FINAL REPORT

Polychlorinated Biphenyls Analysis in Bituminous Materials

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Project 82-1

JHR 84-160 August 1984

This research was sponsored by the Joint Highway Research Advisory Council of the University of Connecticut and the Connecticut Department of Transportation and was carried out in the Civil Engineering Department of the University of Connecticut.

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KEY TO ACRONYMS

EPA - United States Environmental Protection Agency

g - Gram, a measure at mass

GC - Gas chromatography

GC/MS - Gas Chromatography/Mass Spectroccopy

K-D - Kuderna-Danish evaporative concentration

mg/l - Milligram per liter, mass per unit volume measure of concentration

PCB - Polychlorinated Biphenyl

ppb - Parts per billion, mass per unit mass measure of concentration

ppm - Parts per million, mass per unit mass measure of concentration

pp'-DDE - Dichlorodiphenyl dichloroethylene

RRT - Relative retention time

RT - Retention time

TOSCA - Toxic Substances Control Act

ug/l - Microgram per liter, mass per unit volume measure of concentration

INTRODUCTION

Concern has been expressed that polychlorinated biphenyls (PCBs) and possibly other toxic substances may have been improperly disposed of in fuel and waste oils, subsequently finding their way into bituminous materials in Connecticut.[1] Because PCBs are soluble in hydrocarbon solvents and oils, the high cost of disposal and the present non-hazardous classification of waste oil in Connecticut, their presence in bituminous materials is possible. PCBs are known to be ubiquitous in the environment. It is also possible, therefore, for trace PCB contamination in bituminous materials to be the result of background PCB levels from non-specific sources.

The purpose of this research project is two-fold: 1. To develop a qualitative and quantitative method of PCB analysis in bituminous materials, and 2. To determine whether PCBs are present in bituminous materials from selected sampling sites in Connecticut.

BACKGROUND

Polychlorinated biphenyls are a group of synthetic aromatic chlorinated organic compounds which have found widespread use because of their general thermal stability, resistance to acids, bases and oxidants.[2] Common industrial applications include transformer cooling oils, capacitor dielectric fluids, heat transfer and hydraulic oils, dedusting agents, plasticizers in rubber, adhesives, wax extenders, carbonless reproducing paper, and inks.

PCB Production

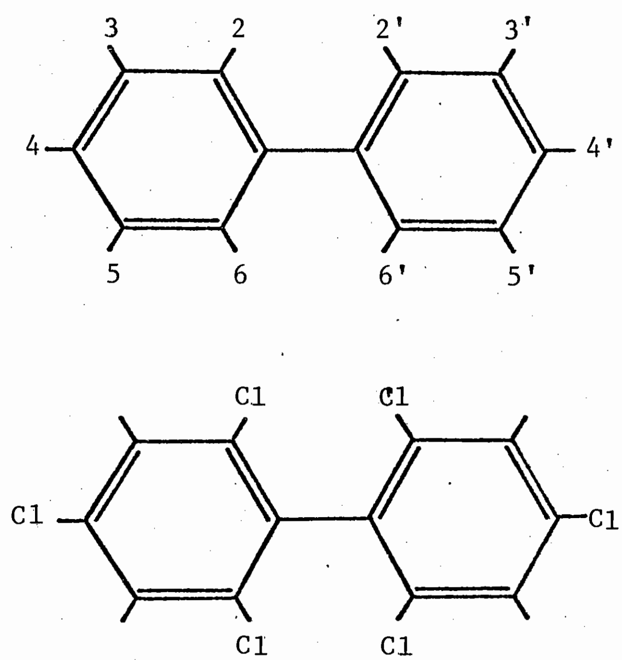
Between 1929 and 1977 about 1.9 billion pounds of PCB were produced in the United States.[2] In 1979, the Environmental Protection Agency (EPA) banned their manufacture and limited their use under provisions of the Toxic Substances Control Act (TOSCA). While they are no longer chemically manufactured, it was estimated that in 1982, 750 million pounds were still in use, 500 million pounds were in the environment and 150 million pounds were exported.[3] Of the 500 million pounds of PCBs in the environment, 300 million pounds were in landfills, 50 million pounds have been degraded and 150 million pounds remained in the air, water and sediments.[4]

The major manufacturer of PCBs in the U.S. was Monsanto. They marketed PCBs under the Aroclor[R] trade name.[2] Aroclor products are characterized by the name Aroclor followed by a four-digit number (e.g. Aroclor 1242), where the first two-digits (e.g. 12) represent a chlorinated biphenyl and the last two digits (e.g. 42) represent the percent chlorine by weight present. Monstanto produced approximately 10 various Aroclor mixtures. The current and former uses of PCB and the Aroclor grade are listed in Table 1.[2]

Chemistry of PCBs

Theoretically, 209 different chlorinated biphenyls can exist by chlorine substitution on the biphenyl structure. The basic biphenyl structure and substitution positions are shown in Figure 1.

GENERAL STRUCTURE AND SUBSTITUTION
OF A BIPHENYL



Chlorine substitution position number

and a partially chlorinated biphenyl is named as follows: 2,2',4,4',6,6' hexachlorobiphenyl

FIGURE 1

STRUCTURE AND SUBSTITUTION NOMENCLATURE
OF THE BIPHENYL MOLECULE

TABLE 1

Current and Former PCB Use and Specific Aroclors Used

Current PCB Use	Aroclors Used
Electrical Capacitors	1221, 1254
Electrical Transformers	1242, 1254, 1260
Vacuum Pumps	1248, 1254
Gas-transmission Turbines	1221, 1242
Former PCB Use	
Hydraulic Fluids	1232, 1242, 1248, 1254, 1260
Plasticizer in Synthetic Resins	1248, 1254, 1260, 1262, 1268
Adhesives	1221, 1232, 1242, 1248, 1254
Plasticizer in Rubbers	1221, 1232, 1242, 1248, 1254, 1268
Heat Transfer Systems	1242
Wax Extenders	1242, 1254, 1268
Dedusting Agents	1254, 1260
Pesticides, Extenders, Inks	1254
Lubricants, Cutting Oils	1254
Carbonless Reproducing Paper	1242

Each Aroclor mixture contains many individual clorinated biphenyls. For example, 32 individual chlorinated biphenyls have been identified in the Aroclor 1242, while in Aroclor 1260, 36 compounds have been identified.[2]

The importance of the distribution of individual PCBs is realized when examing water solubility, toxicity, adsorption on sediments and soils, volatility and bioaccumulation in tissue. Chlorobiphenyl distribution, by weight, in various Aroclor products is shown in Table 2. Water solubility for various Aroclor mixtures is also given in Table 2. Each of the 209 PCBs possible have individual water solubilities in water. It has been determined that water solubility decreases with the degree of chlorination on the biphenyl structure.[2]

Examples of water solubilities of individual PCB compounds are as follows:

1. 2 monchlorobiphenyl - 5.9 ppm;
2. 2,2' dichlorobiphenyl - 1.50 ppm;
3. 2,4,4' trichlorobiphenyl - 0.085 ppm;
4. 2,2', 4,4' tetrachlorobiphenyl - 0.068 ppm; and
5. 2,2', 4,4', 5,5' hexachlorobiphenyl - 0.0088 ppm.

Therefore, the solubility of a particular Aroclor mixture is predominantly the net result of the individual PCBs present in the mixture that have the fewest chlorines on the biphenyl structure. While PCBs are quite insoluble in water, their solubility in organic solvents and oils is substantially greater. Table 3 lists the solubility of Aroclor 1242 in various organic compounds.

PCB STANDARDS

Aquatic Standards

The Environmental Protection Agency has promulgated regulations for PCBs in drinking water supplies. To insure the maximum protection of human health from the

TABLE 2

General Chlorobiphenyl Composition of Select Aroclors

Chlorobiphenyl	Chemical Formula	Percent in Aroclor			
		1242	1248	1254	1260
monochlorobiphenyl	$C_{12}H_9Cl$	3			
dichlorobiphenyl	$C_{12}H_8Cl_2$	13	2		
trichlorobiphenyl	$C_{12}H_7Cl_3$	28	18		
tetrachlorobiphenyl	$C_{12}H_6Cl_4$	30	40	11	
pantachlorobiphenyl	$C_{12}H_5Cl_5$	22	36	49	12
hexachlorobiphenyl	$C_{12}H_4Cl_6$	4	4	34	38
heptachloropiphenyl	$C_{12}H_3Cl_7$			6	41
octachlorobiphenyl	$Cl_{12}H_2Cl_8$				8
nanochlorobiphenyl	$Cl_{12}HCl_9$				1
Average solubility in water		200 ppb	100 ppb	40 ppb	25 ppb

TABLE 3

Solubility of Aroclor 1242 in Organic Compounds*

Solvent	Solubility of Aroclor 1242 25°C	Solubility of Aroclor 1242 Hot
Acid		
Acetic Acid	S	S
Benzoic Acid	10.0 g/100 ml (31°C)	-
Chloro-derivatives		
Carbon Tetrachloride	S	S
Ethylene Dichloride	S	S
Monochloro benzene	S	S
Tetrochloroethane	S	S
Trichloroethane	S	S
Drying Oil		
Linseed Oil	S	S
Hydrocarbon		
Benzene	VS	VS
Gasoline	VS	VS
Kerosene	VS	VS
Mineral Spirits	VS	VS
Paraffin	2.0 g/100 ml (27.5°C)	S
Toluene	VS	VS
Xylene	VS	VS

*Note: S = Soluble; VS = Very soluble.

potential carcinogenic effects of PCB exposure through ingestion of contaminated water and aquatic organisms, the ambient water concentration should be zero.[7] For protection of freshwater aquatic life (i.e. stream standard for PCBs) the maximum allowable 24-hour average PCB concentration is 0.014 ug/l.

Commerical Standards

Under Subchapter R of the Toxic Substances Control Act (CFR 40.761.1), Part 761, Standards for Polychlorinated Biphenyl Manufacturing, processing, distribution in commerce, and use prohibitations have been established. In subpart A,761.1(b), in reference to the regulated levels of PCBs, it is stated that,

"...the terms PCB and PCBs are used in this rule to refer to any chemical substances and combination of substances that contain 50 ppm (on a dry-weight basis) or greater..."[8] of additional importance it is also stated that,

"Any chemical substances and combination of substances that contain less than 50 ppm PCBs because of any dilution, shall be included as PCB and PCBs unless otherwise specifically provided."[8]

In summary, unless a product has been specifically given a PCB standard, the standard that makes PCB a toxic waste, subject to the regulations of TOSCA is 50 ppm, in the most concentrated constituent of a waste material.

Of course, there is a degree of subjective reasoning which may be applied when interpreting these regulations. For example, if waste oil, contaminated with PCB greater than 50 ppm (on a dry-weight basis) is added to an asphalt binder (e.g. AC-20) and the resulting concentration is 5 ppm (on a dry-weight basis) in the AC-20, is the AC-20 now a PCB toxic wastes? If the AC-20 is blended with an aggregate to make an asphalt cement, is the asphalt cement now a PCB toxic waste? According to the letter of the law, perhaps the answer to this question is yes.

However, if the waste oil cannot be sampled (e.g. years after the asphalt was made) then how can it be proved, without reasonable doublt, that the waste oil did indeed contain PCBs at a concentration greater than 50 ppm? The answer is simply that it cannot be determined.

ENVIRONMENTAL FATE OF PCBs

The environmental fate of PCBs is a result of physical-chemical properties of PCBs and pertinent physical, chemical and biological processes that occur with PCBs in the environment. Important physical-chemical properties of various Aroclor mixtures are given in Table 4.

Factors affecting environmental fate of PCBs are as follows:

1. Photolysis,
2. Oxidation,
3. Hydrolysis,
4. Volatilization,
5. Sorption,
6. Bioaccumulation,
7. Biotransformation, and
8. Biodegradation.

An excellent review of the literature on the environmental fate of PCBs has been compiled by EPA.[10] There is evidence that photolysis by short-wave uV irradiation does result in effective dechlorination of PCBs. However, photolysis at normal ambient wave lengths is a very slow process. Therefore, photolysis is thought to be of minor importance to PCB distribution in the ambient environment.

