorobenzene
(PCB)

Hexachlorobenzene
(HCB)
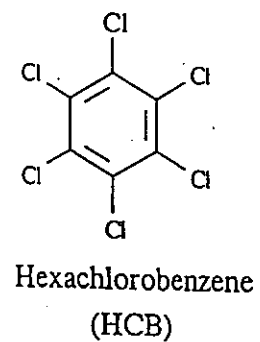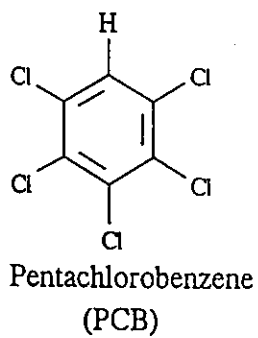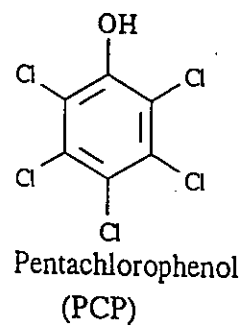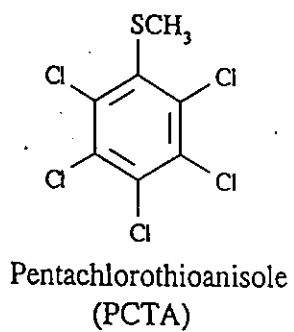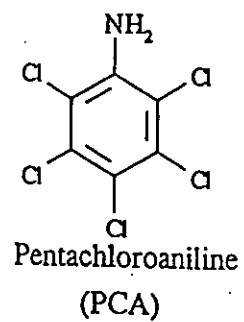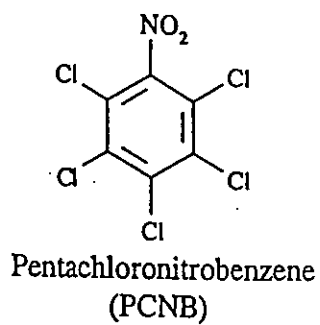
Figure I.   Structures and chemical names of PCNB and other analytes.
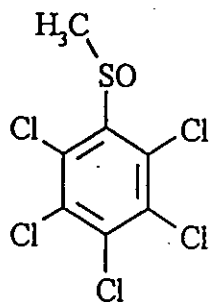
Pentachlorothioanisole sulfoxide

(PCTASO)
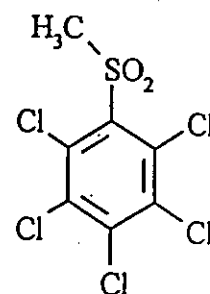
Pentachlorothioanisole sulfone

(PCTASOO)
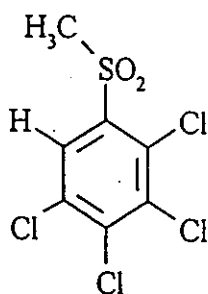
2,3,4,5-Tetrachlorothioanisole sulfone

(TCTASOO)

