

POLYCHLORINATED BIPHENYL (PCB) SAMPLING

September 1997

Prepared for:

SUNY New Paltz
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Adirondack Project No. 970815EA

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EXECUTIVE SUMMARY

On August 18 and 19, 1997, Adirondack Environmental Services, Inc. (Adirondack) performed Polychlorinated Biphenyl (PCB) area air and wipe sampling in the Residence Halls located on the State University of New York (SUNY) New Paltz Campus. All monitoring and analysis activities were performed in accordance with the SUNY New Paltz Polychlorinated Biphenyl Air and Wipe Sampling Strategy, dated August 1997. The purpose of the sampling was to ensure that the four residence halls located on the campus were free of PCB contamination within a statistical confidence limit of 95%.

A total of 29 wipe samples and three field blanks and 30 air samples and three blanks were collected in randomly selected rooms in the four residence halls. Both the wipe sample locations and the area air sample locations were selected with a random number table. Samples were collected, labeled and placed on ice and delivered to the laboratory. Wipe samples were analyzed by the EPA 8080 method and the air samples were analyzed by the New York State Department of Health (NYDOH) method DOH 311-1.

The analysis of the 32 wipe samples collected indicated all results to be less than the detection limit of the analysis <0.1 ug/100cm² (<1ug per 900 cm²). The analysis of the 33 air samples collected indicated results to be less than the detectable limits of the analysis (<0.2 ug/m³) for all of the samples except one. Field sample identification number A24 (laboratory identification number 970820EA12) indicated a concentration of 0. 3 ug/m³ for Aroclor 1242 and 0.2 ug/m³ for Aroclor 1254. This sample was obtained outside Gage Hall.

The results of the wipe and air sampling indicate that all sample results were below Environmental Protection Agency, Occupational Safety and Health Administration and National Institute for Occupational Safety and Health published exposure limits for PCBs.

INTRODUCTION

1.0

On August 18 and 19, 1997, Mr. Paul Watson, CIH, CET and Mr. John Frisone, Industrial Hygiene Technician of Adirondack Environmental Services, Inc. (Adirondack) performed Polychlorinated Biphenyl (PCB) area air and wipe sampling in the four residence halls located on the State University of New York (SUNY), New Paltz Campus. The sampling team was escorted by Mr. Peter Betley of the SUNY New Paltz, Health and Safety Department. All monitoring and analysis activities were performed in accordance with the SUNY New Paltz Polychlorinated Biphenyl Air and Wipe Sampling Strategy, dated August 1997. A copy of the sampling strategy is located in Appendix A. The purpose of the sampling was to ensure that the four residence halls were free of PCB contamination that may have resulted from a release that occurred on December 29, 1991. The PCB material may have entered the residence halls by way of open windows, doors, and the ventilation systems. These residence halls were subsequently cleaned after the release and this sampling was conducted to ensure that the residence halls did not contain any residual material at a statistical confidence limit of 95%.

The sampling strategy and methodology used to complete the sampling is described in Section 2. The sample results and observations are presented in Section 3 and discussed in Section 4. The SUNY New Paltz Air and Wipe Sampling Strategy is included in Appendix A and the exact wipe and air sample locations are identified in Appendix B. The remaining Appendices present the laboratory results and field sampling data sheets.

2.0 SAMPLING STRATEGY AND METHODOLOGY

2.1 SAMPLING STRATEGY

The purpose of the exposure assessment was to obtain statistically representative wipe and air samples of the various residence hall rooms. A sampling strategy was designed to meet this objective. The sampling strategy developed for the facility is presented in Appendix A. This strategy served as a guide for the quantitative monitoring performed in this assessment.

2.2 SAMPLING AND ANALYTICAL METHODOLOGY

2.2.1 Wipe Sampling

Wipe samples were obtained utilizing 900 cm², disposable cardboard templates. Sample technicians utilized latex gloves and laboratory prepared swabs. Wipe sampling was performed by wiping a laboratory prepared filter paper over a 900 square centimeter area. The filter paper was prepared with 10 milliliters (ml) of reagent grade hexane. The pretreated wipes were replaced in the glass vials for storage and transport to the laboratory. All of the samples collected were assigned a sequential sample identifier and sample locations were plotted on site plans supplied by SUNY New Paltz. Wipe sample locations were selected with a random number table. Floor or desk top locations were selected in a similar fashion. All floor samples were obtained in the center of the residence hall rooms, except where otherwise noted on the field sample data sheets. All deak top samples were obtained in the area defined as the student's work area (that area directly in front of where a student may sit at the desk). All wipe samples were documented on a sampling record. This record included the location (identified by name and at least two measurements with a tape measure), date, room number. building and samplers name. All samples were assigned a unique sequential sample number. All samples were labeled, placed on ice and driven to the laboratory for analysis. Samples were analyzed by the EPA 8080 method for the following Aroclors; 1016, 1221, 1232, 1242, 1248, 1254 and 1260.

Identification of any PCB concentrations above 500 micrograms was to be confirmed by gas chromatography/mass spectrometry (GC/MS), however no peaks were identified above this level. The selection of this method was consistent with the methods previously utilized at SUNY New Paltz.

2.2.2 Air Sampling

Thirty-three air samples were collected using Mine Safety Appliances (MSA) Flow-lite air sampling pumps. All air sampling pumps were calibrated before and after sampling with a BIOS Dry Cal primary standard. All air samples were obtained in the center of the residence hall rooms with the doors and windows closed. All air samples were documented on an air sampling record. This record included the exact run time, pre and post calibration information, sample location and sampler's name. All samples were assigned a unique sequential identification number. Three (3) field blanks were submitted as per the sampling strategy. Blanks were not identified to the analytical laboratory and were assigned a sequential sample number.

Samples were analyzed by the DOH 311-1 method Analysis targeted the following Aroclors; 1016, 1221, 1232, 1242, 1248, 1254, and 1260. This method was selected to be consistent with previous air monitoring performed at SUNY New Paltz.

All air and wipe samples were submitted under chain of custody documentation to Adirondack's laboratory in Albany, New York for analysis. Wipe and air samples were stored in separate ice chests for delivery to the laboratory. Adirondack is accredited by the American Industrial Hygiene Association (AIHA), accreditation number 490.

RESULTS AND OBSERVATIONS

3.1 RESULTS

3.0

3.1.1 PCB Wipe Sampling

The analysis of the 32 wipe samples collected indicated all results to be less than the detection limit of the analysis, <0.1 ug/100cm² (<1 ug per 900 cm²). These results can be compared to the Environmental Protection Agency's clean up criteria for a high contact residential/commercial areas of 10 ug/100 cm². Wipe sample locations are presented in Appendix B, Table 1.

3.1.2 PCB Air Sampling

The analysis of the 33 air samples collected indicated results to be less than the detectable limits of the analysis (<0.2 ug/m³) for all of the samples except one. Field sample identification number A24 (laboratory identification number 970820EA12) indicated a concentration of 0.3 ug/m³ for Aroclor 1242 and 0.2 ug/m³ for Aroclor 1254. This sample was obtained outside Gage Hall. These results can be compared to the Occupational Safety and Health Administration's (OSHA) Permissible Exposure Limit (PEL) of 500 ug/m³ for Aroclor 1254 and 1000 ug/m³ for Aroclor 1242. These are the only two Aroclors that OSHA has established PELs. The National Institute for Occupational Safety and Health (NIOSH) Recommended Exposure Limit (REL) for all PCBs is 1 ug/m³. Air sample locations are listed in Appendix B, Table 2.

3.2 **OBSERVATIONS**

Wipe sampling was conducted in unoccupied residence hall rooms. Where possible, SUNY New Paltz identification tags were utilized to identify desk tops that were sampled. However the identification tags were not always readily visible on the furniture and therefore were not documented.

All air monitoring was conducted with the doors and windows closed. All rooms

DISCUSSION AND CONCLUSION

4.0

Based upon the findings, all sample results were less than the detection limits of the analysis except for one air sample, field number A24. This sample was obtained outside Gage Hall. This sample indicated two Aroclors (1242 and 1254) at the detection limit of the analysis.

It may be likely that the two Aroclors were components in the transformer oil (this oil was identified as Aroclor 1260) from the release incident. Commercial PCBs were generally mixtures of many different chlorinated biphenyls that were manufactured to meet specific requirements. These mixtures varied from batch to batch. Another possible explanation of the results is that the source of the material was from an off site release. PCBs can be present as solid or liquid acrosols and as vapors and depending on the type of PCB they can remain airborne for up to ten days. While dispensed in the air PCBs can travel fairly long distances.

The New York State Department of Health is currently conducting quarterly air monitoring on campus. These results should be compared to previously obtained results to determine if these Aroclors have been identified and are, as suspected, transient in nature.

Adirondack recommends that SUNY New Paltz maintain this and all previous sampling results for future reference.

APPENDIX A
SUNY NEW PALTZ AIR AND WIPE SAMPLING
STRATEGY



POLYCHLORINATED BIPHENYL AIR AND WIPE SAMPLING STRATEGY

August 1997

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1.0

INTRODUCTION

The following sampling plan has been developed to ensure that the four residence halls located on the State University of New York (SUNY) New Paltz campus are free of potential Polychlorinated Biphenyl (PCB) contamination. Area air monitoring and wipe sampling on horizontal surfaces have been prescribed. The number of air and wipe samples have been selected to provide a statistical confidence limit of 95%. Sampling methodology, selection of sample locations, and analytical methods are described in the text below.

The purpose of the air and wipe sampling plan is to devise a statistically significant approach to the evaluation of approximately 422 residence hall rooms for PCB contamination.

In order to develop this sampling plan a few assumptions have been made. The first is that the four residence halls (approximately 422 rooms total, a room listing appears in Appendix A) have the same risk of potential exposure and are therefore defined as one homogeneous risk group. This assumption was based upon the information provided from SUNY New Paltz that the PCB release that occurred on December 29, 1991, may have emitted airborne PCBs. The PCB material may have entered the residence halls by way of open windows, doors, and the ventilation systems.