PCBs have been shown to be extremely resistant to oxidation and both acid-base hydrolysis reactions.[10] Volatilization is thought to account for the ubiguitous

TABLE 4

Properties of Selected Aroclor Mixtures

Property	Units	Aroclor 1242	Aroclor 1254	Aroclor 1260
Appearance		Clear Oil	Light-yellow Viscous Liquid	Light-yellow Sticky Resin
Chlorine Density	% by wt. (g/cc) 25°C	42 1.35	54 1.50	60 1.58
Distillation Range °C (ASTM D-20 mod.)		325-366	365-390	385-420
Evaporation loss 100°C/6 hrs. (ASTM D-6 mod.)	% by wt.	0.0-0.4	0.0-0.2	0.0-0.1
Flash Point Cleveland Open Cup	°C	176-180	None to Boiling Pt.	None to Boiling Pt.
Pour Point ASTM #-97	°C	-19	10	31
Vapor Pressure	mm Hg at 25°C	4.06×10^{-4}	7.71×10^{-5}	4.05×10^{-5}

distribution of PCBs in the environment. Although PCBs have a high molecular weight and low vapor pressures, they exhibit high activity coefficients in water that result in a high equilibrium vapor pressures. Thus, volatilization of PCBs is considerably greater (i.e. from aqueous systems) than would be expected, and therefore, it is a significant factor affecting environmental distribution.[11]

Sorption is the major non-destructive mechanism controlling the movement of PCBs in the environment. It has been demonstrated that PCBs are strongly sorbed onto glass, plastic, silts, clays, soils, sediments and suspended solids.[2,10] It has been reported, however, that the affinity of PCBs for oils is greater than the forces controlling sediment-PCB sorption processes.[12] Therefore, sorbed PCBs can be desorbed in the presence of hydrocarbon oils.

Biaccumulation in various terrestrial and aquatic organisms is reported to be in the order of 10^4 to 10^6.[10] PCB bioaccumulation increases with the number of chlorines present. Thus, the most toxic PCBs are also among those that exhibit the most significant bioaccumulation in organisms.

While biodegradation of mono, di, and trichlorobiphenyls has been demonstrated to be a major destructive mechanism, little evidence of substantial biodegradation of penta- and greater PCBs is available.[10] Therefore, biodegradation is an important destructive mechanism for PCBs, but it is limited to the lower chlorinated biphenyls.

Qualitative and Quantitative Analysis of PCBs

For purposes of qualitative and quantitative analysis for PCBs in a wide variety samples, gas chromatography is the most commonly used analytical methods. Other analytical methods for PCB determination include mass spectroscopy[13], infrared spectroscopy[13], ultraviolet spectroscopy[14], nuclear magnetic resonance (NMR) spectroscopy[2], and high-performance liquid chromatography[15]. The Environmental

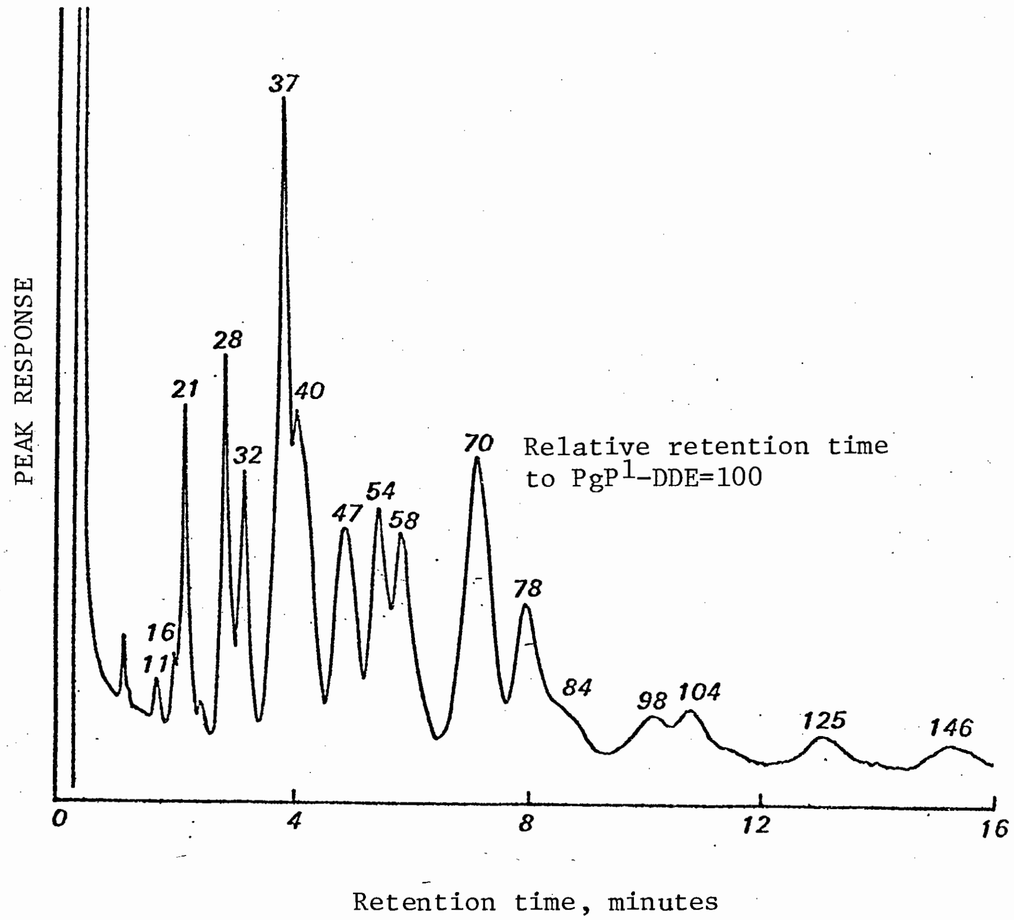
Protection Agency (EPA) has established an analytic test method for the the determination of PCBs in transformer fluid and waste oil that is based on packed column gas chromatography with electron capture detection.[16] The basis of the method, utilized a gas chromatograph with a 1/4" O.D., 6' long, column packed with Gas-Chrom Q 100/120 mesh and coated with 3% OV-1 maintained isothermal at 170°C. An electron capture detector (ECD) is used as the halide-specific detector.

Sample chromatograms generated from this method for Aroclor 1242, 1254, and 1260 are found in Figures 2, 3, and 4 respectively. Additional data in the Figures describes the mean weight percent, relative standard deviation and the number of chlorines as a function of the Relative Retention Time (i.e. time of compound elution relative to the compound PP'-DDE which has a RT=100 for the various compounds.

Peaks used for quantitation of Aroclor 1242 may consist of a 7-peak or 9-peak method of area integration. The 7-peak quantitatiion method represents the summation of area integration under peaks with relative retention times of 21, 28, 32, 37, 47, 54, and 58. The 9-peak quantitation includes all the peaks of the 7-peak method plus peak areas with relative retention times of 70 and 78.

Standard curves may be developed to use for the determination of PCB concentrations in samples by plotting ng (i.e. nanogram) PCB injected versus the 7 or 9 peak areas of integration. Both methods are reported in the analysis of this research.

For quantitation of Aroclor 1260 in this research, area integration of peaks with relative retention times of 70, 84, 104, 125, 146, 160, 174, 203, 232, 280, 332, and 372 are used.



Column: 1/4" O.D. x 6' long Stainless Steel

Packing: Gas Chrom Q 100/120 mesh with 3% OV-1 coating

Column Temperature: 170°C, isothermal

Detector: Electron Capture

FIGURE 2

SAMPLE EPA CHROMATOGRAM OF

AROCLOR 1242

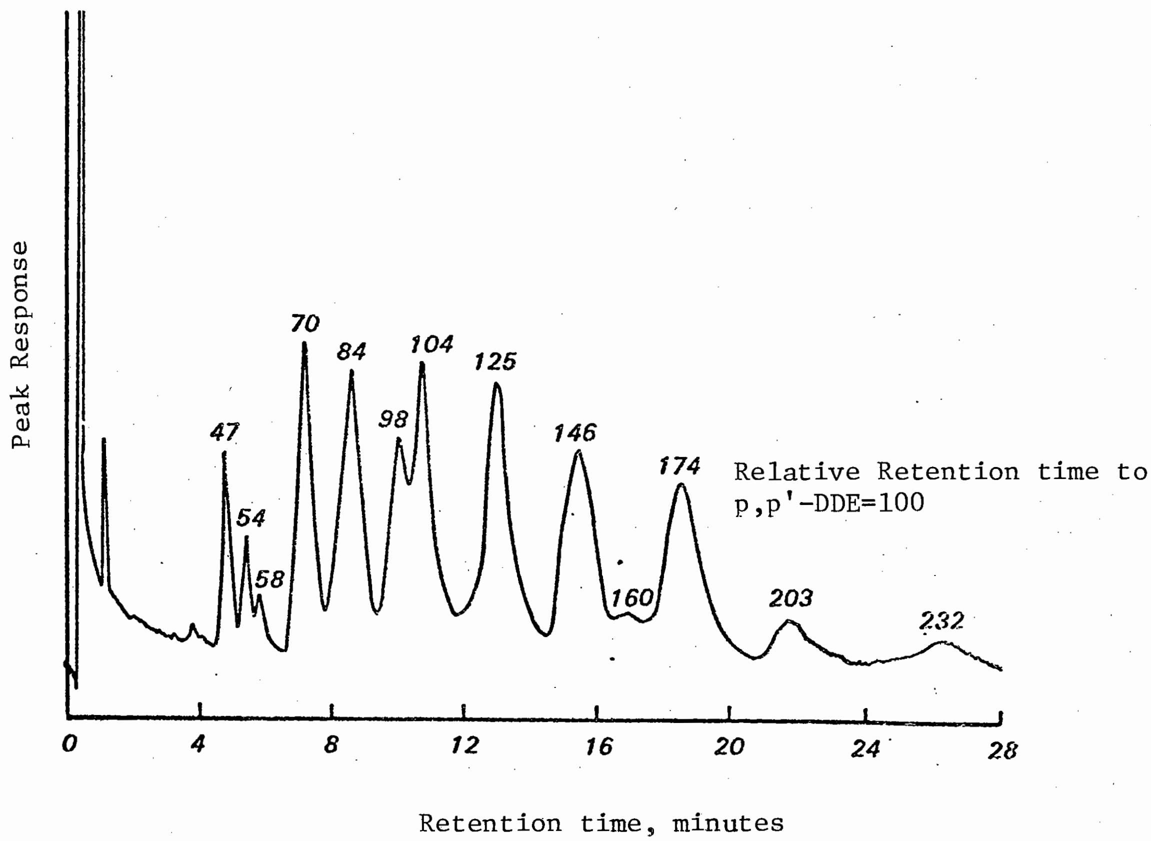
FIGURE 2 (cont'd)

SAMPLE EPA CHROMATOGRAM OF

AROCLOR 1242

RRT[2]	Mean Weight Percent	Relative Std. Dev.[3]	Number of Chlorines[4]
11	1.1	35.7	1
16	2.9	4.2	2
21	11.3	3.0	2
28	11.0	5.0	2 25% 3 75%
32	6.1	4.7	3
37	11.5	5.7	3
40	11.1	6.2	3
47	8.8	4.3	4
54	6.8	2.9	3 33% 4 67%
58	5.6	3.3	4
70	10.3	2.8	4 90% 5 10%
78	3.6	4.2	4
84	2.7	9.7	5
98	1.5	9.4	5
104	2.3	16.4	5
125	1.6	20.4	5 85% 6 15%
146	1.0	19.9	5 75% 6 25%
TOTAL	98.5		

1. Data obtained from Webb and McCall.[6]
2. Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.
3. Relative standard deviation of six analyses (as percentages of the mean of the results).
4. From GC/MS data, peaks containing mixtures of isomers of different chlorine numbers are bracketed.



