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STATUS REPORT

TEMPORAL WATER COLUMN MONITORING PROGRAM

HUDSON RIVER PROJECT

GENERAL ELECTRIC COMPANY

CORPORATE ENVIRONMENTAL PROGRAMS

FAIRFIELD, CONNECTICUT

October 2, 1991

O'BRIEN & GERE ENGINEERS, INC.
5000 BRITTONFIELD PARKWAY
SYRACUSE, NEW YORK 13221

GENERAL ELECTRIC COMPANY
HUDSON RIVER PROJECT
TEMPORAL WATER COLUMN MONITORING PROGRAM

SECTION 1 - INTRODUCTION

1.01 OBJECTIVES

The principal objective of the Temporal Water Column Monitoring Program is to provide high frequency and high resolution PCB monitoring data for the Upper Hudson River.

1.02 SCOPE OF WORK

The Temporal Water Column Monitoring Program includes the collection and analysis of water born PCB according to methods employed by the U.S. Geological Survey (USGS) during the 1970s and 1980s to provide consistency with the historical water column data base. Four of the eight monitoring stations employed during this study are used by the USGS to monitor water column PCB. Water column samples are being analyzed for PCB according to procedures historically employed by the USGS as well as congener specific methods. Conventional parameters are also being monitored as part of this program.

SECTION 2 - SAMPLING LOCATIONS AND PROCEDURES

2.01 SAMPLING LOCATIONS

Water samples are being obtained from eight stations on the river. Four of these stations are located on the bridges utilized as the historical USGS sampling stations in Fort Edward, Schuylerville, Stillwater, and Waterford. Additional stations include the abandoned bridge adjacent to Bakers Falls near Hudson Falls, New York, the western end of the Thompson Island Dam, and the Hoosic River and Batter Kill. Approximate station locations are illustrated on Figure 1.

Samples are collected from near the center of the channel off bridges, with the exception of the Thompson Island Dam, Hoosic River, and Batten Kill stations which are collected from shore

locations (the center of the channel at these stations are not accessible by land). Samples at the Thompson Island Dam station are collected from the west wing wall of the dam. Hoosic River and Batten Kill stations are located approximately 0.5 miles upstream of their confluence with the Hudson River.

2.02 SAMPLE COLLECTION FREQUENCY

The following sampling schedule is being followed for the program:

TIMEFRAME	SAMPLING FREQUENCY
April 1 to June 30, 1991	3 times per week
July 1, 1991 to February 28, 1992	1 time per week

2.03 SAMPLE COLLECTION PROCEDURES

Samples collected from the bridges are vertically stratified composites made up of discrete aliquots collected at 3-foot intervals throughout the water column. Samples are collected with a stainless steel Kemmerer bottle at each station. The Kemmerer bottle sampler is a 1.2 liter cylinder equipped with closeable stoppers at each end. The sampler is lowered to the desired depth in the water column in the open position. A messenger is sent down the suspending cable to close the sampler and a discrete aliquot is collected. Upon retrieval, the sampler is discharged into a stainless steel compositing container. Subsequent aliquots are placed in the container to form the vertically stratified composite.

Upon collection, the composite sample are poured from the stainless steel compositing container into appropriate containers, chilled to 4°C, and transported to the laboratory for analysis. Each sample is assigned a unique sample designation, identifying sample location, date, and time. Standard chain of custody procedures are being followed. The kemmerer bottle sampler is being thoroughly decontaminated between stations. Decontamination procedures are specified in the Quality Assurance/Quality Control section of this report.

At shore stations, water samples are being collected in dedicated, precleaned 1 gallon glass

containers which are submerged to the desired depth, allowed to fill, and retrieved.

2.04 ANALYTICAL TESTING

Water samples collected as part of the Temporal Water Column Monitoring Program are being analyzed for the following parameters:

- total dissolved solids,
- conductivity,
- alkalinity,
- total suspended solids,
- total organic carbon, and
- total PCB by USGS and "Green Bay" congener specific methods.

Analytical methods and protocols are briefly defined in the Quality Assurance/Quality Control section of this report. PCB analyses are being performed by Northeast Analytical, Inc. Other analyses are being performed by OBG Laboratories, Inc.

SECTION 3 - QUALITY ASSURANCE/QUALITY CONTROL

Essential components of the Quality Assurance/Quality Control (QA/QC) procedures being employed during the Temporal Water Column Monitoring Program are summarized below.

3.01 FIELD DOCUMENTATION

Water column sampling procedures are being verified in a daily log maintained by sampling personnel. Daily entries into the logbook include: date, weather conditions, time of sample collection, depths of aliquot collection, and other pertinent observations such as barge traffic, surface debris, and physical appearance of the water column.

3.02 FIELD QA/QC SAMPLES

In order to evaluate data quality, field QA/QC samples are being collected. The types and minimum frequencies of QA/QC samples are as follows: field duplicates (5%), laboratory duplicates (5%), matrix spikes (5%), field blanks (5%), method blanks (10%), external QC check samples (5%), and spiked blanks (5%).

3.03 DECONTAMINATION PROCEDURES

Sampling equipment, including the Kemmerer bottle sampler and the stainless steel compositing container, are decontaminated after the collection of each composite sample. To complete the decontamination procedure, the sampling equipment is rinsed with acetone, then hexane, then allowed to completely air dry, and finally rinsed with distilled water.

3.04 SAMPLE CUSTODY

Sample custody is being maintained through strict adherence to chain of custody procedures, as defined by the U.S. EPA guidance document: A Compendium of Superfund Field Operations Methods (U.S. EPA 600/2-80-018). These procedures include the maintenance of a field logbook in conjunction with a chain of custody form. The chain of custody form accompanies the samples from the field to the laboratory and include a sample description, date and time of collection, sample matrix and type, number and size of sample containers filled, and the type of analysis requested. Sample custody is being maintained by the receiving laboratory throughout the analytical procedure.

3.05 ANALYTICAL METHODS

The analytical methods being performed during this program comply with the requirements of the following methodologies.

- Congener specific PCB analytical methodologies adhere to procedures outlined in Northeast Analytical, Inc. Method NEA-608CAP , Rev. 2.0, 8/89 (Attachment 1). The method includes guidelines set forth in the document: Quality Assurance Plan, Green Bay Mass Balance Study, I.PCBs and Dieldrin, U.S. EPA Great Lakes National Program Office, prepared by Deborah L. Swackhamer, Quality Assurance Coordinator, Field and Analytical Methods Committees, University of Minnesota, December 11, 1987.