The second assumption is that PCB contamination is most likely present in the form of dust contamination and that horizontal surfaces (floors and desk tops) would be the most likely locations to detect any PCB material.

The final assumption that has been made is that the largest population that spends the greatest amount of time in the residence halls is the students. The majority of the students time spent would most likely be in the residence hall rooms, therefore sampling will concentrate on these areas and will exclude hallways, kitchen and common areas. Student contact with any potential PCB material would most likely be on the working area of the desk (approximately center) and the open areas of the floor (approximately center).

In determining that the four residence halls are free of PCB material, it will be necessary to randomly select rooms to be sampled. The sample group must be of sufficient size to ensure that the random sample will have a high probability to contain at least one sample containing PCB contamination if one exists. The number of samples (represented by n) to be collected from the homogenous risk group is dependent on the number of areas in the risk group (represented by N),

and the statistical confidence required (in this instance 95% confidence to collect at least one sample in the highest 10% of potential contamination).

Sample size n can be determined from a complex formula or by using the Table 1, Calculation of Sample Size for a Maximum Risk Subgroup from a Homogenous Exposure Risk Group. Prior to using the table, the following variables must be determined:

N = Size of the population with potential contamination (422 rooms in this case). n = Size of the sample subgroup to be selected from the homogenous risk group. α = Allowed probability of missing all of the areas in the top contamination group (α =0.05 for the 95% confidence level).

 τ = Proportion of the group included as the high exposures-desired high in the exposure subgroup percentage (t=0.1 for the top 10% of all exposures).

From Table 1, the number of air and wipe samples for a population of 422 rooms, with a 95% confidence and a top 10% desired high exposure subgroup yields 29 samples each.

Table 1 - Calculation of Sample Size for a Maximum Risk Subgroup From a Homogeneous Exposure Risk Group

Size of partial Sample for Top 10% (τ =0.1) and Confidence 0.95 (α =0.05) (use n-N if N<11)

Size of Group, N	Number of Required Samples, n	
12	11	
13-14	12	
15-16	13	
17-18	14	
19-21	15	
22-24	16	
25-27	17	
28-31	18	
32-35	19	
36-41	20	
42-50	21	
∞	29	

2.1 SAMPLING STRATEGY

2.1.1 Wipe Samples

Wipe sample locations (29) have been selected by assigning sequential numbers to each residence hall room starting with the number one (approximately 422 rooms, a room listing appears in Appendix A). Identified wipe sample locations appear in Appendix D. Locations must be indicated on drawings supplied by SUNY New Paltz. A random number table generator was utilized to select twenty-nine wipe sample locations. Once the rooms have been identified, the random number generator was utilized to determine whether a floor or desk top surface will be wiped. This was accomplished by assigning a unique number to the desk top location and one to the floor location. The random number table was then used to select the sample location. The sample location (the floor or desk top) must be documented on the SUNY New Paltz supplied site plan.

Samples will be obtained from the approximate center of the desk top or floor surface.

2.1.2 Air Samples

Area air sample locations were determined by assigning sequential number to each residence hall room starting with the number one (approximately 422 rooms, a room listing appears in Appendix A). Sample locations and sample numbers must be identified on drawings supplied by SUNY New Paltz. A random number table was utilized to select twenty-nine sample locations for the area air samples. Samples will be obtained at approximately four to five feet above the floor in order to represent a person's breathing zone. Samples will be taken in the center of the room with the doors and windows closed. One ambient outside air sample will also be obtained for comparison purposes.

2.2 SAMPLING AND ANALYTICAL METHODOLOGY

2.2.1 Wipe Samples

Wipe sampling will be performed by wiping a laboratory prepared four inch square gauze or glass wool wipe over a 900 square centimeter area. The filter paper will be prepared with 10 milliliters of reagent grade hexane. The pretreated wipes will be placed in a glass vial for storage and use. A pre cut 900 square centimeter reusable stainless steel templates will be utilized. This template will be supplied by SUNY New Paltz. The template will be decontaminated in-between each sample by wiping the entire surface with hexane and disposable towels. Disposable cardboard templates of the same size may also be used. These templates must be discarded after each sample. The sample technician will wear disposable gloves during the collection and handling of each sample to prevent potential sample cross contamination and skin exposure. All wipe samples will be documented on a sampling record. This record shall include at a minimum the location (to be identified by name and at least two measurements with a tape measure), date, room number, building and samplers name. All samples will be assigned a unique sequential sample number. Ten percent field blanks (3) will be submitted at the time of analysis. Blanks must not be identified to the analytical laboratory and must be assigned a sequential sample number. Blanks will be unopened laboratory prepared filters. Wipe samples will be obtained on horizontal surfaces only (floors and desk tops).

The analytical method will be EPA Method 8080 utilizing gas chromatography with an electron capture detector (GC/ECD). Identified PCB concentrations above 500 micrograms will be confirmed by gas chromatography/mass spectrometry (GC/MS). The following Aroclors must be targeted and quantified; 1016, 1221, 1232, 1242, 1248, 1254, and 1260. A copy of the method is included in Appendix B. The selection of this method is consistent with the methods previously utilized at SUNY New Paltz.

2.2.2 Air Samples

The New York State Department of Health Method 311-1, Polychlorinated

Biphenyls in Ambient Air, will be utilized as the air sampling and analytical method. Samples will be obtained with an air sampling pump, flexible tubing and appropriate media as outlined in the method. Sample time will be from four to eight hours at one liter per minute. A copy of the method is included in Appendix C. All samples will be pre and post calibrated with a National Institute of Standards and Technology (NIST) traceable primary standard such as a Gillian Gilabrator, Bios DRY CAL or equivalent. All air samples will be documented on an air sampling record. This record shall include at a minimum the run time, pre and post calibration information, sample location and samplers name. All samples will be assigned a unique sequential sample number. Ten percent field blanks (3) will be submitted at the time of analysis. Blanks must not be identified to the analytical laboratory and must be assigned a sequential sample number. Analysis must identify and quantify the following Aroclors; 1016, 1221, 1232, 1242, 1248, 1254, and 1260. This method was selected to be consistent with previous air monitoring performed at SUNY New Paltz.

2.3 SAMPLING HANDLING

2.3.1 Wipe samples

Immediately after wipe sampling, return the sample to the glass vial. Label the vial with an unique sample number and secure the sample for transport. Samples will be shipped to the laboratory in an ice chest in order to maintain the samples at 4° Celsius.

All samples must be accompanied to the laboratory with a properly completed chain of custody record.

All wipe samples must be shipped to the laboratory in a separate container from any air samples.

2.3.2 Air Samples

The air sampling train will be disassembled after completion of sampling. The

florisil tube will be capped immediately and labeled with a unique sample number and will be placed in a clean zip lock bag. The bag will be labeled with the unique sample number and secured for transport to the laboratory.

All samples must be accompanied to the laboratory with a properly completed chain of custody record. Samples will be shipped to the laboratory in an ice chest in order to maintain the samples at 4⁰ Celsius

All air samples must be shipped to the laboratory in a separate container from any wipe samples.

2.4 PRESENTATION OF RESULTS

All supporting sampling documentation including chain of custody records, air sampling data forms, wipe sampling forms, and laboratory analytical results will be presented in a written report. The format will be determined by SUNY New Paltz. Wipe sample results will be compared to the Environmental Protection Agency's clean up criteria for a high contact residential/commercial area of 10 micrograms/100 square centimeters. Air sample results will be compared to the outside sample, sample blanks, and to National Institute for Occupational Safety and Health Administration airborne limits.

- Environmental Protection Agency, 40 CFR Part 761, Polychlorinated Biphenyls Spill Cleanup Policy: Final Rule
- Environmental Protection Agency, <u>Field Manual For Grid Sampling of PCB Spill</u>
 <u>Sites to Verify Cleanup</u>, EPA Document Number 560/5-86-017
- Environmental Protection Agency, <u>Verification of PCB Spill Cleamsp By Sampling and Analysis</u>, EPA Document Number 560/5-86-026
- National Institute for Occupational Safety and Health, <u>Occupational Sampling</u>
 <u>Strategy Manual</u>, DHEW Document Number 77-173

APPENDIX A RESIDENCE HALL ROOM LISTING

SCUDDER HALL, BUILDING #20

Desement	120	31
B1	122	313
B2	124	315
B3		310
B4	Second Floor	311
B5	203	319
B6	205	32:
B7	207	32
B8	208	324
B9	209	325
B10	211	32
B11	213	329
B12	215	30
B13	216	303
B14	217	303
B15	219	301
B16	221	310
B17 Overflow	223	313
	224	314
First Floor	225	318
103	227	320
1 05	229	322
107	201	320
108	202	328
109	204	330
Director's Apartment	206	33
113	210	
115	212	
116	214	
117	218	-
119	220	
121	222	
123	226	
101	228	
102	230	
104	231	
106		
110	Third Floor	
111	302	
Guest Room	304	
112	306	
114	308	
118	309	
•		

328,

Capen Hall, Building #9

	. *	
Basemen		211
B4	$\frac{e^{-1}}{e^{-1}}$	213
B5		215
B6	••	216
B 7	3	217
B8 -		219
B9	••	221
B10		223
BII		224
B12		225
B13		227
B14		229
B15		201
•	$F_{k+1} = \{ e_{k+1} \in \mathcal{E}_{k+1} \mid e_{k+1} \in \mathcal{E}_{k+1} \}$	202
First Flo	or ·	204
103		206
105		210
107	•	212
108	* · · · · · · · · · · · · · · · · · · ·	214
RD's Apa	riment	218
113		220
115	•	222
116		226
117		228
119	$\frac{1}{2} e^{i x}$	230
121		231
123		er .
101HC	•	Third Floor
102HC		302
104HC		304
110		306
118HC		308
120HC		309
122HC		311
124HC		313
		315
Second F	loor	316
203		317
205		319
207		321
208		323
209	•	324
	4	325