Column: 1/4" O.D. X 6' long stainless steel

Packing: Gas Chrom Q 100/120 mesh with 3% OV-1 coating

Column Temperature: 170°C, isothermal

Detector: Electron Capture

FIGURE 3

SAMPLE EPA CHROMATOGRAM OF

AROCLOR 1254

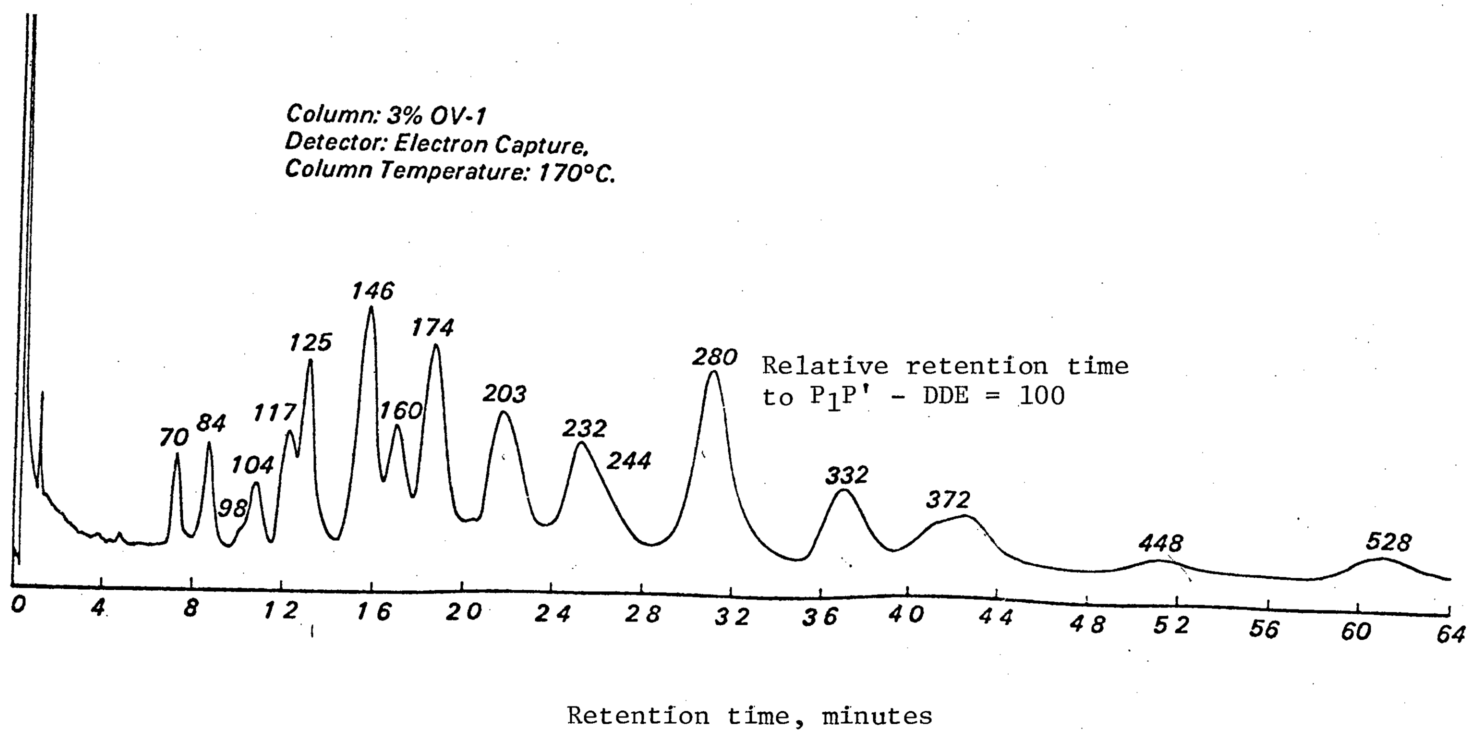
FIGURE 3 (cont'd)

SAMPLE EPA CHROMATOGRAM OF

AROCLOR 1254

RRT[2]	Mean Weight Percent	Relative Std. Dev.[3]	Number of Chlorines[4]
47	6.2	3.7	4
54	2.9	2.6	4
58	1.4	2.8	4
70	13.2	2.7	4 25% 5 75%
84	17.3	1.9	5
98	7.5	5.3	5
105	13.6	3.8	5
125	15.0	2.4	5 70% 6 30%
146	10.4	2.7	5 30% 6 70%
160	1.3	8.4	6
174	8.4	5.5	6
160	1.3	8.4	6
174	8.4	5.5	6
203	1.8	18.6	6
232	1.0	26.1	7
TOTAL	100.0		

1. Data obtained from Webb and McCall.[6]
2. Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.
3. Relative standard deviation of six analyses (as percentages of the mean of the results).
4. From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.



Column: 1/4" O.D. x 6' long Stainless Steel

Packing: Gas Chrom Q 100/120 mesh with 3% OV-1 coating

Column Temperature: 170°C, isothermal

Detector: Electron Capture

FIGURE 4

SAMPLE EPA CHROMATOGRAM OF
AROCLOR 1260

FIGURE 4 (cont'd)

SAMPLE EPA CHROMATOGRAM OF

AROCLOR 1260

RRT[2]	Mean Weight Percent	Relative Std. Dev.[3]	Number of Chlorines[4]
70	2.7	6.3	5
84	4.7	1.6	5
98	3.8	3.5	5
104			5 60% 6 40%
117	3.3	6.7	6
125	12.3	3.3	5 15% 6 85%
146	14.1	3.6	6
160	4.9	2.2	6 50% 7 50%
174	12.4	2.7	6
203	9.3	4.0	6 10% 7 90%
232			6 10%
244	9.8	3.4	7 90%
280	11.0	2.4	7
332	4.2	5.0	7
372	4.0	8.6	8
448	.6	25.3	8
528	1.5	10.2	8
TOTAL	98.6		

1 Data obtained from Webb and McCall.[6]
2 Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent. Overlapping peaks that are quantified as one peak are bracketed.
3 Relative standard deviation of six analyses (as percentages of the mean of the results).
4 From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.
5 Composition determined at the center of peak 104.
6 Composition determined at the center of peak 232.

METHODS AND MATERIALS

Apparatus

1. Gas Chromatograph - A Perkin-Elmer Sigma 1 Gas Chromatograph with a 3% OV-101 on Chromosorb W-HP 1/8" x 6' stainless steel packed column and Electron Capture Detector (ECD) was used for PCB determination. The gas chromatograph was set to attain the following conditions:

    1. Injector temperature - 250°C;
    2. Oven temperature - 180°C, isothermal;
    3. Detector temperature - 300°C;
    4. Carrier gas - 5% Methane in Argon;
    5. Carrier gas flowrate - 20 cc/min;
    6. Make-up gas flowrate - 40 cc/min;
    7. Detector Attenuation - 2.

2. Kuderna-Danish Evaporative Concentrator Apparatus:

A K-D Evaporative Eoncentrator equipped with a 10 ml graduated concentrator tube (Kontes K-570050-1025), a 500 ml evaporative flask (Kontes 57001-0500) and a Synder 3-ball column (Kontes K 503000-0121) was used to concentrate extracted PCBs in hexane.

3. Sample Containers

Prepared extracted samples were stored in washed 40 ml screw-cap vials with pesticide grade teflon cap liners, triple-rinsed with hexane.
PCB extraction from sample matrices was done in 250 ml screw cap Erylenmyer Flasks with aluminum foil cap liners.

4. Microsyringes

Hamilton 5 ul microsyringes (Model 75 RN) were used for sample injection and standard preparation.

5. Electron Balance - Pure 1242 was weighed out on Mettler Electronic Balance and diluted to known volume with hexane.

5. Filter Flask

A 25 mm glass filtering apparatus with a 500 ml side-arm erlenmeyer receiving flask was used for hexane extraction of asphalt binder from aggregate in asphalt cement and oil samples.

Reagents and Materials

1. Fischer pesticide grade hexane and iso-octane solvents were used. Quality of the solvents was periodically checked by injection into the gas chromatograph as a blank run, after concentration in the K-D apparatus.

2. Florisil - Florisil PR grade was used for sample clean-up, after activation at 130°C for a minimum of 12 hours.

3. Sulfuric Acid - American Chemical Society (A.C.S.) Grade Sulfuric Acid was used for acid clean-up.

4. PCB Standards - Both EPA primary standards (pure compound) and Analabs PCB standards (1000 mg/l and 1 mg/l) were used as primary standards for development of calibration curves.

5. Gas Chromatograph Columns - Both packed and fused-silica capillary columns were used in this study. The packed column was 3% OV-101 on Chromosorb W-HP, 1/8" x 6' stainless steel. The capillary column was an OV-101 fused-silica WCOT column, 0.25 mm I.D. x 50 m.

6. Filters - 25 mm Glass-fiber filters were used for separation of hexane extract from aggregate.

7. Mercury - ACS mercury was used for sample clean-up.

Glassware Washing Procedure

Glassware, seals and sample containers were washed to avoid trace PCB contamination in accordance with the following procedure:

1. detergent wash with Alconox detergent in tap water,
2. rinse 5 times with tap water,
3. chromic acid wash,
4. rinse 5 times with Milli-Q water,
5. oven dry at 150°C for minimum of 4 hours,
6. triple rinse with hexane, and
7. air dry while inverted.

Experimental Procedure

Asphalt samples, as received from representatives of the Connecticut Department of Transportation, consisted of pavement cores 4 inches in diameter approximately 1.5 inches thick, surface scrapings of pavement overlays (i.e. sealer and aggregate), and a quantity of AC-20 asphalt oil.

Pavement Sample Preparation

Approximately 60 grams of pavement was removed from the sample core by chipping with a stainless steel chisel and weighed to the nearest 0.001 g. The weighed sample was placed in a 250 ml prepared erylenmeyer flask with a screw-top cap and an aluminum foil cap liner. Approximately 40 ml pesticide-grade hexane was added. The flask was sealed and then placed on reciprocating shaker table for 48 hours for asphalt extraction. The hexane-pavement mixture was then filtered using a weighed 25 mm diameter filtering apparatus with a glass fiber filter. The erylenmeyer flask was rinsed 5 times with hexane, each time filtering the rinsed mixture. This was followed by 5 hexane rinses of the filtering apparatus and filter, ensuring complete recovery of the extracted PCB and asphalt oil into the receiving filter flask. Materials retained on the glass fiber filter and the filter were dried at 105°C for 1 hour and then weighed.

Percent asphalt (i.e. binder) content was calculated as follows:

$$\text{percent binder content} = \frac{A-(B-C)}{A} \times 100 \quad (1)$$

where,

A = weight of initial sample in grams, $\pm$ 0.001 g

B = weight of filter and dried residue, grams $\pm$0.001 g

C = weight of dried filter paper, grams, $\pm$0.001 g.

Filtrate mixture (i.e. hexane, asphalt oil and PCBs) was then placed in a Kuderna-Danish evaporative concentrator apparatus over a steam bath at 100°C. The filtering flask was rinsed 5 times with hexane and the rinsed mixture then placed in the K-D evaporative concentrator after each rinse. The contents of the concentrator was evaporated to a maximum volume of 10 ml.

Clean-up (i.e. procedures to remove impurities that interfere with ECD detection) of the concentrated sample consisted of 3 sulfuric acid washes, Florisil slurry treatment and a mercury extraction to remove sulfur compounds. The Acid Clean-up consisted of placing 5.0 ml concentrated sulfuric acid into a 40 ml screw cap vial with a teflon-cap liner containing the 10 ml concentrated sample, and shaking for 1 minute. The liquid phases were allowed to separate and then the upper phase (i.e. sample in hexane) was transferred to another 40 ml sample vial. This procedure was repeated twice more for a total of 3 acid washes. The acid clean-up was followed by a Florisil slurry clean-up which consisted of adding 0.25 g activated Florisil to the 10 ml concentrated sample and shaking for 1 minute. After the Florisil settled the treated solution was decanted into a 40 ml vial with a teflon-lined screw cap. Sulfur compounds were removed with 0.5 ml elemental mercury additions. Mercury was added to the 10 ml sample, then shaken for 1 minute followed by decanting of the sample. The process was repeated until the mercury did not turn black. This indicated that sulfur removal was complete. The concentrated sample was ready for injection to the gas chromatograph after the mercury clean-up.