- Packed column total PCB data is being generated according to USGS methodologies. The USGS method consists of procedures consistent with EPA Method 608 with total PCB calculated by dividing the total area of the samples identified PCB peaks by the area of all peaks for an Aroclor standard. A standard consisting of two Aroclors is used for quantifying PCB in samples which appear to contain less than 60 percent of a single Aroclor.

- Other laboratory analyses, analytical QA/QC and data reporting requirements adhere to guidelines outlined in: Methods for Chemical Analysis of Water and Wastes, U.S. EPA -

600/4-79-020, Revised 1983. New York State Department of Environmental Conservation, Analytical Services Protocol, NYSDEC, September 1989.

Specific methodologies are summarized in Table 1.

3.06 DATA REPORTING

Analytical data packages are being provided for each analysis. Data report forms are securely bound and all pages are being sequentially numbered. The analytical data reports include the following information:

- case narrative,
- date of sampling,
- case file,
- description of samples,
- description of sample extraction and clean-up procedures,
- indication of analytical method,
- analytical results of all samples plus trip blank, field blank, and method blank, (including tentatively identified compounds, if applicable),
- analytical results of QA/QC sample analyses,
- summarized calibration data,
- detection limits for parameters analyzed,
- QA/QC data summaries (i.e. MS results and summaries),
- copy of completed chain-of-custody forms,
- notebook accountability record,
- appropriate raw instrument outputs (e.g. GC/MS spectral printouts), and
- example calculations for each analysis.

SECTION 4 - WATER COLUMN MONITORING RESULTS

4.01 GENERAL

Preliminary water column monitoring data which has been received from the laboratories and incorporated into the data management system are presented in attached tables (Tables 2, 3, and 4). In some instances, data for PCB have been received without the corresponding conventional data. Where this has occurred, the conventional fields of Table 2 contain a period (.).

The data appearing in the attached tables have not been validated and should be considered as preliminary only. Laboratory contamination problems appear to have effected PCB results on several days. These data have been omitted from this report. The laboratory contamination issue will be fully explored and documented during data validation.

4.02 ANALYTICAL TESTING RESULTS

Total PCB data generated by the USGS procedure along with conventional parameter testing results are contained in Table 2. Table 3 contains a summary of the congener specific testing results. PCB data appearing in Table 3 are presented as the weight percent of each homolog group and the total PCB concentration calculated from the sum of the individual congeners. Specific congener data are being filed in hard copy and electronic format. Table 4 contains the number of observations, range, mean, and standard deviations for PCB data generated by the USGS methodology.

TABLE 1
HUDSON RIVER PROJECT
TEMPORAL WATER COLUMN MONITORING PROGRAM

ANALYTICAL METHODS

PARAMETER	METHOD NUMBER AND REFERENCE
Total suspended solids (TSS)	160.2 (1)
Total Dissolved Solids (TDS)	160.1 (1)
Total Organic Carbon	415.1 (1)
Specific Conductivity	120.1 (1)
Alkalinity	310.1 (1)
Total PCBs	608 (2)
Congener Specific PCBs	NEA-608CAP (3)

(1) *Methods for Chemical Analysis of Water and Wastes*, U.S. EPA-600/4-79-020, Revised 1983.

(2) 40 CFR 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, Organochlorine Pesticides and PCBs.

(3) *Standard Operating Procedure - Laboratory Method NEA-608CAP*, Northeast Analytical, Inc., Revision 3, June 1990 (Appendix A).

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Some high blanks may invalidate dat

Table 2
Hudson River Project
Temporal Water Column Monitoring Program
USGS Total PCB and Conventional Parameter Results*
October 2, 1991

DATE COLLECTED	LOCATION	TOTAL PCB (ppt)	TSS (mg/l)	TOC (mg/l)	Alkalinity (mg/CaCO3/l)	Sp. Cond. (umho)	TDS (mg/1)
04/01/91	Bakers Falls Br.	<11	8	6	13	71	40
04/01/91	Rt.197 Br.	18	6	5	13	70	96
04/01/91	Thompson Island Dam	29	6	5	13	77	57
04/01/91	Rt.29 Br.	29	2	5	15	78	52
04/01/91	Stillwater Br.	37	3	4	29	110	61
04/01/91	Rt.4 Br.	25	4	5	30	120	75
04/03/91	Bakers Falls Br.	20	4	5	12	70	57
04/03/91	Rt.197 Br.	11	5	7	12	71	78
04/03/91	Thompson Island Dam	15	8	6	15	82	74
04/03/91	Rt.29 Br.	11	4	5	17	83	56
04/03/91	Stillwater Br.	13	4	4	28	100	70
04/03/91	Rt.4 Br.	19	5	4	33	120	81
04/05/91	Bakers Falls Br.	<11	6	9	13	75	69
04/05/91	Rt.197 Br.	17	4	7	13	75	61
04/05/91	Thompson Island Dam	31	7	10	17	81	64
04/05/91	Rt.29 Br.	27	5	9	21	88	69
04/05/91	Stillwater Br.	44	4	7	29	110	76
04/05/91	Rt.4 Br.	24	6	8	36	130	110
04/08/91	Bakers Falls Br.	<11	1	5	13	62	48
04/08/91	Rt.197 Br.	26	1	5	12	63	32
04/08/91	Thompson Island Dam	32	1	5	15	70	48
04/08/91	Rt.29 Br.	43	2	6	16	73	51
04/08/91	Stillwater Br.	42	1	.	23	88	59
04/08/91	Rt.4 Br.	32	5	.	30	110	79
04/10/91	Bakers Falls Br.	11	4	.	10	46	38
04/10/91	Rt.197 Br.	31	1	.	9	47	41
04/10/91	Thompson Island Dam	37	1	.	12	57	63
04/10/91	Rt.29 Br.	70	5	.	13	59	51
04/10/91	Stillwater Br.	37	11	.	27	74	58
04/10/91	Rt.4 Br.	50	18	.	19	97	58
04/12/91	Bakers Falls Br.	14	6	.	10	49	35
04/12/91	Rt.197 Br.	22	5	.	11	52	37
04/12/91	Thompson Island Dam	27	6	.	11	56	40
04/12/91	Rt.29 Br.	99	6	.	11	55	50
04/12/91	Stillwater Br.	40	10	.	11	71	45
04/12/91	Rt.4 Br.	21	6	.	11	89	59

* Period (.) denotes data recieved but not incorporated into data management system

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Table 2
Hudson River Project
Temporal Water Column Monitoring Program
USGS Total PCB and Conventional Parameter Results*
October 2, 1991