GAGE HALL, BUILDING #21

Basement Rast	First Floor West	218
B 1	**115	220
B2	4117 July 1	221
B3	119	222
B4	121	
B5	127	Second Floor West
B6	129	227
B7	131 / A s	229
B8	133 Overflow	231:
B9	116	233
B10	118	235
	120	237
Basement West	122	243
B11	123	245
B12	124	247
B13	124 Overflow	249
B14	125	225
B15	126	226
B16	1 28	228
B17	130	230
B18	132	232
B19		234
B20	Second Floor East	236
B21	202	238
	204	239
First Floor East	206	240.
101	208	241
103	209	242
105	210	244
107	211	246
108	212	248
108 Overflow	213	
109	215	Third Floor East
110	217	302
111	219	304
112	223	306
113	224	308
114	201	309
100 Overflow	203	310
102	205	311
104	207	312
106	214	313
RD's Apartment	216	315

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Third Floor West 327

BLISS HALL, BUILDING #22

	.*		
BASEMENT			
B00 Overflow	116		Third Floor
BI Overflow	117		301
B2 Overflow	119	erin erin erin erin erin erin erin erin	303
B3	121	•	305
R4	123		307
B5			310
B6	Second Floor		312
B7	201		314
B8	202	e ·	318
B9	204		320
B10	206	٠	322
B11	210	8	326
B13	212		328:
B14	214		330
B15	218	, - ⁸	331
B16	220	•	302
B17 Overflow	222	:	304
	226		306
FIRST FLOOR	228		308
101	230		309
102	231		311
104	203		313
106	205	•	315
110	207		316
111	208		317
Guest Room	209	e e	319
112 Overflow	211		321
112	213		323
114	215	v ,	324
118	216		325
120	217	•	327
122	219		329
124	221		
103	223		
105	224		
107	225		
108	227	•	
109	229		
Director's Apartment			
113			

APPENDIX B
EPA METHOD 8080

METHOD 8080A

ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHY

1.0 SCOPE AND APPLICATION

1.1 Method 8080 is used to determine the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs). The following compounds can be determined by this method:

	Compound Name	CAS No.*
	Aldrin	309-00-2
	a-BHC	319-84-6
	β-BHC	319-85-7
. "	δ-BHC	319-86-8
	γ-BHC (Lindane)	58-89-9
	Chlordane (technical)	12789-03-6
	4,4'-DDD	72-54-8
	4,4'-DOE	72-55 -9
	4,4'-DOT	50-2 9-3 *
-	Dieldrin	60-57-1
	Endosulfan I	959-98-8
	Endosulfan II	33212-65-9
	Endosulfan sulfate	1031-07-8
	Endrin	72-20-8
	Endrin aldehyde	7421-93-4
	Heptachlor	76-44-8
	Heptachlor epoxide	1024-57-3
	4,4'-Methoxychlor	72-43-5
- **	Toxaphene	8001-35-2
	Aroclar-1016	12674-11-2
	Aroclor-1221	1104-28-2
•	Aroclor-1232	11141-16-5
•	Aroclor-1242	53469-21-9
	Areclar-1248	12672-29-6
	Aroclor-1254	11097-69-1
2	Aroclor-1260	11096-82-5

a Chemical Abstract Services Registry Number.

^{1.2} Table I lists the method detection limit for each compound in organic-free reagent water. Table 2 lists the estimated quantitation limit (EQL) for other matrices.

2.0 SUMMARY OF METHOD

- 2.1 Method 8080 provides gas chromatographic conditions for the detection of ppb concentrations of certain organochlorine pesticides and PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, Waste Dilution) may be analyzed by direct injection. A 2 to 5 μL sample is injected into a gas chromatograph (GC) using the solvent flush technique, and compounds in the GC effluent are detected by an electron capture detector (ECD) or an electrolytic conductivity detector (HECD).
- 2.2 The sensitivity of Method 8080 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8080 may also be performed on samples that have undergone cleanup. Method 3620, Florisil Column Cleanup, by itself or followed by Method 3660, Sulfur Cleanup, may be used to eliminate interferences in the analysis.

3.0 INTERFERENCES

- 3.1 Refer to Methods 3500, 3600, and 8000.
- 3.2 Interferences by phthalate esters can pose a major problem in pesticide determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large late-eluting peaks, especially in the 15% and 50% fractions from the Florisil cleanup. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination. The contamination from phthalate esters can be completely eliminated with a microcoulometric or electrolytic conductivity detector.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 Gas Chromatograph: Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended.

4.1.2 Columns

4.1.2.1 Column 1: Supelcopert (100/120 mesh) coated with 1.5% SP-2250/1.95% SP-2401 packed in a 1.8 m x 4 mm ID glass column or equivalent.

- 4.1.2.2 Column 2: Supelcoport (100/120 mesh) coated with 3% OV-1 in a 1.8 m x 4 mm ID glass column or equivalent.
- 4.1.3 Detectors: Electron capture (ECD) or electrolytic conductivity detector (HECD).
- 4.2 Kuderna-Danish (K-D) apparatus:
- 4.2.1 Concentrator tube: 10 mL, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.
- 4.2.2 Evaporation flask: 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.
- 4.2.3 Snyder column: Three ball macro (Kontes K-503000-0121 or equivalent).
- 4.2.4 Snyder column: Two ball micro (Kontes K-569001-0219 or equivalent).
 - 4.2.5 Springs 1/2 inch (Kontes K-662750 or equivalent).
- 4.3 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).
- 4.4 Water bath: Heated, with concentric ring cover, capable of temperature control $(\pm 5^{\circ}\text{C})$. The bath should be used in a hood.
- 4.5 Volumetric flasks, Class A: sizes as appropriate with ground-glass stoppers.
 - 4.6 Microsyringe: $10 \mu L$.
 - 4.7 Syringe: 5 mL.
- 4.8 Vials: Glass, 2, 10, and 20 mL capacity with Teflon-lined screw caps or crimp tops.
 - 4.9 Balances: Analytical, 0.0001 g and Top loading, 0.01 g.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

5.3 Solvents

- 5.3.1 Hexane, CoH14 Pesticide quality or equivalent.
- 5.3.2 Acetone, CH3COCH3 Pesticide quality or equivalent.
- 5.3.3 Toluene, CoH5CH3 Pesticide quality or equivalent.
- 5.3.4 Isooctane, (CH₃)₃CCH₂CH(CH₃)₂ Pesticide quality or equivalent.

5.4 Stock standard solutions:

- 5.4.1 Prepare stock standard solutions at a concentration of 1000 mg/L by dissolving 0.0100 g of assayed reference material in isooctane and diluting to volume in a 10 mL volumetric flask. A small volume of toluene may be necessary to put some pesticides in solution. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.
- 5.4.2 Transfer the stock standard solutions into vials with Teflonlined screw caps or crimp tops. Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.
- 5.4.3 Stock standard solutions must be replaced after one year, or sooner if comparison with check standards indicates a problem.
- 5.5 Calibration standards: Calibration standards at a minimum of five concentrations for each parameter of interest are prepared through dilution of the stock standards with isooctane. One of the concentrations should be at a concentration near, but above, the method detection limit. The remaining concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions must be replaced after six months, or sooner, if comparison with check standards indicates a problem.
- 5.6 Internal standards (if internal standard calibration is used): To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.
 - 5.6.1 Prepare calibration standards at a minimum of five concentrations for each analyte of interest as described in Sec. 5.5.

- 5.6.2 To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane.
 - 5.6.3 Analyze each calibration standard according to Sec. 7.0.
- 5.7 Surrogate standards: The analyst should monitor the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and organic-free reagent water blank with pesticide surrogates. Because GC/ECD data are much more subject to interference than GC/MS, a secondary surrogate is to be used when sample interference is apparent. Two surrogate standards (tetrachloro-m-xylene (TCMX) and decachlorobiphenyl) are added to each sample; however, only one need be calculated for recovery. Proceed with corrective action when both surrogates are out of limits for a sample (Sec. 8.3). Method 3500 indicates the proper procedure for preparing these surrogates.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1. Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction:

- 7.1.1 Refer to Chapter Two for guidance on choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral, or as is, pH with methylene chloride, using either Method 3510 or 3520. Solid samples are extracted using Method 3540, 3541, or 3550.
- 7.1.2 Prior to gas chromatographic analysis, the extraction solvent must be exchanged to hexane. The exchange is performed during the K-D procedures listed in all of the extraction methods. The exchange is performed as follows.
 - 7.1.2.1 Following K-D of the methylene chloride extract to 1 mL using the macro-Snyder column, allow the apparatus to cool and drain for at least 10 min.
 - 7.1.2.2 Increase the temperature of the hot water bath to about 90°C. Momentarily remove the Snyder column, add 50 mL of hexane, a new boiling chip, and reattach the macro-Snyder column. Concentrate the extract using 1 mL of hexane to prewet the Snyder column. Place the K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration in 5-10 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid

reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

- 7.1.2.3 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of hexane. A 5 mL syringe is recommended for this operation. Adjust the extract volume to 10.0 mL. Stopper the concentrator tube and store refrigerated at 4°C, if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a vial with a Teflon-lined screw cap or crimp top. Proceed with gas chromatographic analysis if further cleanup is not required.
- 7.2 Gas chromatography conditions (Recommended):

7.2.1 Column 1:

Carrier gas (5% methane/95% argon) flow rate: 60 mL/min Column temperature: 200°C isothermal

When analyzing for the low molecular weight PCBs (PCB 1221-PCB 1248), it is advisable to set the oven temperature to 160°C.

7.2.2 Column 2:

Carrier gas (5% methane/95% argon) flow rate: 60 mL/min 200°C isothermal

When analyzing for the low molecular weight PCBs (PCB 1221-PCB 1248), it is advisable to set the oven temperature to 140°C.

- 7.2.3 When analyzing for most or all of the analytes in this method, adjust the oven temperature and column gas flow to provide sufficient resolution for accurate quantitation of the analytes. This will normally result in a retention time of 10 to 12 minutes for 4,4'-DDT, depending on the packed column used.
- 7.3 Calibration: Refer to Method 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.
 - 7.3.1 The procedure for internal or external calibration may be used. Refer to Method 8000 for a description of each of these procedures.
 - 7.3.2 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day. Therefore, the GC column should be primed or deactivated by injecting a PCB or pesticide standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this prior to beginning initial or daily calibration.