Figure I.   (Continued)
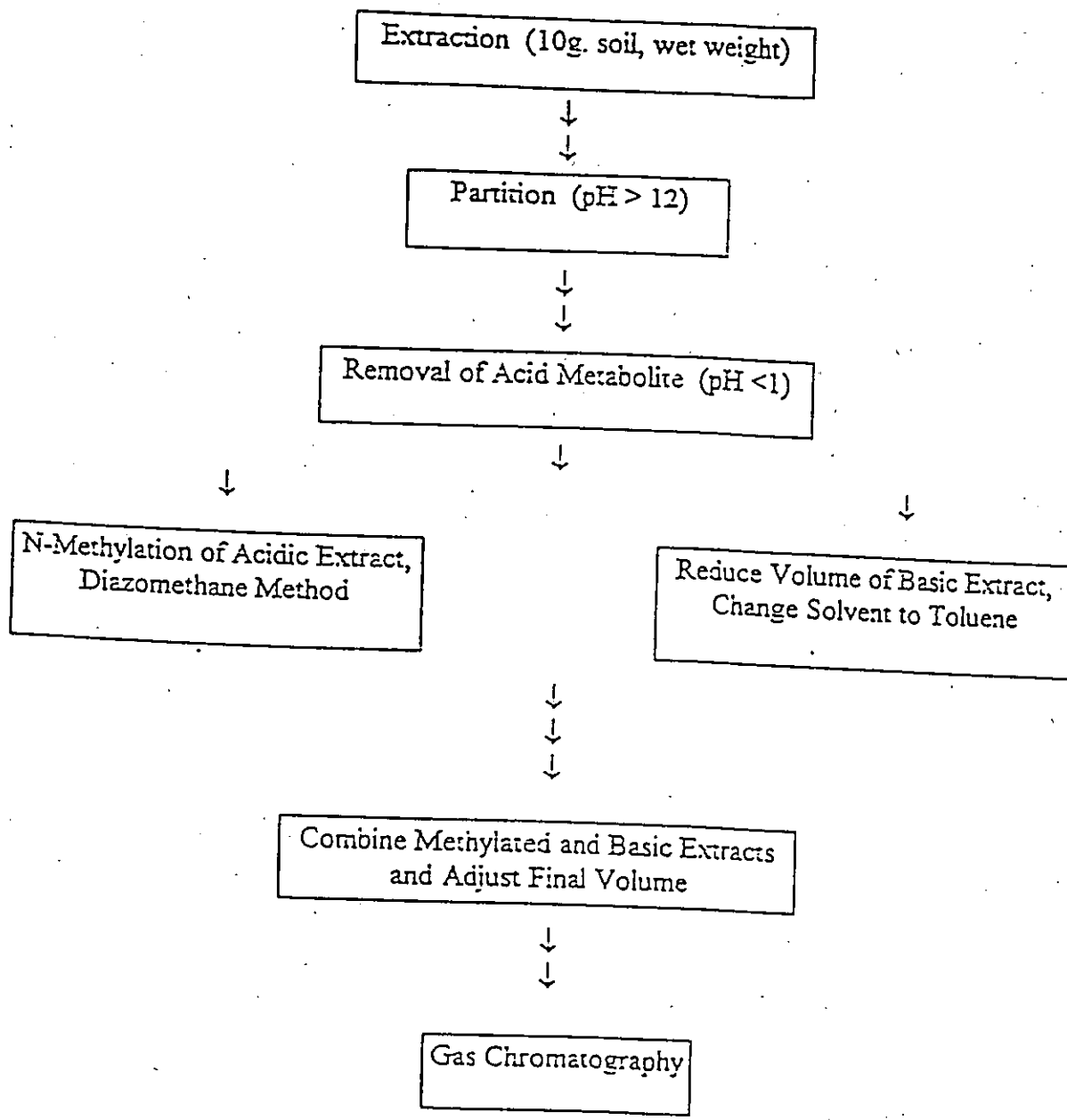
Figure II.   PCNB and Metabolites Analysis Method Flowchart

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Table VI. Field Spike Results from a PCNB Field Dissipation Study Conducted in Texas. Soil Samples Were Spiked with 1.0 ug of Standard Prior to Storage and Shipment.

	CONTROL	PCB	HCB	PCP (a)	PCNB	PCA	PCTA	TCTASOO	PCTASO
Month 0-3, Replicate A									
ug added	0.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
ug found	<0.005	0.910	1.044	1.208	0.820	0.870	0.826	0.894	0.878
% recovery	0	91	104	121	82	87	83	89	88
Avg. % Day Spike Recov.		88.0	102.0	94.5	81.0	86.5	96.0	95.5	95.0
Month 0-3, Replicate B									
ug added	0.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
ug found	<0.005	0.960	0.786	1.174	0.752	0.818	0.816	0.714	0.75
% recovery	0	96	79	117	75	82	82	71	75
Avg. % Day Spike Recov.		80.5	98.0	92.0	85.0	92.5	101.0	94.0	98.0
Month 6-12, Replicate A									
ug added	0.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
ug found	<0.005	0.636	0.574	0.662	0.862	0.644	0.674	0.734	0.488
% recovery	0	64	57	66	86	64	67	73	49
Avg. % Day Spike Recov.		83.0	88.0	101.0	92.5	96.0	90.0	92.5	76.5
Month 6-12, Replicate B									
ug added	0.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
ug found	<0.005	0.604	0.654	0.748	0.706	0.650	0.676	0.812	0.568
% recovery	0	60	65	75	71	65	68	81	57
Avg. % Day Spike Recov.		81.5	86.0	97.5	85.5	86.5	87.5	93.5	78.0

92147RES.WB2 A

Figure·III.