Asphalt Oil Preparation

Approximately 1 g of asphalt oil was introduced to 40 ml hexane in a 250 ml erlyenmeyer flask and placed on a reciprocating shaker for a 48 hour extraction period. With the exception of weighing the filtered material and filter paper, the procedures outline above for pavement samples was exactly followed for asphalt oil preparation.

Soil Sample Preparation

Approximately 60 g of soil or sediment was placed in 40 ml hexane and the procedures outlined for asphalt oil preparation were exactly followed.

Standardization Procedures

All standards were made with 100 percent pure Aroclor mixtures received from EPA. Standard stock solution of Aroclor 1242, 1248, 1254, and 1260 were made by weighing approximately 0.01 g of material within ± 0.0001 g and diluting to 10 ml in hexane. The concentration was calculated (e.g. 1000 ppm) and the standard was stored at 4^{o}C until used. Additionally, several EPA quality assurance standards were obtained, at working concentration levels. Serial dilutions of the stock solution were made resulting in working standards at levels of 0.5 ppm, 1.0 ppm, 5.0 ppm, 10.0 ppm, 20.0 ppm and 40.0 ppm.

RESULTS

Standard Curve Development

Serial injections of the prepared Aroclor 1242 standards (e.g. 1.0 to 2.0 ul sample volume) were made to the gas chromatograph for the development of a working standard curve. Based upon the volume injected and the concentration of the standard, the mass of Aroclor 1242 injected was calculated. Based upon the 7 peak quantitation method as previously described, integration areas for each peak were recorded and the cumulative 7 peak integration area was calculated. A typical chromatogram of an Aroclor 1242 standard with raw data report is given in Figure 5. The 7 peaks and the 9 peaks used for quantitation are noted on the chromatogram. Comparison of the Aroclor 1242 chromatogram developed by EPA shown in Figure 2 with the chromatograms developed in this research shows good agreement.

Results of the standard curve analysis for Aroclor 1242 are listed in Table 5. In general, three or four injections at specific concentrations of 0.5 ppm, 1.0 ppm, 5.0 ppm, 10.0 ppm, 20.0 ppm and 40.0 ppm were made. Both cumulative integration areas for the 7 peak and 9 peak areas are given in Table 5. A plot of the standard curve is shown in Figure 6 for the 7 peak quantitation method with the 95 percent confidence interval about the mean. The least-squares linear regression equation for the curve in Figure 6 is as follows:

$$PKAC7 = 4.59 + 20.00 \, (NG) \qquad (2)$$

where,

PKAC7 = cumulative integration area for 7 Aroclor 1242 peaks

NG = nanograms (10^{-9}g) injected

Description statistics for the predicted equation are as follows:

R^2 = 0.986 (coefficient of determination)

coefficient of variance = 12.92

The equation for the 9-peak quantitation for the standard curve is as follows and a plot is given in Figure 7.

$$PKAC9 = 7.35 + 26.36 (NG) \quad (3)$$

where,

PKAC9 = cumulative integration area for the 9 Aroclor 1242 peaks

NG = nanograms (10^{-9}g) injected

The equations had the following descriptive statistics:

R^2 = 0.985

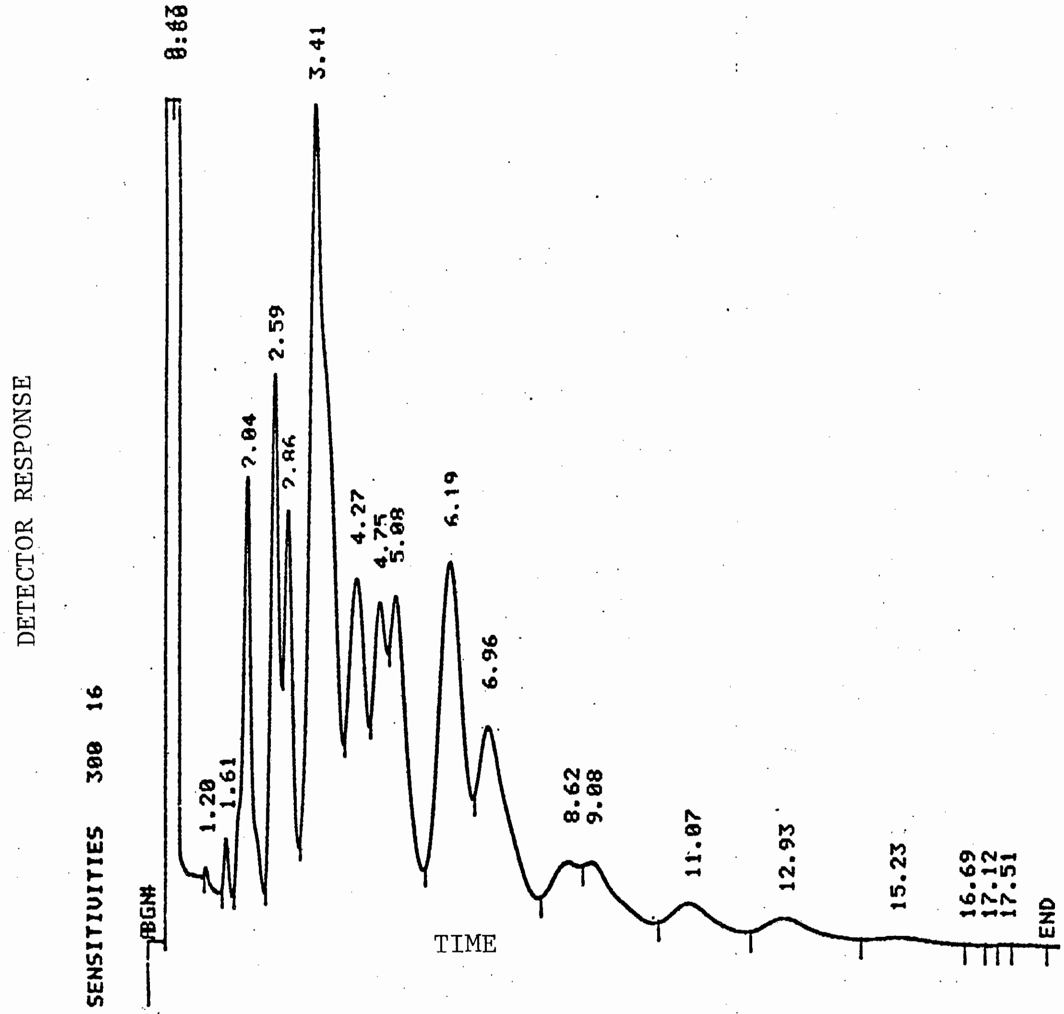
coefficient of variance = 13.04

Examination of the relationship between the 7-peak and 9-peak area integration areas for the Aroclor 1242 standard curve by least square linear regression analysis resulted in the following equation:

$$PKAC7 = 0.92 + 0.758(PKAC9) \quad (4)$$

Descriptive statistics for equation 4 indicate an R^2 = 0.999 and a coefficient of variance = 0.627.

A sample chromatogram for Aroclor 1260 is given in Figure 8. Quantitation was based upon 12 peaks as presented in Figure 4. Examination of the sample chromatogram in Figure 8 exhibits a very close fingerprint to the EPA chromatogram of Figure 4. Quantitation of Aroclor 1260 was based on a 3-point standard curve developed on each day of analysis. Coefficients of determination were consistently greater than 0.99, and as a result were within the linear range of the ECD detector.



DATA ANALYSIS REPORT

TIME	AREA	BC	RRT	RF	C
0.43	74.6137	T	0.043	1.000	15.2794
0.60	144.4454	T	0.060	1.000	29.5795
1.20	3.3510	T	0.120	1.000	0.6862
1.61	2.6825	T	0.161	1.000	0.5493
2.04	16.3628	T	0.204	1.000	3.3508
2.59	16.4384	T	0.259	1.000	3.3662
2.86	13.9187	T	0.286	1.000	2.8503
3.41	60.4800	T	0.341	1.000	12.3851
4.27	22.2054	T	0.427	1.000	4.5472
4.75	16.6278	T	0.475	1.000	3.4050
5.08	23.1091	T	0.508	1.000	4.7323
6.19	33.2889	T	0.619	1.000	6.8169
6.96	25.8278	T	0.696	1.000	5.2890
8.62	8.3347	T	0.862	1.000	1.7068
9.08	11.9513	T	0.908	1.000	2.4474
11.07	7.6009	T	1.107	1.000	1.5565
12.93	5.4662	T	1.293	1.000	1.1194
15.23	1.5696	V	1.523	1.000	0.3214
16.69	0.0172	V	1.669	1.000	0.0035
17.12	0.0133	V	1.712	1.000	0.0027
17.51	0.0249	V	1.751	1.000	0.0051

FIGURE 5

ELECTRON CAPTURE DETECTOR CHROMATOGRAM OF
AROCLOR 1242

TABLE 5

AROCLOR 1242 STANDARD CURVE RESULTS

Trial	Nanogram PCB Injected	Cumulative 9-peak Area	Cumulative 7-peak Area
1	0.423	19.11	14.16
2	0.466	30.37	23.94
3	0.466	31.45	24.06
4	0.444	30.41	23.15
5	1.05	47.70	36.45
6	1.11	54.05	39.45
7	1.11	41.30	30.94
8	5.67	205.73	153.98
9	5.98	215.58	162.22
10	5.98	228.20	169.10
11	10.97	307.47	230.94
12	10.45	302.61	227.13
13	10.97	291.00	219.49
14	12.23	272.82	205.21
15	11.65	251.12	190.31
16	11.65	260.10	196.06
17	11.65	266.85	200.44
18	24.25	619.12	474.42
19	24.25	605.25	457.02
20	23.10	557.64	419.83
21	44.27	1056.74	803.34
22	44.27	1265.00	957.12
23	46.60	1269.36	960.10
24	46.60	1321.29	1002.25