DATE COLLECTED	LOCATION	TOTAL PCB (ppt)	TSS (mg/l)	TOC (mg/l)	Alkalinity (mg/CaCO3/l)	Sp. Cond. (umho)	TDS (mg/1)
04/15/91	Bakers Falls Br.	<11	1	.	11	62	56
04/15/91	Rt.197 Br.	71	3	.	11	63	52
04/15/91	Thompson Island Dam	20	4	.	15	71	47
04/15/91	Rt.29 Br.	25	2	.	13	71	79
04/15/91	Stillwater Br.	20	4	.	24	90	57
04/15/91	Rt.4 Br.	17	3	.	29	110	66
04/17/91	Bakers Falls Br.	<11	2	.	13	71	56
04/17/91	Rt.197 Br.	<11	4	.	13	72	58
04/17/91	Thompson Island Dam	24	4	.	17	79	74
04/17/91	Rt.29 Br.	17	3	.	17	80	74
04/17/91	Stillwater Br.	21	1	.	29	110	76
04/17/91	Rt.4 Br.	<11	2	.	33	120	85
04/19/91	Bakers Falls Br.	<11	3	.	13	75	94
04/19/91	Rt.197 Br.	35	2	.	15	76	47
04/19/91	Thompson Island Dam	28	1	.	17	87	56
04/19/91	Rt.29 Br.	37	3	.	22	95	56
04/19/91	Stillwater Br.	37	2	.	30	110	.
04/19/91	Rt.4 Br.	<11	2	.	38	140	.
04/22/91	Bakers Falls Br.	<11	1	.	14	66	26
04/22/91	Rt.197 Br.	12	1	.	14	67	27
04/22/91	Thompson Island Dam	18	11	.	19	87	33
04/22/91	Rt.29 Br.	<11	17	.	25	100	43
04/22/91	Stillwater Br.	<11	37	.	38	130	54
04/22/91	Rt.4 Br.	20	36	.	44	150	64
04/24/91	Bakers Falls Br.	15	3	8	11	58	71
04/24/91	Rt.197 Br.	24	7	7	12	57	64
04/24/91	Thompson Island Dam	19	7	7	14	66	65
04/24/91	Rt.29 Br.	11	9	6	17	69	43
04/24/91	Stillwater Br.	18	9	7	23	88	60
04/24/91	Rt.4 Br.	20	9	6	30	110	79
04/26/91	Bakers Falls Br.	<11	3	12	10	6	42
04/26/91	Rt.197 Br.	<11	4	8	12	60	47
04/26/91	Thompson Island Dam	13	6	12	14	69	77
04/26/91	Rt.29 Br.	12	4	9	18	78	55
04/26/91	Stillwater Br.	<11	3	6	26	92	71
04/26/91	Rt.4 Br.	<11	4	8	30	110	89

* Period (.) denotes data recieved but not incorporated into data management system

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Table 2
Hudson River Project
Temporal Water Column Monitoring Program
USGS Total PCB and Conventional Parameter Results*
October 2, 1991

DATE COLLECTED	LOCATION	TOTAL PCB (ppt)	TSS (mg/l)	TOC (mg/l)	Alkalinity (mg/CaCO3/l)	Sp. Cond. (umho)	TDS (mg/1)
04/29/91	Bakers Falls Br.	<11	3	6	13	73	44
04/29/91	Rt.197 Br.	<11	6	7	13	75	58
04/29/91	Thompson Island Dam	14	2	6	17	78	60
04/29/91	Rt.29 Br.	12	5	6	21	86	85
04/29/91	Stillwater Br.	12	1	5	29	100	11
04/29/91	Rt.4 Br.	16	3	5	36	130	94
05/01/91	Bakers Falls Br.	<11	3	.	13	69	82
05/01/91	Rt.197 Br.	<11	3	.	14	69	61
05/01/91	Thompson Island Dam	<11	3	.	19	80	48
05/01/91	Rt.29 Br.	21	3	.	28	94	110
05/01/91	Stillwater Br.	33	9	.	29	110	82
05/01/91	Rt.4 Br.	15	5	.	41	140	90
05/03/91	Bakers Falls Br.	<11	.	.	.	.	.
05/03/91	Rt.197 Br.	<11	.	.	.	.	.
05/03/91	Thompson Island Dam	34	.	.	.	.	.
05/03/91	Rt.29 Br.	22	.	.	.	.	.
05/03/91	Stillwater Br.	33	.	.	.	.	.
05/03/91	Rt.4 Br.	18	.	.	.	.	.
05/08/91	Bakers Falls Br.	<11	.	.	.	.	.
05/08/91	Rt.197 Br.	27	.	.	.	.	.
05/08/91	Thompson Island Dam	23	.	.	.	.	.
05/08/91	Rt.29 Br.	18	.	.	.	.	.
05/08/91	Stillwater Br.	18	.	.	.	.	.
05/08/91	Rt.4 Br.	14	.	.	.	.	.
05/10/91	Bakers Falls Br.	<11	.	.	.	.	.
05/10/91	Rt.197 Br.	<11	.	.	.	.	.
05/10/91	Thompson Island Dam	<11	.	.	.	.	.
05/10/91	Rt.29 Br.	<11	.	.	.	.	.
05/10/91	Stillwater Br.	<11	.	.	.	.	.
05/10/91	Rt.4 Br.	<11	.	.	.	.	.
05/13/91	Bakers Falls Br.	<11	.	.	.	.	.
05/13/91	Rt.197 Br.	<11	.	.	.	.	.
05/13/91	Thompson Island Dam	24	.	.	.	.	.
05/13/91	Rt.29 Br.	26	.	.	.	.	.
05/13/91	Stillwater Br.	24	.	.	.	.	.
05/13/91	Rt.4 Br.	18	.	.	.	.	.

* Period (.) denotes data recieved but not incorporated into data management system

Table 2
Hudson River Project
Temporal Water Column Monitoring Program
USGS Total PCB and Conventional Parameter Results*
October 2, 1991