Validation Parameters for the Analysis of PCNB in Soil on Consecutive Days

File name: c:\msoffice\excel\pcnb.xls

% RECOVERY OF PCB

DATE	.005ppm	0.25ppm	1.0ppm	3.0ppm	10.0ppm
12/10/91	88.0	78.1	85.5		
12/10/91	86.2	86.7	82.2		
12/11/91	86.2	80.5	92.2		
12/11/91	84.2	92.5	91.6		
12/12/91	107.0	78.4	79.3		
12/12/91		96.7	91.5		
12/13/91	94.8	72.1	72.1		
12/13/91	98.6	89.6	95.2		
12/16/91	92.6	87.7	96.4		
12/16/91	95.4	90.2	87.8		
12/17/91	87.8	87.4	85.4		
12/17/91	90.2	89.5	93.8		
12/26/91	88.4	78.9		94.0	83.3
12/26/91	88.2	96.0		96.1	90.2
12/27/91	90.2	85.6		91.4	80.5
12/27/91	91.0	72.5		91.5	88.1
Average %	91.3	85.2	87.8	93.3	85.6
SD	5.8	7.6	7.2	2.2	4.4
RSD	6.4	8.9	8.2	2.4	5.2
Range	84.2 to 107.0	72.1 to 96.7	72.1 to 96.4	91.4 to 96.1	80.5 to 90.2
95 % Confidence	3.2	4.1	4.6	3.6	7.0

% RECOVERY OF HCB

DATE	.005ppm	0.25ppm	1.0ppm	3.0ppm	10.0ppm
12/10/91	86.4	88.5	90.9		
12/10/91	91.4	95.6	91.8		
12/11/91	94.2	91.8	92.2		
12/11/91	94.0	100.9	96.0		
12/12/91	111.0	88.5	91.0		
12/12/91		100.9	97.2		
12/13/91	102.2	85.9	85.2		
12/13/91	104.6	95.1	99.9		
12/16/91	110.4	92.9	100.3		
12/16/91	102.8	97.4	92.1		
12/17/91	100.2	93.8	93.0		
12/17/91	102.8	95.4	98.5		
12/26/91	101.0	97.7		102.3	97.7
12/26/91	102.6	104.0		101.7	97.2
12/27/91	99.6	97.0		97.1	97.0
12/27/91	118.8	85.3		96.0	98.2
Average %	101.5	94.4	94.0	99.3	97.5
SD	8.1	5.4	4.5	3.2	0.5
RSD	8.0	5.7	4.7	3.2	0.6
Range	86.4 to 118.8	85.3 to 104.0	85.2 to 100.3	96.0 to 102.3	97.0 to 98.2
95 % Confidence	4.5	2.9	2.8	5.1	0.9

% RECOVERY OF PCP-OMe

DATE	.005ppm	0.25ppm	1.0ppm	3.0ppm	10.0ppm
12/10/91	93.4	80.4	72.3		
12/10/91	101.4	85.4	77.5		
12/11/91	101.8	90.1	86.7		
12/11/91	92.8	80.8	84.0		
12/12/91	109.0	77.8	91.0		
12/12/91		92.5	92.7		
12/13/91	100.8	73.5	72.4		
12/13/91	100.8	86.0	87.0		
12/16/91	92.4	82.6	90.2		
12/16/91	89.8	82.5	83.0		
12/17/91	86.6	84.9	83.6		
12/17/91	91.8	85.1	87.3		
12/26/91	103.2	103.2		101.0	95.2
12/26/91	98.2	98.4		104.1	96.0
12/27/91	100.8	88.3		91.5	94.7
12/27/91	99.2	77.4		92.3	92.7
Average %	97.5	85.5	84.0	97.2	94.7
SD	6.0	7.7	6.8	6.3	1.4
RSD	6.2	9.0	8.1	6.5	1.5
Range	86.6 to 109.0	73.5 to 103.2	72.3 to 92.7	91.5 to 104.1	92.7 to 96.0
95 % Confidence	3.3	4.1	4.3	10.0	2.2