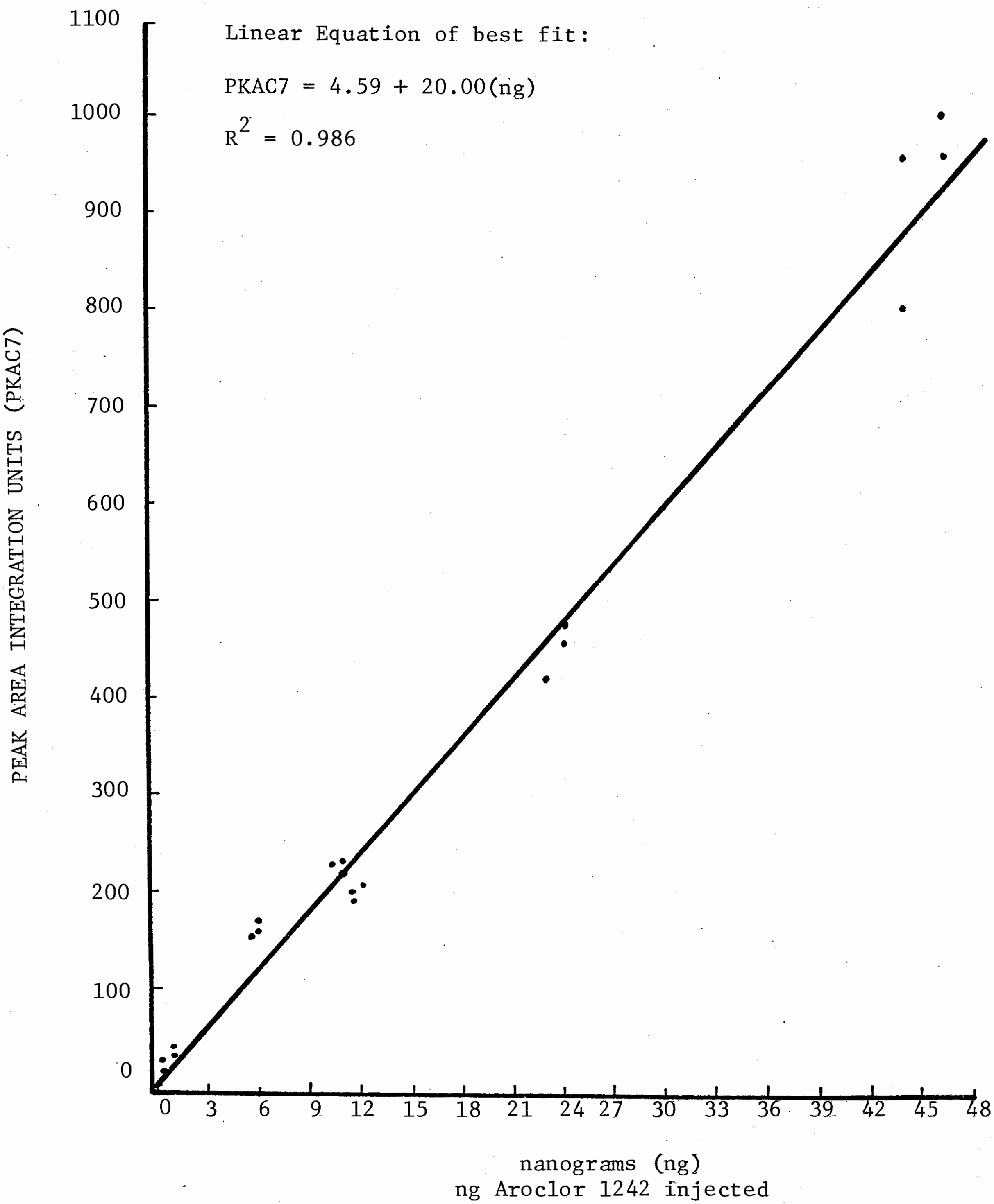


FIGURE 6

SEVEN-PEAK STANDARD CURVE FOR AROCLOR 1242

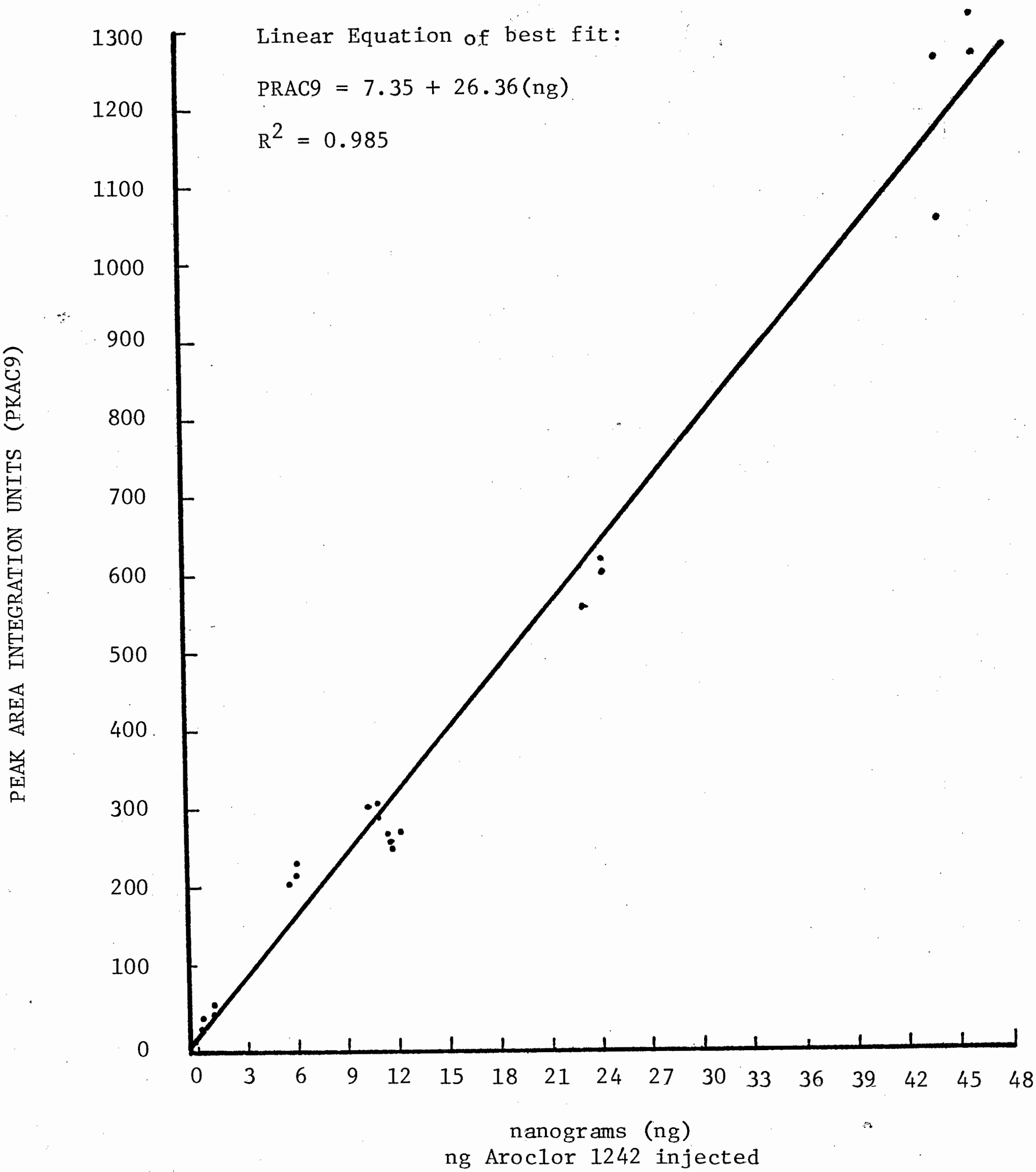
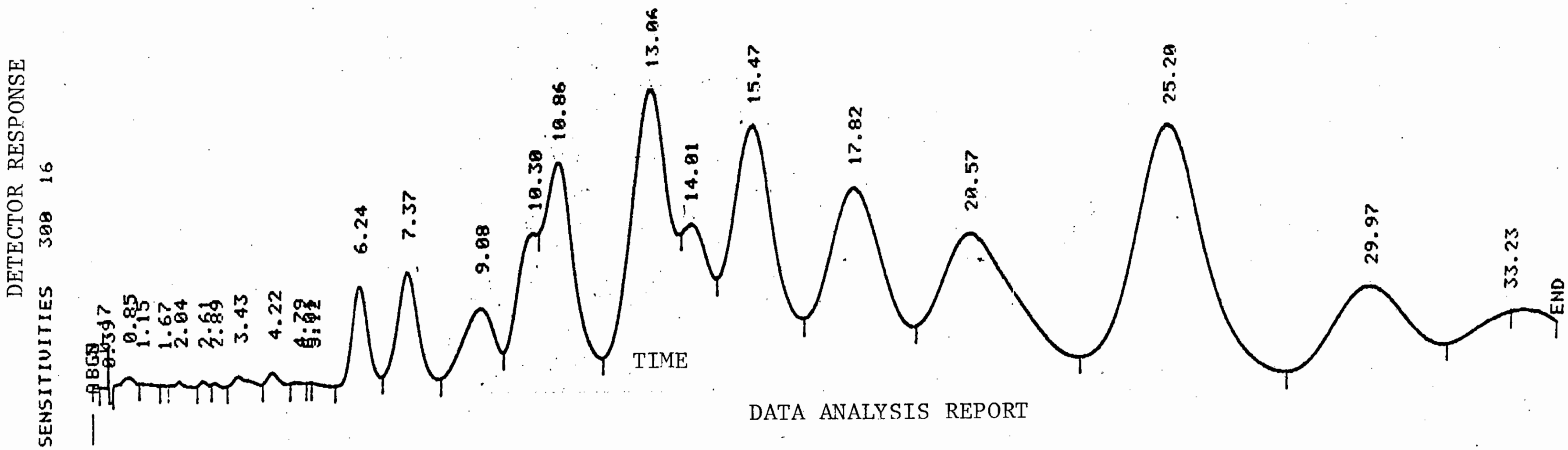


FIGURE 7

NINE-PEAK STANDARD CURVE FOR AROCLOR 1242



DATA ANALYSIS REPORT

TIME	AREA	BC	RRT	RF	C
0.17	0.5628	T	0.017	1.000	0.0671
0.39	0.3218	V	0.039	1.000	0.0384
0.85	3.8128	T	0.085	1.000	0.4544
1.15	2.7566	T	0.115	1.000	0.3285
1.67	0.9062	T	0.167	1.000	0.1080
2.04	3.2457	T	0.204	1.000	0.3868
2.61	1.8417	T	0.261	1.000	0.2195
2.89	1.6769	T	0.289	1.000	0.1999
3.43	4.8774	T	0.343	1.000	0.5813
4.22	3.7118	T	0.422	1.000	0.4424
4.79	1.5468	T	0.479	1.000	0.1844
5.01	0.6754	T	0.501	1.000	0.0805
5.12	2.1621	T	0.512	1.000	0.2577
6.24	17.1622	T	0.624	1.000	2.0454
7.37	24.7769	T	0.737	1.000	2.9530
9.08	25.5244	T	0.908	1.000	3.0421
10.30	27.4816	T	1.030	1.000	3.2753
10.86	66.5139	T	1.086	1.000	7.9273
13.06	102.8199	T	1.306	1.000	1[illegible]2544
14.01	39.8118	T	1.401	1.000	4.7449
15.47	105.3081	T	1.547	1.000	12.5509
17.82	103.1372	T	1.782	1.000	12.2922
20.57	103.6697	T	2.057	1.000	12.3556
25.20	155.4995	V	2.520	1.000	18.5329
29.97	39.2435	V	2.997	1.000	4.6772
33.23	0.0000	V	3.323	1.000	0.0000

FIGURE 8

ELECTRON CAPTURE DETECTOR CHROMATOGRAM OF AROCLOR 1260

PCB Analysis of Asphalt Pavement Cores

Two sets of asphalt pavement cores were received from Connecticut DOT. This first set of cores analyzed were labeled as follows:

Box 1	Location 1	= B1L1
Box 2	Location 2	= B2L2
Box 3	Location 3	= B3L3
Box 4	Location 4	= B4L4
Box 5	Pole 5523	= B6P5523
Box 6	Pole 5851	= B6P5851
Box 7	Pole 5521	= B7P5521

The second set of cores were received in October 1983, and were labeled as follows:

Core 0 = SSTC0

Core 1 = SSTC1

Core 2 = SSTC2

Core 3 = SSTC3

Core 4 = SSTC4

Core 5 = SSTC5

Recovery of PCBs from Test Asphalt and Asphalt Oil

To develop a methodology for the recovery and analysis of PCBs from asphalt oil an AC-20 mixture was spiked with a Aroclor 1242 standard with a concentration of 1000 ppm.

A 100.00 g aliquot of AC-20 was weighed in a beaker to the nearest 0.01g. One milliliter at the 1000 ppm. Aroclor 1242 standard was added to the AC-20 resulting in a PCB concentration at 10 mg/Kg. The oil was then thoroughly mixed with a triple-hexane rinsed stainless steel spatual. After the vial containing 1 ml of the Aroclor 1242 standard was emptied, it was rinsed two times with hexane with rinses added to the oil. The beaker and spatual were placed in a oven at 170^{o}C for 15 minutes to allow oil on the spatual to run back into the beaker. One gram of the oil was diluted to 100 ml hexane. The hexane-PCB mixture was concentrated to 10 ml with a Kuderna-Danish Evaporative Concentrator, then cleaned-up with a Florisil slurry. 1 ml of the concentrate was injected into the gas chromatograph which resulted in a concentration of 9.95 mg/Kg, or a 99.5 percent recovery.

Asphalt spiked with Aroclor 1242 was made in the laboratory based on the following constituents:

700 g course aggregate

250 g fine aggregate

50 g AC-20 asphalt oil.

Two batches of asphalt were made having concentrations of 10 mg/kg and 20 mg/kg of Aroclor 1242 in the asphalt binder. Based upon the constituents of the test asphalts, the percent binder was calculated as 5.94 percent AC-20. Results of the extraction of asphalt from aggregate and the extraction of PCB from the binder are given in Table 6.