DATE COLLECTED	LOCATION	TOTAL PCB (ppt)	TSS (mg/l)	TOC (mg/l)	Alkalinity (mg/CaCO3/l)	Sp. Cond. (umho)	TDS (mg/1)
05/15/91	Bakers Falls Br.	<11	.	.	.	.	.
05/15/91	Rt.197 Br.	15	.	.	.	.	.
05/15/91	Thompson Island Dam	18	.	.	.	.	.
05/15/91	Rt.29 Br.	24	.	.	.	.	.
05/15/91	Stillwater Br.	21	.	.	.	.	.
05/15/91	Rt.4 Br.	19	.	.	.	.	.
05/17/91	Bakers Falls Br.	<11	.	.	.	.	.
05/17/91	Rt.197 Br.	<11	.	.	.	.	.
05/17/91	Thompson Island Dam	23	.	.	.	.	.
05/17/91	Rt.29 Br.	21	.	.	.	.	.
05/17/91	Stillwater Br.	21	.	.	.	.	.
05/17/91	Rt.4 Br.	20	.	.	.	.	.
05/20/91	Bakers Falls Br.	<11	.	.	.	.	.
05/20/91	Rt.197 Br.	17	.	.	.	.	.
05/20/91	Thompson Island Dam	38	.	.	.	.	.
05/20/91	Rt.29 Br.	32	.	.	.	.	.
05/20/91	Stillwater Br.	36	.	.	.	.	.
05/20/91	Rt.4 Br.	30	.	.	.	.	.
05/22/91	Bakers Falls Br.	<11	.	.	.	.	.
05/22/91	Rt.197 Br.	21	.	.	.	.	.
05/22/91	Thompon Island Dam	29	.	.	.	.	.
05/22/91	Rt.29 Br.	32	.	.	.	.	.
05/22/91	Stillwater Br.	35	.	.	.	.	.
05/22/91	Rt.4 Br.	26	.	.	.	.	.
05/24/91	Bakers Falls Br.	<11	4	8	17	98	54
05/24/91	Rt.197 Br.	<11	4	7	19	110	75
05/24/91	Thompson Island Dam	51	2	8	19	100	72
05/24/91	Rt.29 Br.	<11	3	7	26	110	74
05/24/91	Stillwater Br.	<11	2	10	30	110	75
05/24/91	Rt.4 Br.	<11	3	5	40	160	94
05/29/91	Bakers Falls Br.	<11	.	.	.	.	.
05/29/91	Rt.197 Br.	<11	.	.	.	.	.
05/29/91	Thompson Island Dam	<11	.	.	.	.	.
05/29/91	Rt.29 Br.	<11	.	.	.	.	.
05/29/91	Stillwater Br.	<11	.	.	.	.	.
05/29/91	Rt.4 Br.	<11	.	.	.	.	.

* Period (.) denotes data recieved but not incorporated into data management system

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Table 2
Hudson River Project
Temporal Water Column Monitoring Program
USGS Total PCB and Conventional Parameter Results*
October 2, 1991

DATE COLLECTED	LOCATION	TOTAL PCB (ppt)	TSS (mg/l)	TOC (mg/l)	Alkalinity (mg/CaCO3/l)	Sp. Cond. (umho)	TDS (mg/1)
06/17/91	Bakers Falls Br.	<11	1	8	17	91	68
06/17/91	Rt.197 Br.	27	1	7	17	90	70
06/17/91	Thompson Island Dam	35	3	6	21	100	77
06/17/91	Rt.29 Br.	15	1	7	33	130	100
06/17/91	Stillwater Br.	<11	3	6	31	130	82
06/17/91	Rt.4 Br.	16	5	5	56	190	110
06/19/91	Bakers Falls Br.	<11	1	6	18	98	82
06/19/91	Rt.197 Br.	<11	1	7	18	98	75
06/19/91	Thompson Island Dam	13	2	6	20	98	110
06/19/91	Rt.29 Br.	13	1	5	24	110	80
06/19/91	Stillwater Br.	<11	4	6	33	130	66
06/19/91	Rt.4 Br.	<11	6	7	42	160	85
06/21/91	Bakers Falls Br.	<11	3	7	20	98	75
06/21/91	Rt.197 Br.	18	5	8	20	98	73
06/21/91	Thompson Island Dam	<11	4	7	20	92	71
06/21/91	Rt.29 Br.	<11	4	8	20	96	75
06/21/91	Stillwater Br.	46	4	8	26	110	83
06/21/91	Rt.4 Br.	40	8	7	36	150	100
06/24/91	Bakers Falls Br.	<11	5	6	20	100	73
06/24/91	Rt.197 Br.	<11	3	6	19	99	73
06/24/91	Thompson Island Dam	<11	3	6	23	110	78
06/24/91	Rt.29 Br.	41	4	6	24	110	74
06/24/91	Stillwater Br.	21	5	5	27	110	78
06/24/91	Rt.4 Br.	<11	6	6	37	180	110

* Period (.) denotes data recieved but not incorporated into data management system

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Table 3
Hudson River Project
Temporal Water Column Monitoring Program
Summary of Congener Specific PCB Results
October 2, 1991

Sum of Congeners

DATE COLLECTED	LOCATION	TOTAL PCB (ppt)	HOMOLOG DISTRIBUTION (percent weight)									
			MONO	DI	TRI	TETRA	PENTA	HEXA	HEPTA	OCTA	NONA	DECA
04/05/91	Bakers Falls Bridge	<11	-	-	-	-	-	-	-	-	-	-
04/05/91	Route 197 Bridge	16	0.0	11.6	35.9	27.5	12.3	12.7	0.0	0.0	0.0	0.0
04/05/91	Thompson Island Dam	43	13.0	13.4	29.9	27.0	9.0	7.2	0.5	0.0	0.0	0.0
04/05/91	Route 29 Bridge	33	0.0	14.2	38.4	26.0	10.0	9.2	2.1	0.0	0.0	0.0
04/05/91	Stillwater Bridge	67	6.4	9.0	27.4	32.1	7.8	6.3	7.7	3.5	0.0	0.0
04/05/91	Route 4 Bridge	36	17.3	14.2	33.0	17.6	7.3	6.1	1.7	2.8	0.0	0.0
04/12/91	Bakers Falls Bridge	14	0.0	11.0	27.8	18.3	16.6	20.3	6.1	0.0	0.0	0.0
04/12/91	Route 197 Bridge	24	0.0	11.2	28.0	22.3	17.3	16.9	4.4	0.0	0.0	0.0
04/12/91	Thompson Island Dam	<11	-	-	-	-	-	-	-	-	-	-
04/12/91	Route 29 Bridge	96	0.0	17.4	38.6	30.2	7.2	5.1	1.5	0.0	0.0	0.0
04/12/91	Stillwater Bridge	<11	-	-	-	-	-	-	-	-	-	-
04/12/91	Route 4 Bridge	24	0.0	9.1	35.2	28.6	13.3	12.2	1.8	0.0	0.0	0.0
04/19/91	Bakers Falls Bridge	<11	-	-	-	-	-	-	-	-	-	-
04/19/91	Route 197 Bridge	48	0.0	11.1	32.6	33.1	11.3	8.8	3.1	0.0	0.0	0.0
04/19/91	Thompson Island Dam	<11	-	-	-	-	-	-	-	-	-	-
04/19/91	Route 29 Bridge	38	0.0	0.3	22.2	43.2	17.8	11.5	5.0	0.0	0.0	0.0
04/19/91	Stillwater Bridge	<11	-	-	-	-	-	-	-	-	-	-
04/19/91	Route 4 Bridge	15	0.0	15.0	27.7	23.2	18.7	13.6	1.8	0.0	0.0	0.0
04/26/91	Bakers Falls Bridge	<11	-	-	-	-	-	-	-	-	-	-
04/26/91	Route 197 Bridge	<11	-	-	-	-	-	-	-	-	-	-
04/26/91	Thompson Island Dam	<11	-	-	-	-	-	-	-	-	-	-
04/26/91	Route 29 Bridge	12	0.0	14.3	33.8	26.0	14.0	10.1	1.9	0.0	0.0	0.0
04/26/91	Stillwater Bridge	<11	-	-	-	-	-	-	-	-	-	-
04/26/91	Route 4 Bridge	<11	-	-	-	-	-	-	-	-	-	-