% RECOVERY OF PCNB

DATE	.005ppm	0.25ppm	1.0ppm	3.0ppm	10.0ppm
12/10/91	92.4	89.6	91.8		
12/10/91	88.8	95.5	95.4		
12/11/91	99.4	97.8	101.2		
12/11/91	99.4	103.9	100.4		
12/12/91	112.0	96.6	95.6		
12/12/91		105.7	101.1		
12/13/91	112.2	97.0	81.4		
12/13/91	114.2	102.2	105.6		
12/16/91	105.0	97.8	103.8		
12/16/91	100.4	103.0	95.6		
12/17/91	96.0	100.3	98.4		
12/17/91	101.2	100.7	102.9		
12/26/91	102.2	105.6		108.2	100.4
12/26/91	108.6	111.9		106.2	101.4
12/27/91	97.6	102.5		100.0	98.2
12/27/91	103.6	93.7		98.1	99.8
Average %	102.2	100.2	97.8	103.1	100.0
SD	7.3	5.4	6.6	4.8	1.3
RSD	7.1	5.4	6.7	4.7	1.3
Range	88.8 to 114.2	89.6 to 111.9	81.4 to 105.6	98.1 to 108.2	98.2 to 101.4
95 % Confidence	4.0	2.9	4.2	7.7	2.1

% RECOVERY OF PCA

DATE	.005ppm	0.25ppm	1.0ppm	3.0ppm	10.0ppm
12/10/91	104.0	95.5	94.8		
12/10/91	105.6	99.0	98.0		
12/11/91	89.0	101.4	104.1		
12/11/91	104.6	104.1	100.2		
12/12/91	79.2	98.5	99.2		
12/12/91		104.4	101.1		
12/13/91	84.6	99.9	97.1		
12/13/91	83.6	100.1	106.0		
12/16/91	96.4	99.2	100.4		
12/16/91	118.8	101.3	95.9		
12/17/91	110.6	101.1	99.5		
12/17/91	116.4	99.5	101.2		
12/26/91	100.0	106.0		106.9	102.2
12/26/91	111.4	108.3		104.9	102.2
12/27/91	98.6	101.3		99.9	98.6
12/27/91	111.2	98.3		98.2	100.5
Average %	100.8	101.1	99.8	102.5	100.9
SD	12.1	3.2	3.2	4.1	1.7
RSD	12.0	3.2	3.2	4.0	1.7
Range	79.2 to 116.8	95.5 to 108.3	94.8 to 106.0	98.2 to 106.9	98.6 to 102.2
95 % Confidence	6.7	1.7	2.0	6.5	2.8

% RECOVERY OF PCTA

DATE	.005ppm	0.25ppm	1.0ppm	3.0ppm	10.0ppm
12/10/91	90.6	96.7	94.4		
12/10/91	88.6	99.3	95.7		
12/11/91	95.8	101.2	103.6		
12/11/91	99.6	104.0	101.5		
12/12/91	105.6	99.3	99.3		
12/12/91		105.0	101.4		
12/13/91	103.8	101.0	98.0		
12/13/91	105.2	101.6	106.9		
12/16/91	110.8	100.6	103.5		
12/16/91	103.4	104.3	96.1		
12/17/91	99.8	102.0	100.5		
12/17/91	104.2	100.6	102.9		
12/26/91	102.0	106.4		107.2	101.0
12/26/91	101.0	107.7		105.4	101.3
12/27/91	98.6	102.1		99.8	98.0
12/27/91	97.2	98.1		97.9	99.9
Average %	100.4	101.9	100.3	102.6	100.0
SD	5.8	3.0	3.7	4.4	1.5
RSD	5.8	2.9	3.7	4.3	1.5
Range	88.6 to 110.8	96.7 to 107.7	94.4 to 106.9	97.9 to 107.2	98.0 to 101.3
95 % Confidence	3.2	1.6	2.4	7.1	2.4