TABLE 6

Extraction of AC-20 Asphalt Oil and Aroclor 1242 from Laboratory Manufactured Asphalt

Sample No.	Spiked Aroclor 1242 Concentration (mg/kg)	AC-20 Content Design (percent)	PCB Mass in Concentrate (ug)	Asphalt Oil Extracted (g)	Initial Sample Weight (g)	Asphalt Oil Recovered (percent)	PCB Concentration in Binder (mg/kg)	PCB Recovered (percent)	PCB Concentration in Pavement (mg/kg)
1	10	5.00	11.43	1.146	23.030	99.52	9.97	99.70	0.50
2	20	5.00	21.41	1.129	23.700	95.27	18.96	94.80	0.90

After a sample from each core was extracted with hexane, concentrated and injected to the gas chromatograph the resulting chromatogram was qualitatively evaluated to identify what type(s) of PCBs, if any, were present in the sample.

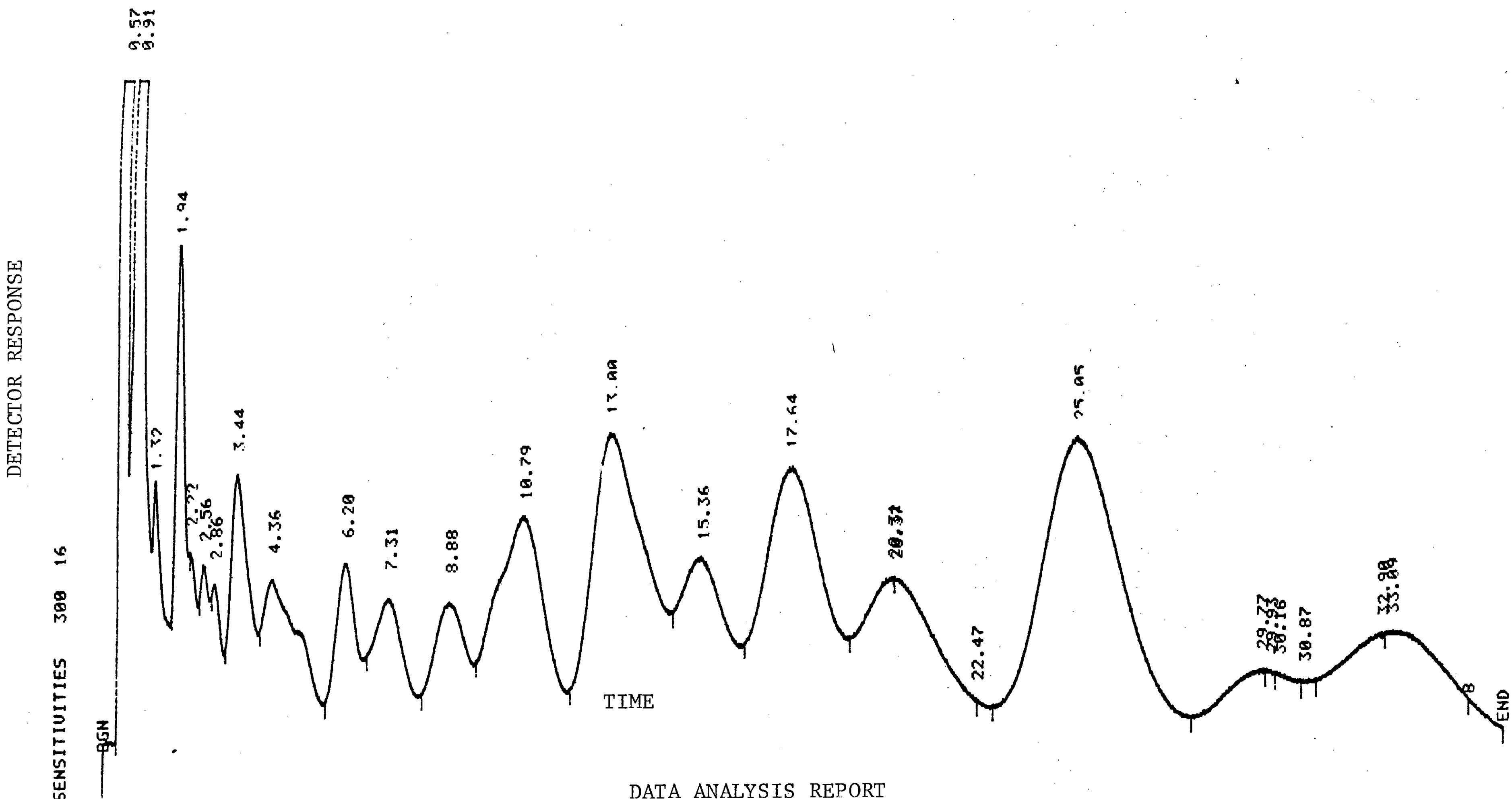
An example of the qualitative analysis used is demonstrated in Figure 9. It can be readily seen that both Aroclor 1242 and Aroclor 1260 are present in the SSTC1 (Core L) sample. Another sample of PCB qualitative analysis from asphalt samples is shown in Figure 10 where only Aroclor 1242 was found to be present. The sample chromatogram in Figure 10 is from Box 6 - Pole 5851.

Results from the analysis of the first set of cores are given in Table 7. Replicates of B2L2 were run and it is evident that the recovery of binder was significantly variable; 2.94 percent and 4.76 percent binder. This probably results from the small sample size used and the subsequent error because of aggregate content in the samples. Of the seven cores received, three were found to contain Aroclor 1242 ranging from 2.48 mg/Kg to 6.24 mg/kg. The results of the second set of cores analyzed for PCB and binder content and are found in Table 8.

In these cores, both Aroclor 1242 and 1260 were found. PCBs were found in all samples. Average Aroclor 1242 content in the binder and pavement was 1.76 mg/Kg and 55 ug/kg, respectively.

Average Aroclor 1260 content in the binder and pavement was 1.81 mg/Kg and 55 mg/Kg, respectively. Binder content averaged 3.08 percent.

It should be noted it was reported that cores from the first two sets (i.e. Table 7 and Table 8 results) were taken from the same location, however, results in Table 7 were obtained by chipping pavement from the bottom of the core and results in Table 8 were from asphalt chipping from the top of cores. It is



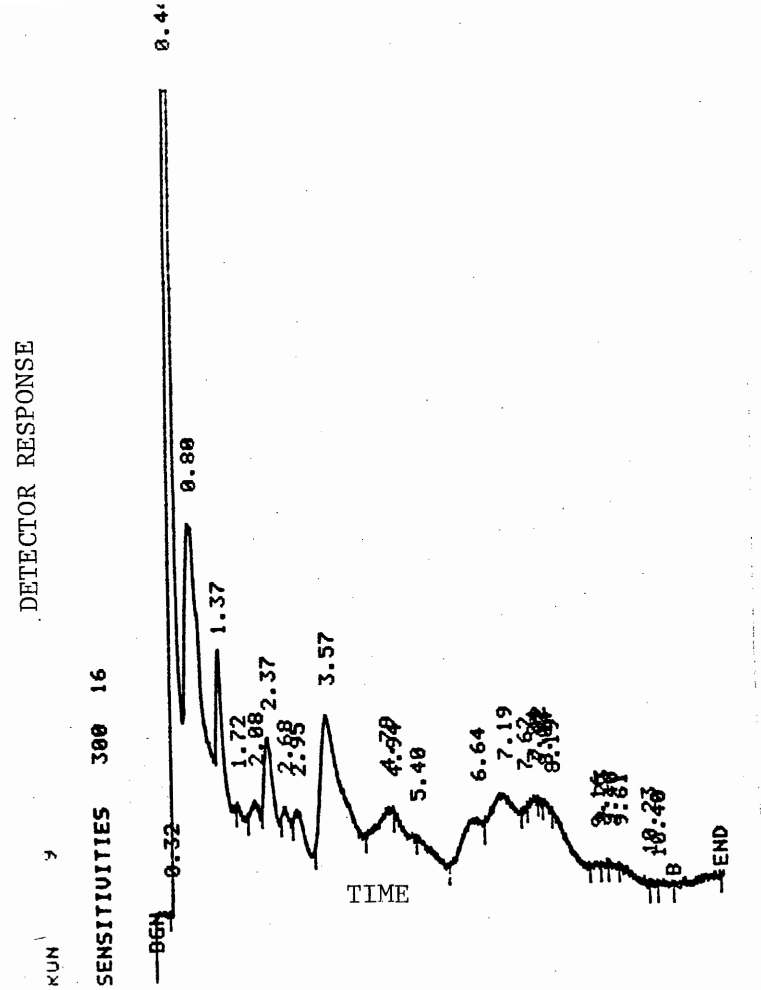
DATA ANALYSIS REPORT

Time	Area BC	RRT	RF	C
0.57	178.3398 T	0.057	1.000	48.7936
0.91	17.2364 T	0.091	1.000	4.7159
1.32	3.8502 T	0.132	1.000	1.0534
1.94	6.2368 T	0.194	1.000	1.7064
2.22	1.9421 T	0.222	1.000	0.5314
2.56	2.1160 T	0.256	1.000	0.5789
2.86	2.1039 T	0.286	1.000	0.5756
3.44	6.7347 T	0.344	1.000	1.8426
4.36	7.9286 T	0.436	1.000	2.1693
6.20	5.0249 T	0.620	1.000	1.3748
7.31	5.5145 T	0.731	1.000	1.5088
8.88	5.7024 T	0.888	1.000	1.5602
10.79	14.2816 T	1.079	1.000	3.9074

Time	Area BC	RRT	RF	C
13.00	21.3952 T	1.300	1.000	5.8537
15.36	11.0931 T	1.536	1.000	3.0351
17.64	20.5900 T	1.764	1.000	5.6334
20.31	6.2297 T	2.031	1.000	1.7045
20.37	7.9971 T	2.037	1.000	2.1880
22.47	0.3069 T	2.247	1.000	0.0840
25.05	28.4966 U	2.505	1.000	7.7967
29.77	1.8940 T	2.977	1.000	0.5182
29.93	0.4425 T	2.993	1.000	0.1211
30.16	0.9855 T	3.016	1.000	0.2696
30.87	0.3372 T	3.087	1.000	0.0923
32.90	4.1753 T	3.290	1.000	1.1424
33.09	4.5428	3.309	1.000	1.2429

FIGURE 9

ELECTRON CAPTURE DETECTOR CHROMATOGRAM OF SSTC1 ASPHALT PAVEMENT CORE SAMPLE



DATA ANALYSIS REPORT

TIME	AREA	BC	RRT	RF	C	NAME
0.32	0.0080	T	0.032	1.000	0.0164	!
0.44	18.1664	T	0.044	1.000	36.7791	!
0.80	5.9059	T	0.080	1.000	11.9569	!
1.37	2.3672	T	0.137	1.000	4.7927	!
1.72	0.8089	T	0.172	1.000	1.6378	!
2.08	1.1372	T	0.208	1.000	2.3025	!
2.37	1.8237	T	0.237	1.000	3.6923	!
2.68	0.7908	T	0.268	1.000	1.6012	!
2.95	1.2514	T	0.295	1.000	2.5335	!
3.57	4.3432	T	0.357	1.000	8.7931	!
4.79	1.5318	T	0.479	1.000	3.1013	!
4.94	1.5058	T	0.494	1.000	3.0487	!
5.40	1.1672	T	0.540	1.000	2.3631	!
6.64	1.6675	T	0.664	1.000	3.3760	!
7.19	2.4820	T	0.719	1.000	5.0250	!
7.62	0.4494	T	0.762	1.000	0.9100	!
7.84	0.6901	T	0.784	1.000	1.3972	!
7.92	0.3807	T	0.792	1.000	0.7709	!
8.04	0.5252	T	0.804	1.000	1.0633	!
8.19	1.4876	T	0.819	1.000	3.0119	!