7.4 Gas chromatographic analysis:

- 7.4.1 Refer to Method 8000. If the internal standard calibration technique is used, add 10 μL of internal standard to the sample prior to injection.
- 7.4.2 Method 8000 provides instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-concentration standard after each group of 10 samples in the analysis sequence.

NOTE: A 72 hour sequence is not required with this method.

- 7.4.3 Examples of GC/ECD chromatograms for various pesticides and PCBs are shown in Figures 1 through 5.
 - 7.4.4 Prime the column as per Sec. 7.3.2.
- 7.4.5 DDT and endrin are easily degraded in the injection port if the injection port or front of the column is dirty. This is the result of buildup of high boiling residue from sample injection. Check for degradation problems by injecting a mid-concentration standard containing only 4,4'-DDT and endrin. Look for the degradation products of 4,4'-DDT (4,4'-DDE and 4,4'-DDD) and endrin (endrin ketone and endrin aldehyde). If degradation of either DDT or endrin exceeds 20%, take corrective action before proceeding with calibration, by following the GC system maintenance outlined in of Method 8000. Calculate percent breakdown as follows:

Total DDT degradation peak area (DDE + DDD)

Total DDT peak area (DDT + DDE + DDD)

Total endrin degradation peak area (endrin aldehyde + endrin ketone)

Total endrin peak area (endrin + endrin aldehyde + endrin ketone)

- 7.4.6 Record the sample volume injected and the resulting peak sizes (in area units or peak heights).
- 7.4.7 Using either the internal or external calibration procedure (Nethod 8000), determine the identity and quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes.
- 7.4.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo cleanup using Method 3620. The resultant extract(s) may be analyzed by GC directly or may undergo further cleanup to remove sulfur using Method 3660.

7.5 Cleanup:

- 7.5.1 Proceed with Method 3620, followed by, if necessary, Method 3660, using the 10 mL hexane extracts obtained from Sec. 7.1.2.3.
- 7.5.2 Following cleanup, the extracts should be analyzed by GC, as described in the previous sections and in Method 8000.
- 7.5.3 If only PCBs are to be measured in a sample, the sulfuric acid/permanganate cleanup (Method 3665), followed by Silica Cleanup (Method 3630) or Florisil Cleanup (Method 3620), is recommended.

7.6 Calculations (excerpted from U.S. FDA, PAM):

- 7.6.1 Calculation of Certain Residues: Residues which are mixtures of two or more components present problems in measurement. When they are found together, e.g., toxaphene and DDT, the problem of quantitation becomes even more difficult. In the following sections suggestions are offered for handling toxaphene, chlordane, PCB, DOT, and BHC. A 10% DC-200 stationary phase column was used to obtain the chromatograms in Figures 6-9.
- 7.6.2 Toxaphene: Quantitative calculation of toxaphene or Strobane is difficult, but reasonable accuracy can be obtained. To calculate toxaphene on GC/ECD: (a) adjust sample size so that toxaphene major peaks are 10-30% full-scale deflection (FSD); (b) inject a toxaphene standard that is estimated to be within ±10 ng of the sample; (c) construct the baseline of standard toxaphene between its extremities; and (d) construct the baseline under the sample, using the distances of the peak troughs to baseline on the standard as a guide (Figures 7, 8, and 9). This procedure is made difficult by the fact that the relative heights and widths of the peaks in the sample will probably not be identical to the standard. A toxaphene standard that has been passed through a Florisil column will show a shorter retention time for peak X and an enlargement of peak Y.
- 7.6.3 Toxaphene and ODT: If DDT is present, it will superimpose itself on toxaphene peak V. To determine the approximate baseline of the DDT, draw a line connecting the trough of peaks V and V with the trough of peaks W and X and construct another line parallel to this line which will just cut the top of peak W (Figure 61). This procedure was tested with ratios of standard toxaphene-DDT mixtures from 1:10 to 2:1 and the results of added and calculated DDT and toxaphene by the "parallel lines" method of baseline construction were within 10% of the actual values in all cases.
 - 7.6.3.1 A series of toxaphene residues have been calculated using total peak area for comparison to the standard and also using area of the last four peaks only in both sample and standard. The agreement between the results obtained by the two methods justifies the use of the latter method for calculating toxaphene in a sample where the early eluting portion of the toxaphene chromatogram is interfered with by other substances.

- 7.6.3.2 The baseline for methoxychlor superimposed on toxaphene (Figure 8b) was constructed by overlaying the samples on a toxaphene standard of approximately the same concentration (Figure 8a) and viewing the charts against a lighted background.
- 7.6.4 Chlordane is a technical mixture of at least 11 major components and 30 or more minor ones. Gas chromatography-mass spectrometry and nuclear magnetic resonance analytical techniques have been applied to the elucidation of the chemical structures of the many chlordane constituents. Figure 9a is a chromatogram of standard chlordane. Peaks E and F are responses to trans- and cis-chlordane, respectively. These are the two major components of technical chlordane, but the exact percentage of each in the technical material is not completely defined and is not consistent from batch to batch. Other labelled peaks in Figure 9a are thought to represent: A, monochlorinated adduct of pentachlorocyclopentadiene with cyclopentadiene; B, coelution heptachlor and α -chlordene; C, coelution of β -chlordene and γ -chlordene; D, a chlordane analog; G, coelution of cis-nonachlor and Compound K, " a chlordane isomer. The right "shoulder" of peak F is caused by transnonachlor.
 - 7.6.4.1 The GC pattern of a chlordane residue may differ considerably from that of the technical standard. Depending on the sample substrate and its history, residues of chlordane can consist of almost any combination of constituents from the technical chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight. Only limited information is available on which residue GC patterns are likely to occur in which samples types, and even this information may not be applicable to a situation where the route of exposure is unusual. For example, fish exposed to a recent spill of technical chlordane will contain a residue drastically different from a fish whose chlordane residue was accumulated by ingestion of smaller fish or of vegetation, which in turn had accumulated residues because chlordane was in the water from agricultural runoff.
 - 7.6.4.2 Because of this inability to predict a chlordane residue GC pattern, it is not possible to prescribe a single method for the quantitation of chlordane residues. The analyst must judge whether or not the residue's GC pattern is sufficiently similar to that of a technical chlordane reference material to use the latter as a reference standard for quantitation.
 - 7.6.4.3 When the chlordane residue does not resemble technical chlordane, but instead consists primarily of individual, identifiable peaks, quantitate each peak separately against the appropriate reference materials and report the individual residues. (Reference materials are available for at least 11 chlordane constituents, metabolites or degradation products which may occur in the residue.)

- 7.6.4.4 When the GC pattern of the residue resembles that of technical chlordane, quantitate chlordane residues by comparing the total area of the chlordane chromatogram from peaks A through F (Figure 9a) in the sample versus the same part of the standard chromatogram. Peak G may be obscured in a sample by the presence of other pesticides. If G is not obscured, include it in the measurement for both standard and sample. If the heptachlor epoxide peak is relatively small, include it as part of the total chlordane area for calculation of the residue. If heptachlor and/or heptachlor epoxide are much out of proportion as in Figure 6j, calculate these separately and subtract their areas from total area to give a corrected chlordane area. (Note that octachlor epoxide, a metabolite of chlordane, can easily be mistaken for heptachlor epoxide on a nonpolar GC column.)
- To measure the total area of the chlordane 7.6.4.5 chromatogram, proceed as in Sec. 7.6.2 on toxaphene. Inject an amount of technical chlordane standard which will produce a chromatogram in which peaks E and F are approximately the same size as those in the sample chromatograms. Construct the baseline beneath the standard from the beginning of peak A to the end of peak F as shown in Figure 9a. Use the distance from the trough between peaks E and F to the baseline in the chromatogram of the standard to construct the baseline in the chromatogram of the sample. Figure 9b shows how the presence of toxaphene causes the baseline under chlordane to take an upward angle. When the size of peaks E and F in standard and sample chromatograms are the same, the distance from the trough of the peaks to the baselines should be the same. Measurement of chlordane area should be done by total peak area if possible.
 - NOTE: A comparison has been made of the total peak area integration method and the addition of peak heights method for several samples containing chlordane. The peak heights A, B, C, D, E, and F were measured in millimeters from peak maximum of each to the baseline constructed under the total chlordane area and were then added together. These results obtained by the two techniques are too close to ignore this method of "peak height addition" as a means of calculating chlordane. The technique has inherent difficulties because not all the peaks are symmetrical and not all are present in the same ratio in standard and in sample. This method does offer a means of calculating results if no means of measuring total area is practical.
- 7.6.5 Polychlorinated biphenyls (PCBs): Quantitation of residues of PCB involves problems similar to those encountered in the quantitation of toxaphene, Strobane, and chlordane. In each case, the chemical is made up of numerous compounds. So the chromatograms are multi-peak. Also in each case, the chromatogram of the residue may not match that of the standard.
 - 7.6.5.1 Mixtures of PCBs of various chlorine contents were sold for many years in the U.S. by the Monsanto Co. under the

tradename Aroclor (1200 series and 1016). Though these Aroclors are no longer marketed, the PCBs remain in the environment and are sometimes found as residues in foods, especially fish.