Figure IV

Validation Parameters for the Analysis of PCNB in Soil

File name: c:\msoffice\excel\pcnbanal.xls

DATE	PCB % RECOVERY 0.1ppm	PCB % RECOVERY 1.0ppm	HCB % RECOVERY 0.1ppm	HCB % RECOVERY 1.0ppm	PCP-OMe % RECOVERY 0.1ppm	PCP-OMe % RECOVERY 1.0ppm	PCNB % RECOVERY 0.1ppm	PCNB % RECOVERY 1.0ppm
5/25/93	85.0	85.2	89.6	93.4	94.0	98.8	88.2	93.6
5/25/93	86.8	85.0	91.4	93.4	96.6	96.4	92.2	93.4
8/24/93	93.8	95.6	100.8	100.2	107.8	114.4	100.4	110.8
8/25/93	82.6	87.8	100.0	93.0	108.6	114.2	101.0	99.2
1/11/94	91.2	99.4	108.4	105.2	103.8	103.4	102.0	113.0
1/11/94	94.4	96.8	114.2	105.4	101.2	101.6	109.0	109.2
1/12/94	85.8	97.2	109.0	105.0	101.8	103.0	101.6	107.2
1/12/94	88.2	94.0	108.2	100.2	100.2	95.2	115.2	100.4
3/14/94	100.0	100.0	113.5	110.6	98.0	110.4	97.8	108.8
3/14/94	93.2	96.2	114.2	104.8	104.8	108.2	97.2	104.2
3/15/94	92.0	93.8	104.4	100.0	105.4	102.4	94.0	98.6
3/15/94	87.4	98.2	109.8	104.4	107.4	106.8	103.8	104.0
Average %	90.0	94.1	105.3	101.3	102.5	104.6	100.2	103.5
SD	4.9	5.3	8.3	5.7	4.7	6.4	7.3	6.5
RSD	5.5	5.6	7.9	5.6	4.6	6.1	7.3	6.3
Range	82.6 to 100.0	85.0 to 100.0	89.6 to 114.2	93.0 to 110.6	94.0 to 108.6	95.2 to 114.4	88.4 to 115.2	93.4 to 113.0
95 % Confidence	3.1	3.3	5.3	3.6	3.0	4.0	4.6	4.2

DATE	PCA % RECOVERY 0.1ppm	PCA % RECOVERY 1.0ppm	PCTA % RECOVERY 0.1ppm	PCTA % RECOVERY 1.0ppm	TCTASOO % RECOVERY 0.1ppm	TCTASOO % RECOVERY 1.0ppm	PCTASO % RECOVERY 0.1ppm	PCTASO % RECOVERY 1.0ppm
5/25/93	86.2	95.0	87.8	94.6	94.4	95.8	92.4	92.0
5/25/93	89.0	94.2	90.6	94.4	97.8	94.4	92.0	90.0
8/24/93	111.2	99.4	83.8	88.8	92.6	103.0	110.2	112.2
8/25/93	98.0	92.6	92.6	91.6	116.4	91.6	101.6	98.2
1/11/94	94.8	99.0	88.8	87.4	109.0	99.0	114.2	105.0
1/11/94	101.8	102.4	93.8	89.6	116.8	108.8	113.2	113.0
1/12/94	96.6	97.8	87.6	92.2	114.4	102.4	102.6	89.4
1/12/94	91.6	92.2	84.0	87.2	106.8	94.8	92.4	78.4
3/14/94	94.0	113.2	101.6	107.4	113.2	108.2	97.0	103.4
3/14/94	99.6	108.0	100.8	104.2	105.4	113.2	99.6	111.8
3/15/94	96.8	99.6	87.6	93.0	83.4	94.6	80.4	110.4
3/15/94	101.0	107.2	92.8	95.4	112.8	106.4	93.0	118.4
Average %	96.7	100.1	91.0	93.8	105.3	101.0	99.1	101.9
SD	6.5	6.6	5.7	6.3	10.8	7.0	10.0	12.2
RSD	6.8	6.6	6.3	6.7	10.3	6.9	10.1	12.0
Range	86.2 to 111.2	92.2 to 113.2	83.8 to 101.6	87.2 to 107.4	83.4 to 116.8	91.6 to 113.2	80.4 to 114.2	78.4 to 118.4
95 % Confidence	4.2	4.2	3.6	4.0	6.9	4.4	6.4	7.8

Figure V

APPENDICES

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