FIGURE 10
ELECTRON CAPTURE CHROMATOGRAM OF
BGP5851 (Box 6, Pole 5851)
ASPHALT PAVEMENT CORE

TABLE 7

Binder and PCB Content of Asphalt Cores

Sample Code	Concen-trate Volume	PCB Mass Detected	Asphalt Oil Extracted	Initial Sample Mass	PCB Concen-tration in Oil	PCB Type	PCB Concen-tration in Pavement	Binder Content in Asphalt
	(ml)	(ug)	(g)	(g)	mg/kg	Aroclor	ug/kg	percent
B2L2(a)	10	6.90	1.145	24.070	6.02	1242	286.	4.76
B2L2(b)	10	7.18	1.652	56.110	4.34	1242	128.	2.94
B1L1(a)	10	ND	1.01	17.264	ND	--	ND	5.83
B1L1(b)	10	ND	0.97	18.630	ND	--	ND	5.21
B3L3	10	ND	0.83	18,860	ND	--	ND	4.40
B4L4	10	ND	0.79	17.570	ND	--	ND	4.50
B6P3523	10	ND	0.95	18.550	ND	--	ND	5.12
B6P5851	10	2.28	0.918	18.910	2.48	1242	120.	4.86
B6P5521	10	5.49	0.88	17.441	6.24	1242	314.	5.09

TABLE 8

Binder and PCB Content of Asphalt Cores, Second Set

Sample Core	Concentrate Volume	PCB Type	PCB Detected	Asphalt Oil Extended	Initial Sample Mass	PCB Concentration in Oil as Aroclor 1242	PCB Conc in Pavement as Aroclor 1242	PCB Type	PCB Detected	PCB Conc in Oil as Aroclor 1260	PCB Conc in Pavement as Aroclor 1260	Binder Content in Pavement	Total PCB Concentration in Oil	Total PCB Concentration in Pavement
	ml	Aroclor	ug	g	g	mg/kg	ug/kg		ug	mg/kg	ug/kg	percent	mg/kg	ug/kg
SSTC0	10	1242	6.37	2.709	65.521	2.35	94.0	1260	4.01	1.48	59.0	4.13	3.83	153
SSTC1	8	1242	4.14	1.973	65.812	2.10	63.0	1260	12.0	6.08	183.	3.00	8.18	246
SSTC2	10	1242	3.30	1.982	65.069	1.66	49.0	1260	0.95	0.48	14.0	2.96	2.14	63.0
SSTC3	10	1242	1.73	1.915	63.649	0.91	27.0	1260	1.43	0.75	22	3.00	1.66	49.0
SSTC4	10	1242	2.24	1.609	71.283	1.39	31.0	1260	2.30	1.43	33	2.26	2.80	64.0
SSTC5	10	1242	4.10	1.917	61.738	2.14	66.0	1260	1.16	0.61	19	3.11	2.75	85.0

interesting that the top of the core contained both Aroclor 1242 and 1260, while the bottom of the cores contained only Aroclor 1242.

A third set of asphalt samples were received from the Connecticut DOT which consisted of scraping from a surface-coating treatment consisting of small pea-stone size gravel in oil.

The code to same names and reported locations are as follows:

Rt. 21A = CT. Rte. 21, 0.5 mi. N. of Rte. 12, southbound lane

Rt. 21B = CT. Rte. 21, 1.0 mi. N. of Rte. 12, northbound lane

Rt. 21C = CT. Rte. 21, 1.5 mi. N. of Rte. 12, northbound lane

Rt. 169A = CT. Rte. 169, 0.5 mi. N. of Rte. 44, northbound lane

Rt. 169B = CT. Rte. 169, 1.0 mi. N. of Rte. 44, northbound lane

Rt. 169C = CT. Rte. 169, 1.5 mi. N. of Rte. 44, northbound lane

Rt. 272A = CT. Rte. 272, Drakeville 0.30 mi. into project

Rt. 272B = CT. Rte. 272, Drakeville 0.60 mi. into project

Rt. 272C = CT. Rte. 272, Drakeville, end of project

Rt. 197A = CT. Rte. 197, 0.5 mi. E of Rt. 169, eastbound lane

Rt. 197B = CT. Rte. 197, 1.0 mi. E of Rt. 169, eastbound lane

Rt. 197C = CT. Rte. 197, 2.0 mi. E of Rt. 169, eastbound lane

Rt. 61A = CT. Rte. 61, Morris, 1.0 mi. into project

Rt. 61B = CT. Rte. 61, Morris 2.0 mi. into project

Rt. 61C = CT. Rte. 61, Bethlehem, 3.0 mi. into project

Rt. 61D = CT. Rte. 61, Bethlehem, (library)

Results from the analysis of the above samples are reported in Table 9. Aroclor 1242 was the only PCB, if any, found in these samples. The average PCB content in the binder and pavement of the Rt. 21 samples was 4.99 mg/Kg and 542 ug/kg,

TABLE 9

PCB and Binder Content of Surface Coating Treatment Samples

Sample Code	Concen-trate Volume	PCB Type	PCB Detected	Asphalt Oil Extracted	Initial Sample Mass	PCB Concen-tration in Oil	PCB Concen-tration in Pavement	Binder Content
	ml	Aroclor	ug	g	g	mg/ug	ug/kg	percent
Rt 21A	10	1242	11.15	2.151	26.890	5.18	414.	8.00
Rt 21B	10	1242	INT*	1.563	28.870	--	--	5.41
Rt 21C	10	1242	19.10	1.916	28.500	9.97	670.	6.72
Rt 169A	10	1242	6.76	1.992	28.640	3.39	236.	6.96
Rt 169B	10	1242	7.66	1.902	28.870	4.03	265.	6.59
Rt 169C	10	1242	8.82	1.724	29.200	5.11	302.	5.90
Rt 272A	10	1242	3.93	2.294	28.800	1.71	79.0	7.97
Rt 272B	10	1242	8.46	1.66	28.880	5.09	293	5.75
Rt 272C	10	1242	4.88	2.137	28.445	2.28	171.	7.51
Rt 197A	10	1242	2.49	2.164	30.870	1.15	80.0	7.01
Rt 197B	10	1242	8.55	1.828	29.569	4.67	289.	6.18
Rt 197C	10	1242	6.90	1.268	28.697	5.44	240.	4.42
Rt 61A	10	1242	3.67	2.768	29.308	1.32	125	9.45
Rt 61B	10	1242	5.81	1.778	28.093	3.26	207.	6.33
Rt 61C	10	1242	ND**	1.959	28.357	ND	ND	6.91
Rt 61D	10	1242	ND	2.868	30.087	ND	ND	9.53

*INT - Interference, not possible to qualitate at low level.
**ND - None detected.

respectively. The Rt. 21B sample contained an interference and was not quantified. This was the only sample received which was not quantified because the interference could not be removed. Rt. 169 samples averaged binder and pavement PCB concentrations of 4.18 mg/Kg and 268 mg/Kg, respectively. It was observed that the PCB concentration increased with distance in a northerly direction away from Rt. 44. Rt. 272 samples had average PCB concentrations in the binder and pavement of 3.03 mg/Kg and 181 ug/Kg, respectively. PCB concentrations in the binder in the Rt. 197 samples increased with distance to an easterly direction from Rt. 169. Of the Rt. 61 samples, two had no detected levels of PCBs and the two closest to the beginning of the project had 1.32 mg/Kg and 3.26 mg/Kg Aroclor 1242 concentrations.

Analysis of Soil Samples Under and Near Asphalt Pavements

Several soil samples were obtained by removing a pavement core and sampling soild from the area immediately below from which the core was taken. Additionally, soil samples were taken from a catch basin which received runoff from an asphaltic pavement and at point where the catch basin drained into a small pond.

Soil samples were coded with respect to the following locations:

Rt. 6AS - Route 6, 150' south of Rt. 64 intersection
Rt. 6BS - Route 6, 140' south of Rt. 64 intersection
Rt. 64AS - Route 64, 150' east of Rt. 6 intersection
Rt. 64BS - Route 64, 160' east of Rt. 6 intersection
Rt. 6CB - Route 6, catch basin, west side of road, 150 south of Rt. 64 intersection
Rt. 6PD - pond sediment sample, at entrance to pond from above catch basin

Examination of Table 10 indicates that the levels of PCB found in all samples were quite low. The highest PCB concentration in the soil samples from under the asphalt pavement cores was 95 ug/Kg of Aroclor 1242. Sediment from both the catch basin and the pond sediment were also quite low at 67 ug/Kg and 50 ug/Kg of Aroclor 1242, respectively. Aroclor 1260 was not detected in any of these samples.

TABLE 10

PCB RESULTS FROM SOIL

SAMPLES ADJACENT TO ASPHALT PAVEMENTS

Sample Code	Concentrate Volume (ml)	PCB Mass ug	PCB Type Aroclor	Sample Mass g	PCB Concentration in soil ug/Kg
Rt. 6AS	7	1.844	1242	66.6275	28
Rt. 6BS	7	5.845	1242	61.6667	95
Rt. 64AS	8	2.444	1242	72.4806	34
Rt. 64BS	8	4.214	1242	54.7707	77
Rt. 6CB	7	3.990	1242	59.2036	67
Rt. 6PD	8	4.722	1242	94.3765	50

CONCLUSIONS AND RECOMMENDATIONS

A method for the efficient extraction of PCBs from asphalt pavement and asphalt oil and subsequent analysis by electron-capture gas chromatography was developed. Recovery efficiencies of PCBs from asphalt oil and asphalt pavement using the test method were 99.5 percent and 94.8 to 99.7 percent, respectively.

Most asphalt pavement core samples contained Aroclor 1242. One set of asphalt pavement cores contained both Aroclor 1242 and Aroclor 1260. The maximum asphalt pavement PCB concentration measured in this research was 314 ug/Kg of Aroclor 1242 in sample B7P5521, Box 7 - Pole 5521. In the sample, the binder PCB concentration was 6.24 mg/Kg.

All samples of surface seal-coats (i.e. mixtures of cutter and aggregate) contained Aroclor 1242. The maximum PCB concentration in the seal-coats was 670 ug/Kg. This sample had a cutter PCB concentration of 9.97 mg/Kg as Aroclor 1242.

Soil samples taken from areas under asphalt pavement and in road-runoff zones contained Aroclor 1242 ranging from 28 ug/Kg to 95 ug/Kg. All samples contained PCBs. These results, although only preliminary, do indicate that PCBs are strongly retained in the asphalt matrix and do not significantly leach out under ambient environmental conditions.

The significance of the PCB analyses conducted in this study is that none of the asphalt pavements, binders or cutters, received from the Connecticut DOT, are PCB wastes, as defined in the Toxic Substances Control Act (TOSCA). The lower cut-off level of PCBs that are to be regulated under TOSCA is 50 ppm on a dry weight basis (i.e. PCB Waste, by definition). The highest PCB level measured any sample received for analysis in this study was 670 ug/Kg (ppb), (i.e. 0.670 ppm) as Aroclor 1242 (dry weight basis). It can be seen that the levels of PCB detected in this study are only about 1/100 of the 50 ppm standard.

If it is assumed, however, that all of the PCBs in the asphaltic products sampled, originated in the asphaltic portion of the sample, the concentrations are significantly greater. The highest concentration in the hexane-extracted asphalt oil was 9.97 mg/Kg as Aroclor 1242. This is still well below the TOSCA level for a PCB waste (i.e. 50 mg/Kg).

Because this research did not address the sources of PCBs in bituminous materials, the origin of the PCBs in the samples analyzed cannot be determined. There is a possibility that waste oil, contaminated with PCBs at concentrations greater than 50 mg/Kg, was added to some of the products tested and only because of dilution were the PCB levels observed in this study less than 50 mg/Kg. This hypothesis, however, cannot be confirmed with information presently available.

It is recommended that additional research be initiated addressing the sources of PCBs in asphaltic and bituminous materials in order that their presence be minimized.

Future work should investigate the PCB content of soils near a highway containing PCBs. Ideally, the site should be located in a non-urban and non-industrial location to minimize other sources of PCBs.

Finally, work should be initiated that investigates the leachability of PCBs from asphaltic and bituminous materials by a variety of materials such as acidic rainfall and anthropogenic organic compounds in highway runoff.