Table 3
Hudson River Project
Temporal Water Column Monitoring Program
Summary of Congener Specific PCB Results
October 2, 1991

DATE COLLECTED	LOCATION	TOTAL PCB (ppt)	HOMOLOG DISTRIBUTION (percent weight)									
			MONO	DI	TRI	TETRA	PENTA	HEXA	HEPTA	OCTA	NONA	DECA
05/03/91	Bakers Falls Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/03/91	Route 197 Bridge	12	0.0	23.4	34.6	20.1	11.0	11.0	0.0	0.0	0.0	0.0
05/03/91	Thompson Island Dam	75	28	19.6	26.1	13.5	5.1	3.1	4.4	0.0	0.0	0.0
05/03/91	Route 29 Bridge	37	27.6	21.2	29.5	14.5	3.9	3.3	0.0	0.0	0.0	0.0
05/03/91	Stillwater Bridge	49	9	12.6	26.0	22.6	11.5	10.8	7.6	0.0	0.0	0.0
05/03/91	Route 4 Bridge	30	11.6	22.3	35.2	16.4	6.7	7.7	0.0	0.0	0.0	0.0
05/10/91	Bakers Falls Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/10/91	Route 197 Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/10/91	Thompson Island Dam	<11	-	-	-	-	-	-	-	-	-	-
05/10/91	Route 29 Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/10/91	Stillwater Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/10/91	Route 4 Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/17/91	Bakers Falls Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/17/91	Route 197 Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/17/91	Thompson Island Dam	49	32	22.9	26.8	13.5	3.3	1.8	0.0	0.0	0.0	0.0
05/17/91	Route 29 Bridge	40	32.4	21.4	27.7	14.0	3.0	1.5	0.0	0.0	0.0	0.0
05/17/91	Stillwater Bridge	40	21	25.3	31.8	15.2	4.2	2.6	0.0	0.0	0.0	0.0
05/17/91	Route 4 Bridge	32	11.3	28.3	37.2	17.3	4.4	1.5	0.0	0.0	0.0	0.0
05/24/91	Bakers Falls Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/24/91	Route 197 Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/24/91	Thompson Island Dam	118	36	19.0	28.6	13.1	3.1	0.6	0.0	0.0	0.0	0.0
05/24/91	Route 29 Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/24/91	Stillwater Bridge	<11	-	-	-	-	-	-	-	-	-	-
05/24/91	Route 4 Bridge	<11	-	-	-	-	-	-	-	-	-	-

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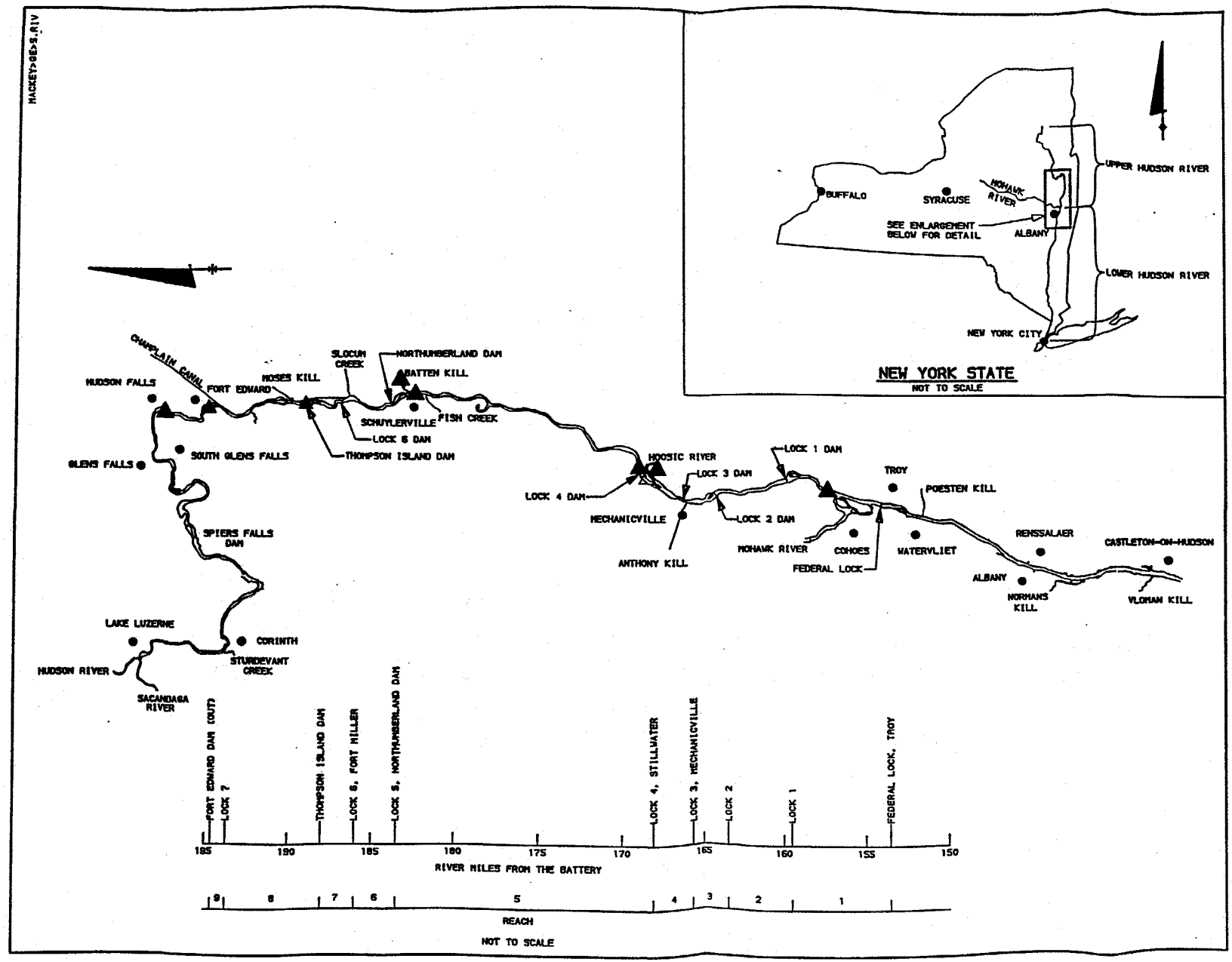
Table 4
Hudson River Project
Temporal Water Column Monitoring Program
Summary of Total PCB Results*
October 2, 1991