- 7.6.5.2 PCB residues are quantitated by comparison to one or more of the Aroclor materials, depending on the chromatographic pattern of the residue. A choice must be made as to which Aroclor or mixture of Aroclors will produce a chromatogram most similar to that of the residue. This may also involve a judgment about what proportion of the different Aroclors to combine to produce the appropriate reference material.
- 7.6.5.3 Quantitate PCB residues by comparing total area or height of residue peaks to total area of height of peaks from appropriate Aroclor(s) reference materials. Measure total area or height response from common baseline under all peaks. Use only those peaks from the sample that can be attributed to chlorobiphenyls. These peaks must also be present in the chromatogram of the reference materials. Mixtures of Aroclors may be required to provide the best match of GC patterns of sample and reference.
- 7.6.6 DDT: DDT found in samples often consists of both o,p'- and p,p'-DDT. Residues of DDE and DDD are also frequently present. Each isomer of DDT and its metabolites should be quantitated using the pure standard of that compound and reported as such.
- 7.6.7 Hexachlorocyclohexane (BHC, from the former name, benzene hexachloride): Technical grade BHC is a cream-colored amorphous solid with a very characteristic musty odor; it consists of a mixture of six chemically distinct isomers and one or more heptachloro-cyclohexanes and octachloro-cyclohexanes.
 - 7.6.7.1 Commercial BHC preparations may show a wide variance in the percentage of individual isomers present. The elimination rate of the isomers fed to rats was 3 weeks for the α -, γ -, and δ -isomers and 14 weeks for the β -isomer. Thus it may be possible to have any combination of the various isomers in different food commodities. BHC found in dairy products usually has a large percentage of β -isomer.
 - 7.6.7.2 Individual isomers $(\alpha, \beta, \gamma, \text{ and } \delta)$ were injected into gas chromatographs equipped with flame ionization, microcoulometric, and electron capture detectors. Response for the four isomers is very nearly the same whether flame ionization or microcoulometric GLC is used. The α -, γ -, and δ -isomers show equal electron affinity. β -BHC shows a much weaker electron affinity compared to the other isomers.
 - 7.6.7.3 Quantitate each isomer (α , β , γ , and δ) separately against a standard of the respective pure isomer, using a GC column which separates all the isomers from one another.

8.0 QUALITY CONTROL

- 8.1 Refer to Chapter One for specific quality control procedures. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.
- 8.2 Quality control required to evaluate the GC system operation is found in Method 8000.
 - 8.2.1 The quality control check sample concentrate (Method 8000) should contain each single-component parameter of interest at the following concentrations in acetone or other water miscible solvent: 4,4'-DOD, 10 mg/L; 4,4'-DOT, 10 mg/L; endosulfan II, 10 mg/L; endosulfan sulfate, 10 mg/L; endrin, 10 mg/L; and any other single-component pesticide, 2 mg/L. If this method is only to be used to analyze for PCBs, chlordane, or toxaphene, the QC check sample concentrate should contain the most representative multi-component parameter at a concentration of 50 mg/L in acetone.
 - 8.2.2 Table 3 indicates the QC acceptance criteria for this method. Table 4 gives method accuracy and precision as functions of concentration for the analytes of interest. The contents of both Tables should be used to evaluate a laboratory's ability to perform and generate acceptable data by this method.
- 8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits (limits established by performing QC procedures outlined in Method 8000).
 - 8.3.1 If recovery is not within limits, the following is required.
 - Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
 - Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
 - Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration".
- 8.4 <u>GC/MS confirmation</u>: Any compounds confirmed by two columns may also be confirmed by GC/MS if the concentration is sufficient for detection by GC/MS as determined by the laboratory generated detection limits.
 - 8.4.1 The GC/MS would normally require a minimum concentration of 10 ng/ μ L in the final extract, for each single-component compound.
 - 8.4.2 The pesticide extract and associated blank should be analyzed by GC/MS as per Sec. 7.0 of Method 8270.

- 8.4.3 The confirmation may be from the GC/MS analysis of the base/neutral-acid extractables extracts (sample and blank). However, if the compounds are not detected in the base/neutral-acid extract even though the concentration is high enough, a GC/MS analysis of the pesticide extract should be performed.
- 8.4.4 A reference standard of the compound must also be analyzed by GC/MS. The concentration of the reference standard must be at a level that would demonstrate the ability to confirm the pesticides/PCBs identified by GC/ECD.

9.0 METHOD PERFORMANCE

- 9.1 The method was tested by 20 laboratories using organic-free reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations. Concentrations used in the study ranged from 0.5 to 30 μ g/L for single-component pesticides and from 8.5 to 400 μ g/L for multi-component parameters. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships for an electron capture detector are presented in Table 4.
- 9.2 The accuracy and precision obtained will be determined by the sample matrix, sample-preparation technique, optional cleanup techniques, and calibration procedures used.

10.0 REFERENCES

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TABLE 1.
GAS CHROMATOGRAPHY OF PESTICIDES AND PCBs*

•	Retention time (min)			Method	
Analyte	Col. 1	Co1. 2		Detection limit (μg/L	
Aldrin	2.40	4.10		0.004	
œ-BHC	1.35	1.82		0.003	
6-BHC	1.90	1.97	**	0.006	
s-BHC	2.15	2.20		0.009	
y-BHC (Lindane)	1.70	2.13	t+	0.004	
chlordane (technical)	e	•		0.014	
1,4'- 00 0	7.83	9.08		0.011	
,4'-DDE	5.13	7.15		0.004	
,4'-DOT	9.40	11.75	•	0.012	
ieldrin	5.45	7.23		0.002	
ndosulfan I	4.50	6.20	•	0.014	
indosulfan II	8.00	8.28		0.004	
ndosulfan sulfate	14.22	10.70		0.066	
ndrin	6.55	8.10		0.006	
ndrin aldehyde	11.82	9.30		0.023	
eptachlor	2.00	3.35		0.003	
eptachlor epoxide	3.50	5.00		0.083	
ethoxychlor	18.20	26.60	· · · · · · · · · · · · · · · · · · ·	0.176	
oxaphene	e		1.0	0.24	
CB-1016	e	e	•	, nd	
CB-1221	6	e	26 16	nd	
CB-1232	e	e		nd	
CB-1242	ē	e	,s	0.065	
CB-1248	e	e	* ! *=	nd	
CB-1254	ě	ē	7	nd	
CB-1260	ē	e .	4	nd nd	

^{*}U.S. EPA. Method 617. Organochlorine Pesticides and PCBs. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

e = Multiple peak response.

nd = not determined.

TABLE 2. DETERMINATION OF ESTIMATED QUANTITATION LIMITS (EQLs) FOR VARIOUS MATRICES*

Matrix			ang A	Factor
			4 - A.	
Ground water Low-concentration soil High-concentration soil Non-water miscible wast	and sludges	with by son	GPC cleanup ication	10 670 10,000 100,000

a EQL = [Method detection limit (see Table 1)] X [Factor found in this table]. For non-aqueous samples, the factor is on a wet-weight basis. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable.

TABLE 3. QC ACCEPTANCE CRITERIA*

Analyte	Test conc. (µg/L)	Limit for s (µg/L)	Rang <u>e</u> for x (µg/L)	Range P, P, (%)
Aldrin	2.0	0.42	1.08-2.24	42 120
α-BHC	2.0	0.48	0.98-2.44	42-122 37-134
β-BHC	2.0	0.64	0.78-2.60	17-147
δ-BHC	2.0	0.72	1.01-2.37	19-140
γ-BHC	2.0	0.46	0.86-2.32	32-127
Chlordane Chlordane	50	10.0	27.6-54.3	45-119
4,4'-000	10	2.8	4.8-12.6	31-141
4,4'-DDE	2.0	0.55	1.08-2.60	30-145
4,4'-DDT	10	3.6	4.6-13.7	25-160
Dieldrin	2.0	0.76	- 1.15-2.49	36-146
Endosulfan I	2.0	0.49	1.14-2.82	45-153
Endosulfan II	10	6.1	2.2-17.1	D-202
Endosulfan Sulfate	10	2.7	3.8-13.2	26-144
Endrin	10	3.7	5.1-12.6	30-147
leptachlor , , ,	2.0	0.40	0.86-2.00	34-111
eptachlor epoxide	2.0	0.41	1.13-2.63	37-142
oxaphene	50	12.7	27.8-55.6	41-126
C8-1016	50	10.0	30.5-51.5	50-114
CB-1221	50	24.4	22.1-75.2	15-178
CB-1232	50	17.9	14.0-98.5	10-215
CB-1242	50	12.2	24.8-69.6	39-150
CB-1248	50	15.9	29.0-70.2	38-158
CB-1254	50	13.8	22.2-57.9	29-131
CB-1260	50	10.4	18.7-54.9	8-127

s = Standard deviation of four recovery measurements, in $\mu g/L$.

*Criteria from 40 CFR Part 136 for Method 608. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

 $[\]overline{x}$ = Average recovery for four recovery measurements, in $\mu g/L$.

P, P. = Percent recovery measured.

D = Detected; result must be greater than zero.

TABLE 4.
METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION®

	Accuracy, as recovery, x'	Single analyst precision, s,'	Overall precision,
Analyte	(µg/L)	(μg/L)	S' (μg/L)
Aldrin	0.81C+0.04	0.16x-0.04	0.20x-0.01
α-BHC	0.84C+0.03	0.13x+0.04	$0.23\overline{x}-0.00$
B-BHC	0.81C+0.07	0.22x+0.02	0.33x-0.95
S-BHC	0.81C+0.07	0.18x+0.09	0.25x+0.03
Y-BHC	0.82C-0.05	0.12x+0.06	0.22x+0.04
Ćhlordane	0.82C-0.04	0.13x+0.13	0.18x+0.18
4,4'-000	0.840+0.30	$0.20\overline{x}-0.18$	$0.27\bar{x}-0.14$
4,4'-DDE	0.85C+0.14	0.13x+0.06	0.28x-0.09
4,4'-DDT	0.93C-0.13	$0.17\bar{x}+0.39$	$0.31\bar{x}-0.21$
Dieldrin	0.90C+0.02	0.12x+0.19	0.16x+0.16
Endosulfan I	0.97C+0.04	0.10x+0.07	0.18x+0.08
Endosulfan II	0.93C+0.34	$0.41\bar{x}$ -0.65	0.47x-0.20
Endosulfan Sulfate	0.89C-0.37	0.13x+0.33	0.24x+0.35
Endrin	0.89C-0.84	0.20x+0.25	0.24x+0.25
leptachlor	0.69C+0.04	0.06x+0.13	0.16x+0.08
deptachlor epoxide	0.89C+0.10	0.18x-0.11	0.25x-0.08
Toxaphene	0.80C+1.74	0.09x+3.20	0.20x+0.22
PCB-1016	0.81C+0.50	$0.13\overline{x} + 0.15$	$0.15\bar{x} + 0.45$
PCB-1221	0.96C+0.65	0.29x-0.76	$0.35\bar{x}-0.62$
PC8-1 23 2	0.91C+10.79	$0.21\bar{x}-1.93$	0.31x + 3.50
PCB-1242	0.91C+10.79	$0.21\bar{x}-1.93$	$0.31\bar{x} + 3.50$
PCB-1248	0.91C+10.79	$0.21\bar{x}-1.93$	0.31x + 3.50
PCB-1254	0.91C+10.79	$0.21\overline{x}-1.93$	0.31x+3.50
PCB-1260	0.91C+10.79	$0.21\overline{x}-1.93$	$0.31\overline{x} + 3.50$

x' = Expected recovery for one or more measurements of a sample containing concentration C, in $\mu g/L$.

s,' = Expected single analyst standard deviation of measurements at an average concentration of \bar{x} , in $\mu g/L$.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of x, in $\mu g/L$.