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APPENDIX A

Fused-Silica Capillary Chromatography of PCBs

For purposes of obtaining a more specific profile of the particular compounds in a PCB contaminated material, fused-silica capillary gas chromatography columns may be used. Compound separation with capillary columns is far superior to packed columns (e.g. those used in this study and recommended by EPA for PCB analysis). Capillary columns have an inside diameter of 0.25 mm (i.e. typical) while packed column usually have an 1/8" (3.2 mm) inside diameter. The major disadvantage of capillary gas chromatography of PCBs is that the columns easily overload (although there may be some on-going hardware developments that will minimize this problem) from both excessive liquid and/or compounds injected. As it is, a 0.5 ul injection is split 1:100 by a capillary splitting valve before it is placed on the capillary column. Thus, only 0.005 ul of liquid is actually injected into the column.

The capillary method may have importance to the Connecticut DOT for determining a source(s) of PCB contamination because of the enhanced resolution of compounds. Figure 11 is a capillary chromatogram of Aroclor 1242. The operating conditions are as follows;

volume injected - 0.5 ul
split ratio - 1:100
column: fused-silica WCOT, 0.25 mm I.D. x 50 m long
injector temperature - 250°C
oven temperature - 180°C, isothermal
detector temperature - 300°C
carrier gas - 5% CH_4 in Argon (ECD grade)
make-up gas: 5% CH_4 in Argon
make-up flowrate: adjusted to achieve 60cc/min
flowrate at ECD detector
carrier gas pressure: 30 psig
detector: Electron Capture
gas chromatograph: Perkin-Elmer Sigma 1

Data Analysis report is also found, appended to Figure 11.

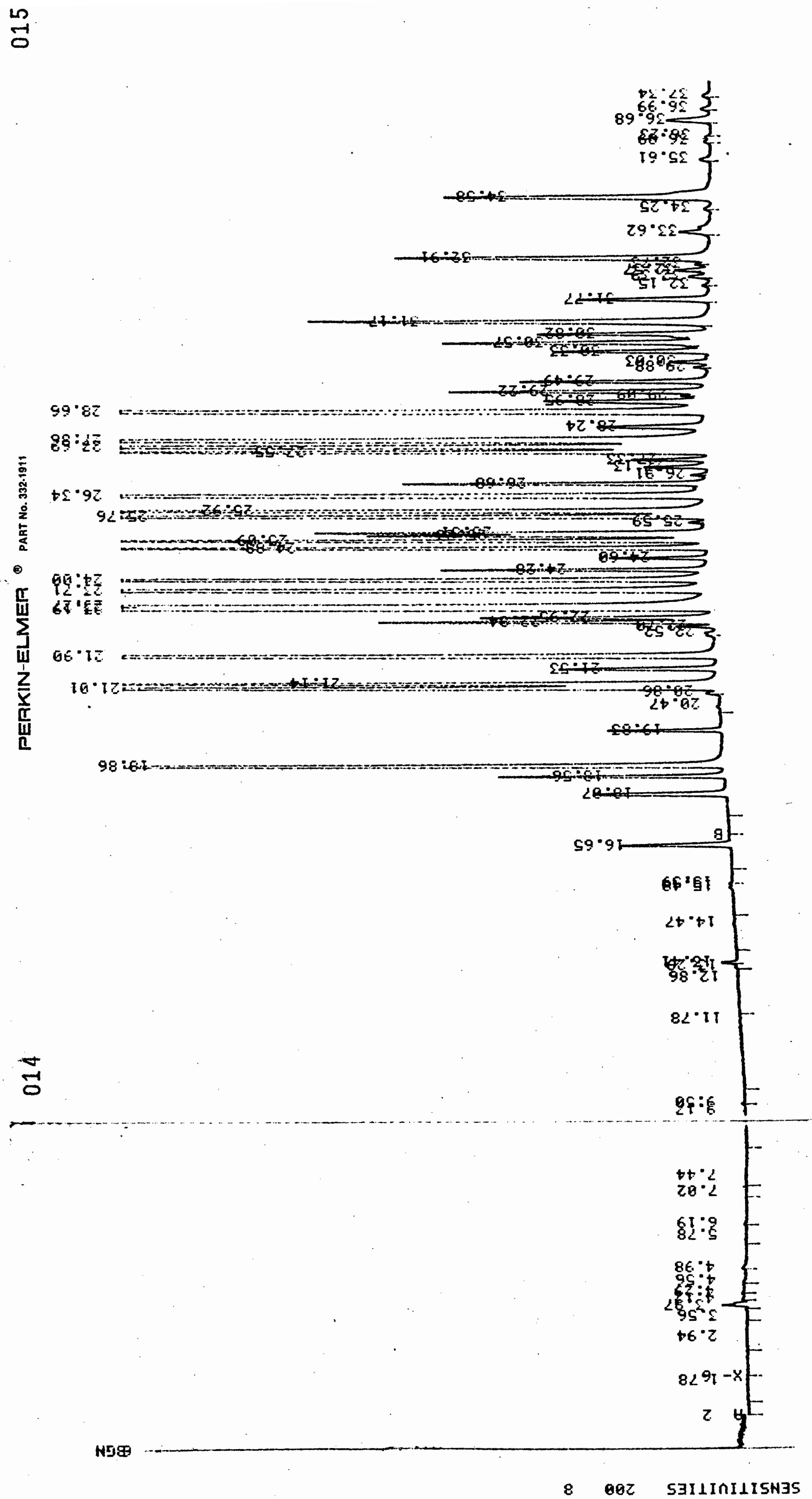

FIGURE 11

ECD CAPILLARY CHROMATOGRAM OF AROCLOR 1242

FIGURE 11 (cont'd)

RUN 1 10 : 4.6 12 / 30 / 82

SENSITIVITIES 200 8

TIME	AREA	BC	RRT	RF	C	NAME
1.78	0.0460	V	0.178	1.000	0.0470	!
2.94	0.0296	V	0.294	1.000	0.0303	!
3.56	0.0063	V	0.356	1.000	0.0065	!
3.97	0.0943	T	0.397	1.000	0.0964	!
4.12	0.0301	T	0.412	1.000	0.0307	!
4.29	0.0401	T	0.429	1.000	0.0410	!
4.56	0.0406	T	0.456	1.000	0.0415	!
4.98	0.0549	V	0.498	1.000	0.0561	!
5.78	0.0240	V	0.578	1.000	0.0245	!
6.19	0.0216	V	0.619	1.000	0.0221	!
7.02	0.0089	V	0.702	1.000	0.0091	!
7.44	0.0371	V	0.744	1.000	0.0379	!
9.17	0.0145	V	0.917	1.000	0.0148	!
9.50	0.0139	V	0.950	1.000	0.0143	!
11.78	0.0284	V	1.178	1.000	0.0290	!
12.86	0.0376	V	1.286	1.000	0.0385	!
13.29	0.0127	T	1.329	1.000	0.0130	!
13.41	0.0547	V	1.341	1.000	0.0559	!
14.47	0.0327	V	1.447	1.000	0.0335	!
15.48	0.0488	T	1.548	1.000	0.0499	!
15.59	0.0231	V	1.559	1.000	0.0236	!
16.65	0.4259		1.665	1.000	0.4349	!
18.07	0.6956	T	1.807	1.000	0.7103	!
18.56	0.7700	T	1.856	1.000	0.7862	!
18.86	3.6080	T	1.886	1.000	3.6839	!
19.83	0.4340	T	1.983	1.000	0.4431	!
20.47	0.2001	T	2.047	1.000	0.2043	!
20.86	0.1077	T	2.086	1.000	0.1100	!
21.01	3.5635	T	2.101	1.000	3.6384	!
21.14	2.3771	T	2.114	1.000	2.4271	!
21.53	0.7490	T	2.153	1.000	0.7648	!
21.90	4.1899	T	2.190	1.000	4.2780	!
22.52	0.1465	T	2.252	1.000	0.1496	!
22.70	0.1716	T	2.270	1.000	0.1752	!
22.84	1.0936	T	2.284	1.000	1.1166	!
22.95	0.8341	T	2.295	1.000	0.8517	!
23.19	4.9324	T	2.319	1.000	5.0362	!
23.27	7.7952	T	2.327	1.000	7.9591	!
23.71	5.3936	T	2.371	1.000	5.5070	!
24.00	3.9377	T	2.400	1.000	4.0205	!
24.28	1.0546	T	2.428	1.000	1.0768	!
24.60	0.4914	T	2.460	1.000	0.5018	!
24.88	2.4435	T	2.488	1.000	2.4949	!
25.09	2.4205	T	2.509	1.000	2.4714	!
[illegible]	[illegible]	T	[illegible]	[illegible]	[illegible]	!

Time	Area BC		RRT	RF	C	
25.27	1.0579	T	2.527	1.000	1.0802	!
25.31	1.4352	T	2.531	1.000	1.4655	!
25.59	0.1548	T	2.559	1.000	0.1581	!
25.76	3.4054	T	2.576	1.000	3.4770	!
25.92	3.0878	T	2.592	1.000	3.1528	!
26.34	5.1880	T	2.634	1.000	5.2971	!
26.68	1.1926	T	2.668	1.000	1.2177	!
26.91	0.1600	T	2.691	1.000	0.1634	!
27.13	0.3573	T	2.713	1.000	0.3649	!
27.33	0.3477	T	2.733	1.000	0.3550	!
27.55	2.4949	T	2.755	1.000	2.5474	!
27.69	4.1854	T	2.769	1.000	4.2734	!
27.86	5.6057	T	2.786	1.000	5.7236	!
28.24	0.7825	T	2.824	1.000	0.7990	!
28.66	5.3107	T	2.866	1.000	5.4224	!
28.95	0.8929	T	2.895	1.000	0.9117	!
29.09	0.1308	T	2.909	1.000	0.1336	!
29.22	1.1086	T	2.922	1.000	1.1319	!
29.49	0.9038	T	2.949	1.000	0.9228	!
29.88	0.1080	T	2.988	1.000	0.1104	!
30.03	0.2479	T	3.003	1.000	0.2532	!
30.33	0.6746	T	3.033	1.000	0.6888	!
30.57	1.2273	T	3.057	1.000	1.2531	!
30.82	0.8914	T	3.082	1.000	0.9101	!
31.17	2.1258	T	3.117	1.000	2.1705	!
31.77	0.6910	T	3.177	1.000	0.7056	!
32.15	0.0727	T	3.215	1.000	0.0743	!
32.39	0.1453	T	3.239	1.000	0.1484	!
32.57	0.1898	T	3.257	1.000	0.1938	!
32.73	0.0838	T	3.273	1.000	0.0856	!
32.91	1.6506	T	3.291	1.000	1.6853	!
33.62	0.3078	T	3.362	1.000	0.3144	!
34.25	0.0481	T	3.425	1.000	0.0492	!
34.58	1.5781	T	3.458	1.000	1.6113	!
35.61	0.0818	T	3.561	1.000	0.0835	!
36.09	0.0384	T	3.609	1.000	0.0392	!
36.23	0.0331	V	3.623	1.000	0.0339	!
36.68	0.2653	T	3.668	1.000	0.2709	!
36.99	0.0584	T	3.699	1.000	0.0597	!
37.34	0.0870	V	3.734	1.000	0.0888	!
38.54	0.0080	V	3.854	1.000	0.0082	!
38.85	0.0879	V	3.885	1.000	0.0897	!
39.45	0.0210	V	3.945	1.000	0.0215	!
40.60	0.0434	V	4.060	1.000	0.0444	!
41.09	0.0101	V	4.109	1.000	0.0104	!
41.54	0.0631	V	4.154	1.000	0.0644	!
42.01	0.0065		4.201	1.000	0.0067	!
43.88	0.0245	T	4.388	1.000	0.0250	!
44.37	0.0769	V	4.437	1.000	0.0785	!
46.08	0.0174	V	4.608	1.000	0.0178	!
47.21	0.0612	V	4.721	1.000	0.0625	!
49.26	0.0543	V	4.926	1.000	0.0555	!