LOCATION	NUMBER OF OBSERVATIONS	MINIMUM** (ppt)	MAXIMUM (ppt)	MEAN (ppt)	STANDARD DEVIATION
BAKERS FALLS BRIDGE	28	1	20	7	4.4
ROUTE 197 BRIDGE	28	3	42	16	10.0
THOMPSON ISLAND DAM	28	7	51	24	10.9
ROUTE 29 BRIDGE	28	4	99	25	20.4
STILLWATER BRIDGE	28	3	46	23	13.6
ROUTE 4 BRIDGE	28	4	50	18	10.6

* Generated by USGS methodologies
** Below method detection limit of 11 ppt

FIGURE 1



▲ Temporal Water Column Monitoring Program Sampling Locations

GENERAL ELECTRIC
HUDSON RIVER PROJECT

HUDSON RIVER
RIVER LOCATION MAP

5 0 5
1" = 5 MILES

MAY 1991

O'BRIEN & GERE
ENGINEERS, INC.

312407

ATTACHMENT 1

STANDARD OPERATING PROCEDURE

LABORATORY METHOD NEA-608CAP

NORTHEAST ANALYTICAL, INC.
301 NOTT STREET
SCHENECTADY, NEW YORK 12305
(518) 346-4592

STANDARD OPERATING PROCEDURE

LABORATORY METHOD NEA-608CAP

REVISION 3 (6/90)

Lab Method NEA-608CAP

Congener-Specific Polychlorinated Biphenyl (PCB) Analysis

Method for Congener-Specific Polychlorinated Biphenyl (PCB) Quantification and Identification by Capillary Column/Gas Chromatography with Electron Capture Detection

1.0 Scope

1.1 This method is applicable in the determination and quantification of Polychlorinated Biphenyls (PCB) in sediments and soils. This method is a congener-specific determination, employing a high resolution fused-silica capillary chromatographic column. The method includes guidelines set forth in the document: "Quality Assurance Plan, Green Bay Mass Balance Study, I. PCBs and Dieldrin, US EPA Great Lakes National Program Office", prepared by Deborah L. Swackhamer, Quality Assurance Coordinator, Field and Analytical Methods Committees, University of Minnesota, December 11, 1987. This document outlines quality assurance and quality control procedures to be followed by laboratories participating in the Green Bay Mass Balance Study. Where applicable, Northeast Analytical, Inc., will incorporate and utilize this information in quality control of data generated. Instrumental analysis and conditions (Mullin, M.D., 1985, PCB Workshop, US EPA Large Lakes Research Station, Grosse Ile, MI, June.) cited in the Green Bay Mass Balance Study document will be refined to be applicable to an in-house data management software package.

1.2 This method will be applied specifically to the determination of PCBs by congener, in sediments. The results of these analyses will be used in studies related to biodegradation of PCBs. Highly sensitive techniques will generally not be required, since samples will contain appreciable quantities

of PCBs. Capillary column methods will be used to effectively separate up to 117 or more peaks representing 187 PCB congeners.

2.0 Summary of Method

2.1 This method provides detailed instructions for gas chromatographic conditions, sample extraction, and sample clean-up techniques for analysis of PCBs by capillary gas chromatography.

2.2 This method utilizes a mixed Aroclor standard (1232/1248/1262 in the ratio of 25:18:18) for calibration. Method detection limit and practical quantitation limit will be established experimentally using the procedure in USEPA 40 CFR, Part 136, Appx. B.

2.3 In general, samples are first extracted with a pesticide-grade solvent. The extracts are further processed by concentrating or diluting, depending on the concentration of PCB, and carried through a series of clean-up techniques. The sample is then analyzed by direct liquid injection onto the gas chromatographic column and detected by an electron capture detector. This method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

2.4 A key component of this method is the importance placed on the chromatographic separation that must be achieved for this congener specific technique. A total of 117 chromatographic peaks are detected, containing 187 PCB congeners in various ratios. This allows an almost complete profile of environmentally occurring PCBs.

2.5 Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize himself with the hazards of handling the compounds used for standards and samples themselves.

3.0 Interference

3.1 One of the major sources of interference in the analysis of PCBs is that organochlorine pesticides are coextracted from the samples. A few of these ECD responding pesticides can be separated cleanly from the PCB profile by the resolving characteristics of the capillary column. Several of the commonly found pesticides and degradation products (DDT, DDE, DDD) overlap the PCB profile envelope and co-elute with several of the minor PCB congeners found in environmental samples. The analyst must be careful in chromatographic pattern review and flag peaks that are suspected of being contaminated so that they are not included in total PCB values generated.

3.2 There are several clean-up protocols available that can be used to fractionate PCBs from organochlorine pesticides found in environmental samples. These techniques and there effectiveness will be described in the

sample preparation and clean-up section of this manual. Other separation and clean-up techniques will be included in the section describing procedures to handle samples that contain oil and grease, hydrocarbons, and elemental sulfur.

3.3 Laboratory contamination can occur by introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters give response on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification.

4.0 Sample Archiving

4.1 Extracts and sediment samples will be retained after analysis. The solvent extracts will be stored in a freezer, while the dry sediment samples can be stored at room temperature, protected from the light.

5.0 Equipment and Apparatus

5.1 Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Northeast Analytical, Inc. will use a Varian Model 3400 gas chromatograph, equipped with capillary on-column injection (Septum Programmable Injector), temperature programmable oven, Model 8000 automatic sampler, and fast time constant electron capture detector. A data system (Dynamic Solutions, Maxima Workstation) for chromatographic operations and integration of detector signal is interfaced to the gas chromatograph.

5.1.1 Column: The gas chromatograph column to be used for analysis will be a DB-1 (J&W Company), bonded polydimethylsilicone, 30 meter fused silica capillary column with an internal diameter of 0.25 mm and phase coating thickness of 0.25 microns. This column is capable of resolving 117 chromatographic peaks from the full spectrum of all PCB congeners that could be expected in an environmental sample. Refer to Appendix A and Appendix B for a complete description of PCB congeners identified in each GC chromatographic peak and achievable analytical separations.