C = True value for the concentration, in $\mu g/L$.

 $[\]overline{x}$ - Average recovery found for measurements of samples containing a concentration of C, in $\mu g/L$.

Figure 1
Gas Chromatogram of Pesticides

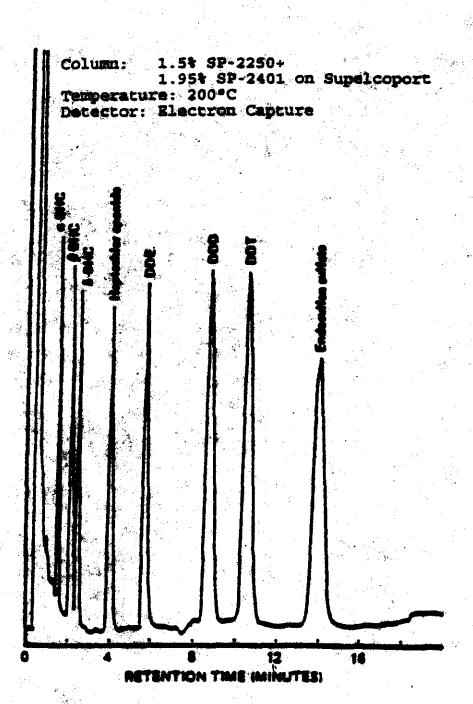


Figure 2
Gas Chromatogram of Chlordane

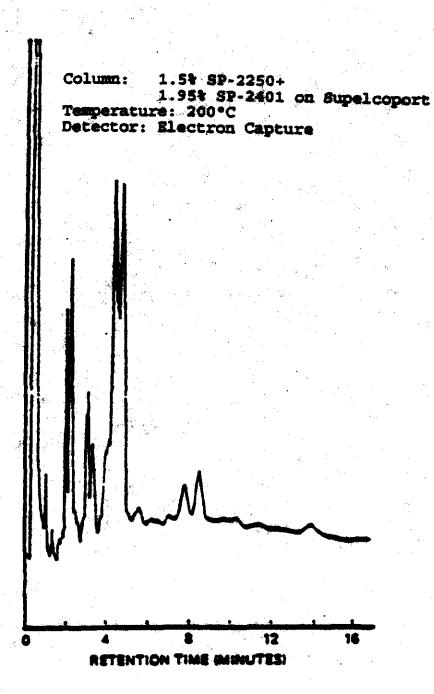


Figure 3
Gas Chromatogram of Toxaphene

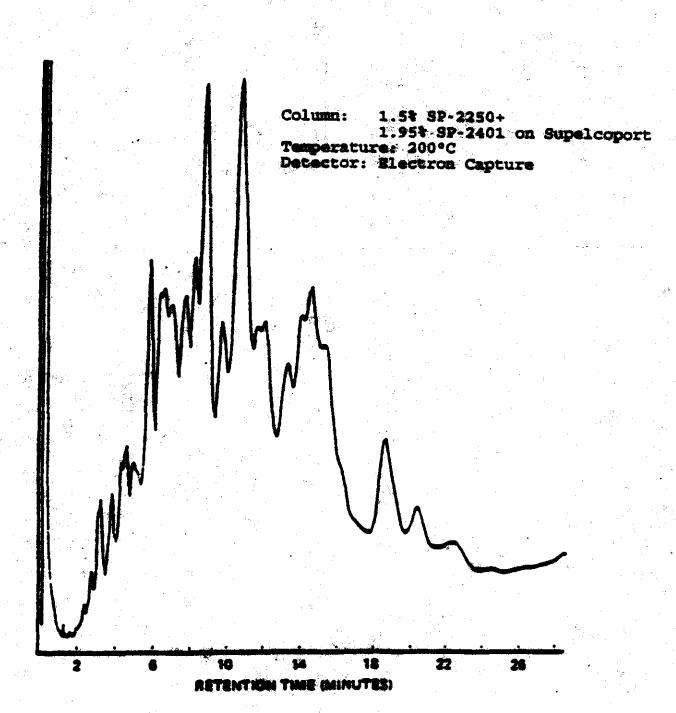


Figure 4
Gas Chromatogram of Aroclor 1254

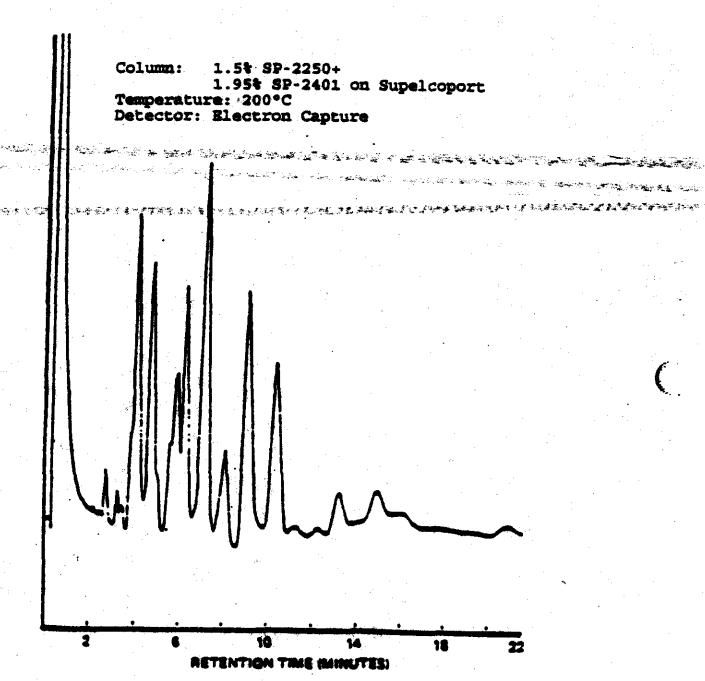
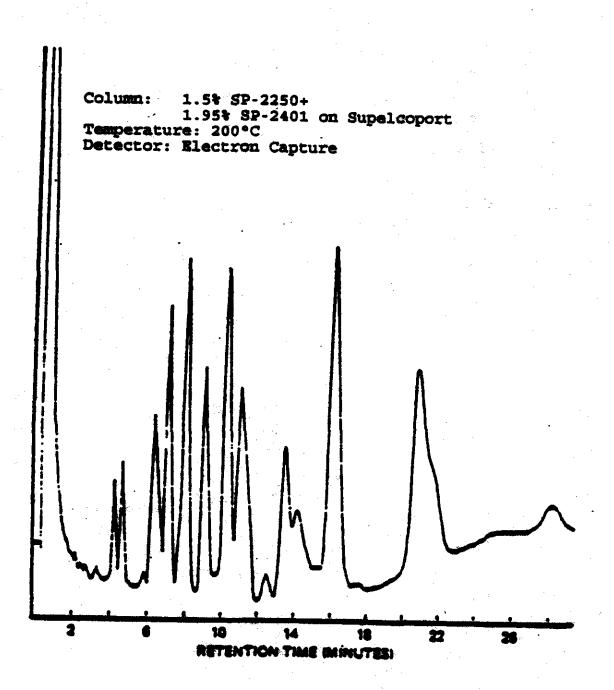


Figure 5
Gas Chromatogram of Aroclor 1260



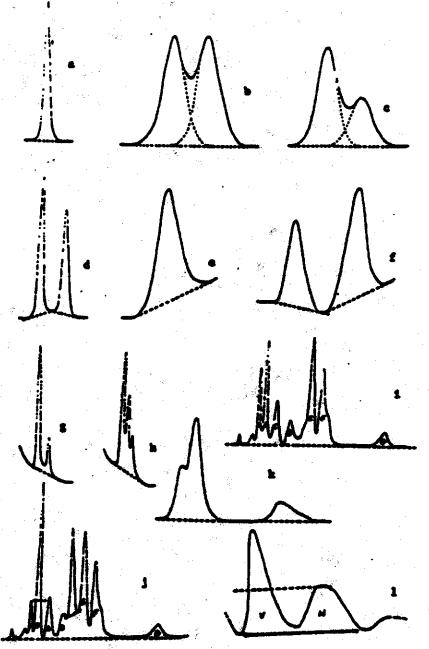


Fig. 6-Baseline construction for some typical gas chromotagraphic peaks. a: symmetrical separated flat baseline; b and c: overlapp flat baseline; d: separated (pen does not return to baseline between peaks); e: separated sloping baseline; f: separated (pen goes below baseline between peaks); g: α - and γ -BHC sloping baseline; h: α -, β - and γ -BHC sloping baseline; i: chlordane flat baseline; j: heptachior and heptachlor epoxide superimposed on chlordane; k: chair-shaped peaks, unsymmetrical peak; l: p,p'-DBT superimposed on toxaphene.

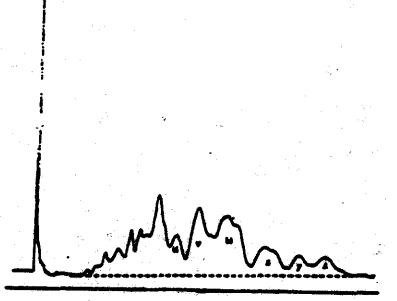


Fig. - 7a -- Baseline construction for multiple residues with standard toxaphene.