5.2 Chromatograph Data System: A data system for measuring peak height and peak area. A Maxima 820 workstation (Dynamic Solutions), PC-based data system, will be employed to record detector response and digitally store the chromatographic information on the computer hard disk. This system will allow for chromatographic review of data from the gas chromatograph, electronic peak integration for precise calculations, data base structuring of the analytical information, and archival capabilities.

5.3 Soxhlet Extractor: Complete system for use in extracting PCBs from soils, and sediments. The soxhlet extractor consists of a water cooled condensor, a 250 mL, or 500 mL round bottom flask, soxhlet repetitive flushing unit, appropriate heating mantle and controller.

5.4 Kuderna-Danish (K-D) apparatus: Complete system to evaporate extraction solvent and concentrate sample extract. The K-D apparatus consists of a concentrator tube (10 mL), evaporation flask (500 mL), and 3-ball snyder column.

5.5 Boiling Chips: Solvent extracted, silicon carbide or equivalent.

5.6 Volumetric Flasks: 10, 25, 50, and 100 mL, ground-glass stopper. For standards and sample dilutions.

5.7 Microsyringe: 10, 25, 100, 250, 500 uL for standard preparation.

5.8 Vials: Glass, 10, and 20 mL capacity for sample extracts.

5.9 Bottles: Glass, 50 and 100 mL capacity for sample storage.

5.10 Sample Concentrator: An evaporator unit that utilizes a stream of purified nitrogen gas to gently evaporate solvent from samples.

6.0 Reagents

6.1 Solvents: Pesticide grade or nano-grade quality. Hexane, acetone, toluene, methylene chloride, methanol, diethyl ether, dimethylformamide, and pentane.

6.2 Magnesium Sulfate: Anhydrous, suitable for pesticide analysis.

6.3 Sodium sulfate: Anhydrous, suitable for pesticide analysis.

6.4 Sulfuric acid: Concentrated, ACS reagent grade.

6.5 Mercury: Triple distilled.

6.6 Magnesium silicate (Florisil): 60-100 mesh, activated at 650 degrees C., reagent grade suitable for pesticide analysis.

6.7 Glass Wool: Silane treated, solvent washed to remove impurities.

6.8 Silica Gel: 100-200 mesh, grade 923, Aldrich, deactivated to 7.5% with water.

6.9 Potassium Hydroxide: Pellets, reagent grade.

6.10 Octachloronaphthalene: Obtained from Ultra Scientific (Hope, RI), with a purity greater than 95%.

6.11 Polychlorinated biphenyls: Neet commercial material for standard preparation. These materials are multi-component mixtures of PCB congeners and are the actual materials that were used in products such as transformers and capacitors. Monsanto was the largest producer of PCB formulations and sold them under the tradename Aroclor. These standards should be compared to EPA PCB reference materials to verify commercial materials, to be used as standards, have the same pattern and congener distribution.

6.12 Stock Standard Solutions:

6.12.1 Stock standards are prepared from individual Aroclor formulations by weighing approximately 0.025 g to the nearest 0.1 mg, and dissolving and diluting to volume in a 25 mL volumetric flask. This will give a stock concentration of 1,000 ppm.

6.12.2 The stock standard is transferred into screw-cap vials and stored in a freezer, protected from light. Stock standards should be checked at frequent intervals for signs of evaporation, especially just prior to preparing calibration standards.

6.12.3 Stock PCB standards must be replaced after one year, or sooner if comparison with EPA certified check standards indicate a problem.

6.12.4 Stock standards for the following are prepared by the above procedure:

Aroclor 1232
Aroclor 1248
Aroclor 1262

6.13 Calibration Standard: The calibration standard is prepared by combining Aroclor 1232, Aroclor 1248, and Aroclor 1262 in a 25:18:18 ratio with a final mixture concentration of 2.5 ug/mL, 1.8 ug/mL, and 1.8 ug/mL respectively (total = 6.1 ug/mL). The final concentration of the mixed standard may vary to accommodate instrument sensitivity or more closely represent sample concentrations, but the same ratio values must be maintained. These ratios are strictly maintained so that the percent composition data remains applicable, since it was developed for use under these fixed mixture parameters.

6.13.1 Using a 500 uL microsyringe, accurately add 0.25 mL of stock Aroclor 1232 standard (1,000 ppm) to a 100 mL volumetric flask. Add 0.18 mL of stock Aroclor 1248 and Aroclor 1262 to the same 100 mL of stock volumetric flask. Add the internal standard to the 100 mL volumetric flask as outlined in Section 6.14, and make volume to the 100 mL mark with hexane.

6.13.2 Store calibration standard in the refrigerator at 4 degrees C. in a tightly capped bottle. Calibration standards must be replaced

after six months, or sooner, if comparison with EPA check standards indicate a problem.

6.13.3 Since the instrument calibration is based on a single point calibration standard, concentration of PCBs in the sample extracts mustbe within a factor of five of the calibration standard value. Sample extracts that fall outside this range should be diluted or concentrated to be within the accepted concentration range. To facilitate in meeting the established concentration range for samples, sample extracts are to be screened by packed column gas chromatography with electron capture detection to determine their approximate concentrations. Sample extract solution concentrations for this screening procedure will be calculated by using a mixed Aroclor standard (Aroclor 1242 and 1260) with instrument calibration based on the peak weight percent method of Webb and McCall. This concentration data is necessary so that the extract concentration can be adjusted to match (within a factor of five) the concentration of the three Aroclor calibration mixture. Besides assessing the PCB extract concentration, the packed column screen will supply information on possible contamination and interfering co-extractables which would indicate further sample clean-up is necessary.

6.14 Internal Standard: The internal standard used for capillary gas chromatography of PCBs will be octachloronaphthalene (OCN). Weigh, to the nearest 0.1 mg, solvent, quantitatively transfer the OCN using six successive 2 mL washings to a 50 mL volumetric flask. Be sure to rinse the 10 mL vial walls carefully so that all OCN is completely transferred to the 50 mL volumetric flask. Make the solution to volume using toluene and mix the internal standard solution by shaking the flask several times. This will give a concentration of OCN of 200 ppm. Calculate the exact concentration of OCN based on the amount weighed. Carefully transfer the internal standard solution to 10 mL vials, tightly cap, and store in a freezer.

6.14.1 The OCN internal standard is added to all calibration standards, performance check standards, blanks, samples, and QC samples at an amount to give the same concentration as the major PCBs found in standards and samples. In most cases this will be achieved by spiking 9 uL of OCN internal standard solution to 10 mL of standard or sample extract to give an concentration of 0.1818 ppm.