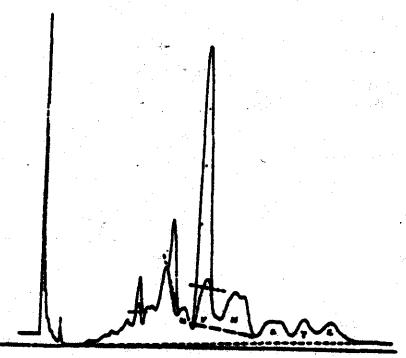


Fig. - 7b -- Baseline construction for multiple residues with toxaphene, DDE and o,p'-, and p,p'-DDT

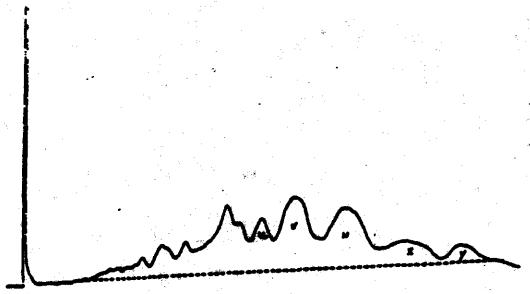


Fig. - &a -- Baseline construction for multiple residues: standard tomphene.

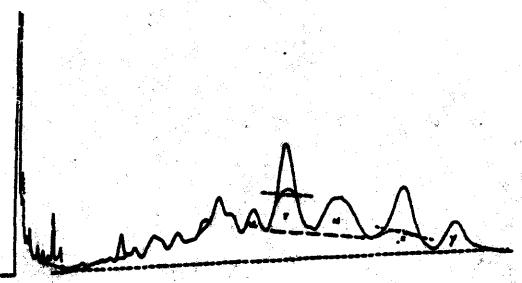


Fig. - 8b -- Baseline construction for multiple residues: sice bran with BHC, toomphene, DDT, and methosychlor.

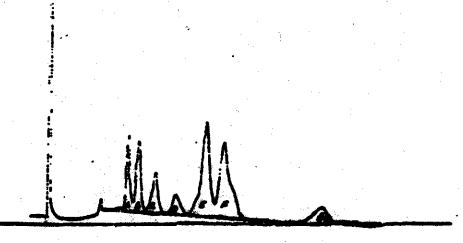


Fig. - 9a -- Bessline construction for multiple residues: standard chlordene.

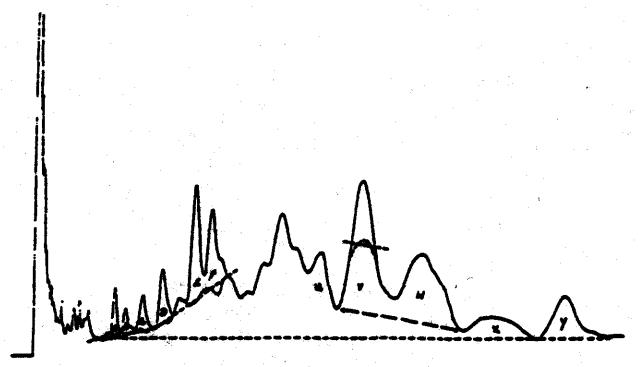
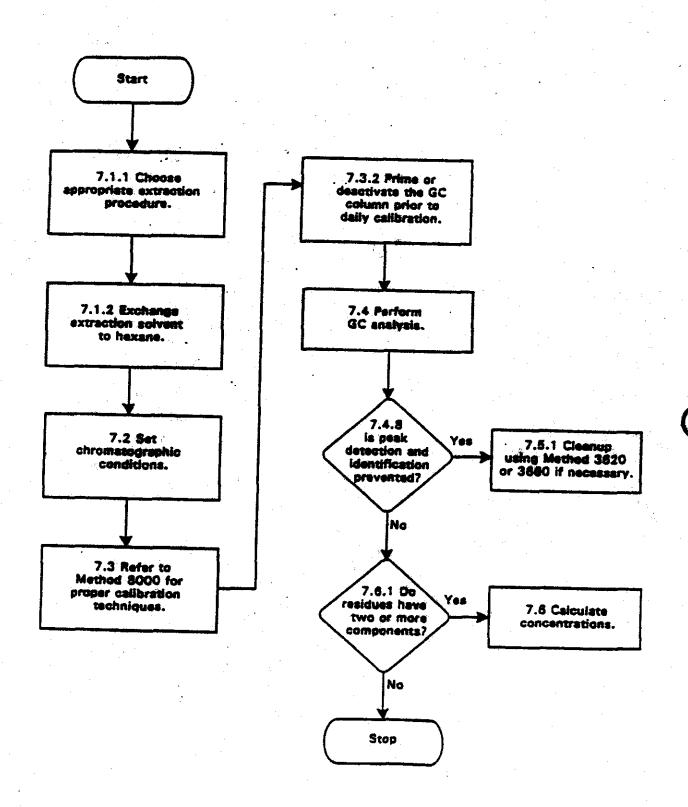


Fig. - 9b -- Baseline construction for multiple residues: rice bran with chlordene, toxaphene, and DDT.

ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHY



APPENDIX C
NEW YORK STATE DEPARTMENT OF HEALTH
METHOD 311-1

NEW YORK STRIE DEPARTMENT OF HEALTH WADSNORTH CENTER FOR LABORATORIES AND RESEARCH ALEANY, N.Y. 12201

10/6/81

POLYCHICRINATED BIFHENYIS IN AMBIENT ATR

1. Scope and Application

- 1.1 This method covers the determination of polychlorinated higheryls (PCBs) as Arcclors 1016/1242, 1221, 1254, 1260 in ambient air.
- 1.2 A 24 hour sample containing Arcelor 1016 collected at approximately 1.0 liter per minute is efficiently trapped in one cartridge, even at 95% relative hamidity.
- 1.3 The minimum detactable concentration is 0.033 ug/1.5 ml of extract which is approximately 0.02 ug/cubic meter when sampled for 24 hours at 1 to 2 liters per mirate.

2. Summary of Method

2.1 Polychlorinated biphenyls are quantitatively trapped on florisil in a cartridge. The PCB is described by extraction with herane. Sample clean-up is performed, if necessary. Analysis for PCBs is carried out on a gas chromatograph with a 3t SE-30 on CAS-CERCM Q column and electron capture detector. Results are reported in ug/cubic meter.

3. Interferences

- 3.1 Solvents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated base lines, causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis. Specific selection of respents and purification of solvents by distillation in all-glass systems may be required. Refer to Section 510-1, Analytical Handbook.
- 3.2 Organophosphorus pesticides a number of organophosphorus pesticides, such as those containing a nitro group, e.g., parathion, also respond to the electron capture detector and may interfere with the determination of the organochlorine pesticides. Such compounds can be identified by their response to the flame photometric detector.
- 3.3 Interferences from chlorinated pesticides can be removed, if necessary, according to the method of Trotter.

4. Apparatus and Materials

- 4.1 Pasteur pipets no. P5180-2, 5-3/4 inch length
- 4.2 Pasteur pipet bulb
- 4.3 Air or nitrogen for evaporating sample
- 4.4 Vacuum pump capable of maintaining an air pressure differential greater than 0.8 atmosphere at the desired flow rate
- 4.5 Muffle furnace equipped with temperature control and indicator
- 4.6 Gas chromatograph:
 - 4.6.1 Electron capture detector
 - 4.6.2 Column dimensions 6' X 1/4"
 - 4.6.3 3% SE-30 on GAS-CHROM Q, 80/100 mesh, 2 mm I.D., Conditioned at 220 C.
 - 4.6.4 Alternate column 1.5% OV17/1.95% QF-1, on GAS-CERCM Q,80/100
- 4.7 Test tubes graduated, ground-glass stoppered, 5 or 10 ml size
- 4.8 Tygon tubing 1/4" I.D., 3/8" O.D. for cartridge connections
- 4.9 Teclion tubing 7 mm I.D.
- 4.10 Caplugs size 9/32 in SC (Caplugs Division, Protective Closures Co. Inc., 2150 Klascod Rvs., Buffalo, NY)
- 4.11 Wrist Action Shaker, Burrell.

5. Reagents, Solvents and Standards

- 5.1 Florisil 60-100 mesh, Fisher Scientific Co., #F-100. Wesh Florisil with several portions of herane and dry on a steembath to remove herane residue. Place Florisil in a muffle furnece at 320 C overnight. Descrivate the Florisil by shaking 100 grams with 2 ml distilled water for 15 minutes on a mechanical wrist action shaker. Store Florisil in a screw-cap jar.
- 5.2 Glass wool hexane rinsed
- 5.3 Hexanes nanograde, Mallinckrodt, no. 4159

- 5.4 PCB standards (Monsento) Arcelor 1016, 1221, 1254,1260. Other Arcelors may be of interest, these are the most common. For each Arcelor of interest prepare the following dilute standards:
 - 5.4.1 1 mg/ml stock PCB solution weigh 100 mg of PCB and place in a 100 ml volumetric flask. Dilute to the mark with hexane.
 - 5.4.2 10 ug/al PCB solution dilute 1.0 ml of stock PCB solution to 100 ml with became in a volumetric flask.
 - 5.4.3 2.0 ug/al PCB solution dilute 1.0 al of 10 ug/al PCB solution to 5 al with herase in a volumetric flask.
 - 5.4.4 1.0 ug/ml PCB solution dilute 1.0 ml of 10 ug/ml PCB solution to 10 ml with heave in a volumetric flask.
 - 5.4.5 G.4 ug/ml PCB solution dilute 1.0 ml of 10 ng/ml PCB solution to 25 ml with heaves in a volumetric flask.
 - 5.4.6 4.0 ug/al PCB solution dilute 2 ml of 10 ug/al PCB solution to 5 ml with herane in a volumetric flask.
 - 5.4.7 0.04 ug/ml PCB solution dilute 0.1 ml of 10 ug/ml PCB solution to 25 ml with herene in a volumetric flask.

6. Quality Control

- 6.1 All reagents must be checked for contamination by GIC prior to use.
- 6.2 At least one blank is analyzed with each batch of cartridges or suchles.

7. Procedure

- 7.1 Preparation of Sampling Cartridges
 - NGTE: If a large number of cartridges are to be prepared, it is recommended that a particulate mask and disposable plastic gloves be used.
 - 7.1.1 All glassware must be free of PCB. Rinse all glassware with at least 3 separate portions of hexane. Bake glassware for two hours at 350 C. Collect a final hexane rinse and concentrate to 0.5 ml for analysis to verify that all interfering chromatogram peaks are removed.
 - 7.1.2 Out 1" to 1 1/4" from the finely drawn end of a Pasteur pipet using a triangular file. Fire polish the cut end.

RCB's in ATR (311-1)

- 7.1.3 Place a piece of hemane-rinsed glass wool into a Pasteur pipet. Push it down to the pointed end of the pipet. To the pipet, add about 0.4 grams of florisil. Place a small plug of hemane-rinsed glass wool at the top of the Florisil. Prepare several sampling cartridges in this manner, including a sufficient number to be used as blank cartridges.
- 7.1.4 After construction of the cartridge, attach the cartridge cutlet to a vacuum hose, keeping the cartridge vertical. Turn on the vacuum pump and tap the side of the cartridge lightly for about five seconds to settle and pack the Florisil. Push the glass wool plug down to the surface of the Florisil to insure close contact. This packing procedure eliminates the need to keep the cartridge vertical during sampling. Store the cartridges in an air-tight container.
- 7.1.5 Assemble cartridge into a sampling train as shown in Figure 1. One cartridge is usually sufficient to form an efficient sampling train. If a train containing two cartridges in series is desired, connections should be made with a short piece of Teflon tuking, butting the glass tubes, and using Tygon sleeving to prevent leaks. Measure and record the flow rate. Seal the ends of the cartridge train with aluminum foil caps or Caplugs to prevent contamination.
- 7.1.6 If at all possible, an inlet probe should not be used. If this cannot be avoided, a glass probe should be used. Terlon and Tygon have been found to adsorb PCBs and therefore are not recommended.
- 7.1.7 The sample should be collected for an appropriate period of time, usually 24 hours. The cartridge train should be rescaled with aluminum foil or Capluge before transporting back to the laboratory.

7.2 Analysis of Florisil Cartridges

- 7.2.1 After flow rate measurement, disassemble the sampling train. Label the cartridges.
- 7.2.2 Elute the PCBs into a graduated 5 or 10 ml test tube with a ground glass stopper using about 5 ml of nanograde hexane, adding the hexane to the inlet of the cartridge. Collect the eluxe in the graduated test tube.
- 7.2.3 Concentrate the extract by nitrogen (MP) stream evaporation to bring the PCB concentration of the extract into working range, usually 1 to 5 ml volume. Place extract into 2 ml capacity vials with injectable caps for use with automated gas chromatograph sampler.

- 7.2.4 Run samples and standards on the gas chromatograph under the following conditions:
 - 7.2.4.1 Column temperature 180 C isothermal for Arcclors 1016/1242,1221,1254 and 1260.
 - 7.2.4.2 Detector temperature electron capture detector, 275 C
 - 7.2.4.3 Inlet temperature 200 C
 - 7.2.4.4 Carrier gas nitrogen, 40 ml/min
 - 7.2.4.5 Carrier flow rate 60 cc/min
 - 7.2.4.6 Injection size 5 ul for manual injection; 1 ul for automated injection.
- 7.2.5 Blank certridge content should be subtracted from sample cartifidges to determine not arctior centent. Amounts greater than 0.033 up arctior in the blank centridge extracts may indicate contemination of the certaidge train either before of after sampling (i.e., glasseure contemination).
- 7.2.6 Peaks should be quantitated by area comparisons under specific Arcolor peaks of standards of known ascends.

8. Calculations

8.1 Air Whitme Sampled

where, V = volume of air sampled in cabic meters

R = air flew rate in liters per minute

T = time sampled in minutes

8.2 ug PCB Collected: Read from standard curve or by proportion to standard on day of run.

$$ug PCB = (A-B) C$$

where, A = ug/ml PCB in sample extract

B = ug/al PCB in blank extract (usually is zero)

C = ml of herene extract

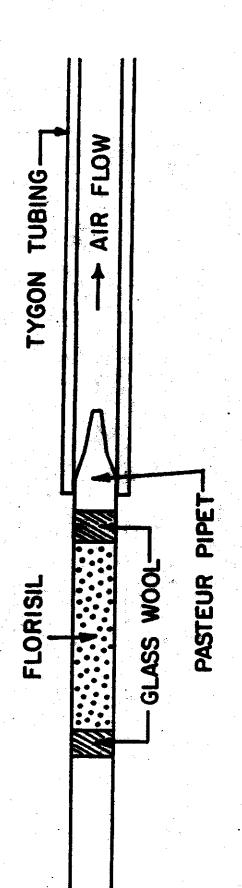
8.3 Report results as un/cubic mater in air sampled. Report as either total PCB or specific Arcelor.

9. References

- 9.1 Dell'Acqua, B. NYSCH, In-house experiment.
- 9.2 Giam, C.S., Chan, H.S., Neff, G.S. 1975. Rapid and Inexpensive Mathed for Detection of Polychlorinated Bighenyls and Phthalates in Air. Analytical Chemistry 47(13): 2319-20.
- 9.3 Trotter, W.J. 1973. Removing the Interference of DDT and its Amalogs in the Amalysis for Residues of Polychlorinsted Riphenyls. Journal of the Association of Official Analytical Chemists 58(3): 461-5.
- 9.4 Determination of Polychlorinated Riphenyls in Ashient Air, MYSUM, Division of Isboratories and Research, Environmental Health Institute, APC method 26., June 1979.
- 9.5 NIOSH Health and Hazard Evaluation Report No. 86-472; p. 18, 19; HEFAS6-479-1832; Commercial Office Buildings, Boston, MA.

HANDBOOKS (311-1)
July 1980; Corrected January 14, 1981; Corrected March 12, 1981

FLORISIL CARTRIDGE



APPENDIX D PCB WIPE SAMPLE LOCATIONS

SUNY New Paltz Wipe Sample Locations

Bliss Hall, Building #22

Basement

B4 - Floor Area

First Floor

103 - Floor Area

119 - Desk

Second Floor

205 - Desk

216 - Floor Area

Third Floor

326 - Floor Area

309 - Floor Area

Gage Hall, Building #21

Basement East

B8-Floor Area

First Floor East

105 - Desk

RD's Apartment - Floor Area

First Floor West

117 - Floor Area

133 Overflow - Desk

Second Floor East

218 - Desk

221 - Floor Area

Third Floor East

302 - Floor Area

Third Floor West

343 - Desk

328 - Desk

Scudder Hall, Building #20

First Floor

115 - Desk

116 - Desk

112 - Desk

Second Floor

205 - Floor Area

216 - Desk

230 - Desk

Third Floor

306 - Desk

309 - Floor Area

Capen Hall, Building #9

First Floor

110 - Floor Area

Second Floor

220 - Floor Area

Third Floor

323 - Desk

318 - Floor Area

APPENDIX E PCB AIR SAMPLE LOCATIONS

SUNY New Paltz PCB Air Sample Locations

Bliss Hall, Building #22

First Floor

112 Overflow

114

Second Floor

214

218

230

203

223

Gage Haii, Building #21

First Floor East

112

Second Floor East

223

203

Second Floor West

227

231

225

Third Floor East

302

305

Third Floor West

325

Scudder Hall, Building #20

Basement

B3

First Floor

107

Second Floor

209

222

Third Floor

320

322

328

Capen Hall, Building #9

First Floor

121

Second Floor

217

226

Third Floor

302

311

315

APPENDIX B WIPE AND AIR SAMPLE LOCATIONS

Table 2
SUNY New Paltz
PCB Air Sample Locations

	Sample Location	Field ID Number
Bliss Hall, Build	ng #22	
First Floor:	112 Overflow	A20
	114	A19
Second Floor:	214	A31A
	218	A22
	230	A33
	203	A30
	223	A21
Gage Hall, Build	ing #21	
First Floor East:	112	A30
Second Floor East:	223	A26
	203	A10
Second Floor West:	227	A27
	231	A29
	225	A28
Third Floor East:	302	A12
	305	A25
Third Floor West:	325	All
Scudder Hall, Bu	ilding #20	
Basement:	B3	A3
First Floor:	107	A13
Second Floor:	209	A14
	222	A15
Third Floor:	320	A18
	322	A17
	328	A16
Capen Hall, Buil	ding #9	
First Floor:	121	A4
Second Floor:	217	A6
	226	A5
Third Floor:	302	A7
	311	A8
	315	A9
Field Blank		Al
Field Blank		A2
Field Blank		A23
Outside Gage Hall		A24

	Sample Location	Field ID Number
Bliss Hall, Build	ing #22	
Basement:	B4 – Floor Area	W21
First Floor:	103 – Floor Area	W26
· · · · · · · · · · · · · · · · · · ·	119 Desk	W27
Second Floor:	205 – Desk	W24
•	216 - Floor Area	W25
Third Floor:	326 - Floor Area	W23
**	309 - Floor Area	W22
Gage Hall, Bulle	ling #21	
Basement East:	B8 – Floor Area	W19
First Floor East:	105 - Desk	W17
	RD's Apartment - Floor Area	W17
First Floor West:	117 - Floor Area	W16
	133 Overflow - Desk	W15
Second Floor East:	218 – Desk	W14
	221 - Floor Area	W13
Third Floor East	302 - Floor Area	W20
Third Floor West	343 - Desk	W12
	328 - Desk	W11
Scudder Hall, Br	silding #20	
First Floor:	115 – Desk	W10
•	116 - Desk	W9
	112 - Desk	W8
Second Floor:	205 – Floor Area	W7
	216 – Desk	W6
	230 – Desk	W5
Third Floor:	306 – Desk	W3
	309 - Floor Area	W4
Capen Hall, Buil	ding #9	
First Floor:	110 - Floor Area	W32
Second Floor:	220 - Floor Area	W31
Third Floor:	323 - Desk	W29
	318 – Floor Area	W28
Field Blank		W1
Field Blank		W2
Field Blank	/	W30