6.14.2 The internal standard will be added to calibration standards, sample extracts, blanks, and QC samples just prior to gas chromatographic analysis. Thus, the internal standard is used as a quantification spiking standard and will eliminate sample injection and instrument variations, but will not correct for analytical losses during sample preparation.

6.15 Performance Check Standard: A performance check standard is prepared from quality assurance/quality control standards obtained from the USEPA Environmental Monitoring and Support Laboratory (EMSL), Cincinnati, Ohio. The performance standard is a mixed Aroclor standard of Aroclor 1232, 1248, and 1262 in the fixed ratio used to prepare the calibration standard

and must be strictly adhered to. The performance standard is made to a concentration at the lower end of the range that sample PCB extract concentrations must fall into (a factor of five of the calibration standard).

6.15.1 The performance standard is composed of: 0.50 ug/mL Aroclor 1232, 0.36 ug/mL Aroclor 1248, and 0.36 ug/mL Aroclor 1262, a total of 1.22 ug/mL PCB. This mixture is prepared from a concentration of 5,000 ug/mL in isooctane. The USEPA standards are diluted to 50 ug/mL by pipetting 0.5 mL of standard into a 50 mL volumetric flask and diluting to volume with hexane.

6.15.2 Using a 500 uL microsyringe transfer 500 uL of 50 ug/mL Aroclor 1232, 360 uL of 50 ug/mL Aroclor 1248, and 360 uL of 50 ug/mL Aroclor 1262 to a 50 mL volumetric flask. Using a 100 uL microsyringe, add 45 uL of OCN internal standard (final concentration of 0.1818 ug/mL). Dilute to volume with hexane and mix well by shaking and inverting flask several times. The prepared performance check solution will contain a total of 1.22 ug/mL PCB (0.5 ug/mL Aroclor 1232, 0.36 ug/mL Aroclor 1248, and 0.36 ug/mL Aroclor 1262).

6.15.3 Transfer the performance check standard to 10 mL vials, cap tightly, and store in a freezer. A new performance check standard must be prepared every three months.

6.16 Matrix Spike Standard: The matrix spike standard is prepared in the same manner as the calibration standard outlined in Section 6.13. The only change is that the internal standard is not added to the matrix spike standard. The matrix spike standard is used in quality control to determine percent recoveries of the analytical sample procedures. The matrix spike standard is added at a concentration of the same magnitude as the concentrations exhibited by the samples.

6.16.1 Using the Aroclor stock standards, prepare a stock matrix spike standard as follows. Accurately pipette 12.5 mL of 1,000 ug/mL Aroclor 1232, 9.0 mL of 1,000 ug/mL Aroclor 1248, and 9.0 mL of 1,000 ug/mL Aroclor 1262 into a 50 mL volumetric flask. Dilute to 50 mL with hexane, and mix by inverting the flask several times. The matrix spike standard will contain a total of 610.0 ug/mL PCB (250 ug/mL Aroclor 1232, 180 ug/mL Aroclor 1248, and 180 ug/mL Aroclor 1262).

6.16.2 Transfer the matrix spike standard to 10 mL vials, cap tightly, and store in a freezer. A new matrix spike standard must be prepared every six months, or sooner, if comparison with calibration standards indicate a problem.

7.0 Procedure

7.1 Calibration:

7.1.1 Gas Chromatographic Operation Parameters: Establish the gas chromatographic operation parameters as follows:

GC Column: DB-1 (J&W, bonded polydimethylsilicone), 30 meters, 0.25 mm internal diameter, 0.25 micron phase coating.

Oven Temperature Program: 50 degrees C. for 0.08 min hold time, 50 degrees C. to 220 degrees C. at 6.0 degrees C./min, hold at 220 degrees C. for 32 min.

GC Column Velocity: 30 cm/sec, Helium.

Detector: Electron Capture Detector (ECD), attenuation 1, range 10, autozero on.

Detector Temperature: 250 - 300 degrees C.

Injector Temperature Program: 45 degrees C. for 0.04 min hold time, 45 degrees C. to 250 degrees C. at 250 degrees C./min, hold at 250 degrees C. for 62.0 min.

Detector Make-up Gas: 30 mL/min, Nitrogen.

Auxiliary Temperature: 300 degrees C.

Autosampler: Multi-vial mode, 0.5 uL sample volume, Fast injection rate (4.0 uL/sec.), 0.0 min injection time, 3 purge pulses.

7.1.2 Initial GC Calibration: Prior to running samples the system must be calibrated and the system performance must be verified.

7.1.2.1 Establish the gas chromatographic operation parameters outlined in Section 7.1.1 and prepare the calibration standard composed of a mixture of Aroclors 1232, 1248, and 1262 as outlined in Section 6.13.

7.1.2.2 Load the gas chromatograph autosampler with a vial containing the calibration standard, bracketed by 2 wash vials containing acetone and proceed with the chromatographic analysis. During the chromatographic analysis, GC data acquisition should be performed for future peak integration and data manipulation.

7.1.2.3 Our laboratory will use a computer based data acquisition workstation (Dynamic Solutions, Maxima 820 workstation), interfaced to the gas chromatograph. The workstation processes the detector signal, performs an analog to digital conversion, and stores the digitized chromatograms on the computer hard disk. All data

analysis will be done on the computer specialized software package including peak integration, calculating calibration curves/response factors, report generation, chromatogram hardcopies, and archival of data.

7.1.2.4 Calculate the response factor for each separated and identified peak. Appendix A identifies which congener and or congeners compose each resolvable GC peak in the calibration standard, along with the amount that each congener or co-eluting group of congeners are represented in the calibration standard. Throughout this document the IUPAC PCB numbering system will be used for congener identification, unless otherwise stated. Appendix B is an example of an acceptable chromatogram of the calibration standard, along with peak congener labels for cross reference to data in Appendix A. Chromatographic resolution should be sufficient so as to separate congeners 17 and 18 into two peaks with a valley less than half height of congener 17. Response factors are calculated relative to the internal standard by the following equation:

$$RRF = (Ax/Ais)x(Cis/Cx)$$

Where:
- RRF = Relative response factor of congener(s).
- Ax = Area of peak for the cogener(s).
- Ais = Area of peak for the internal standard.
- Cx = Concentration of the cogener(s).
- Cis = Concentration of the internal standard.

7.1.2.5 For congeners that are not found in the mixed Aroclor standard and do not have amount values in the Appendix A table, the following calibration scheme will be employed. Response factors for these additional 19 chromatographic peaks will be adapted from the tabulated data of response factors and relative retention times of all the 209 PCB congeners found in Mullin et al, 1984 (see reference 9.10). These relative response factors will be adjusted for the specific data analysis and quantification employed in this document, based on the calculated response factors generated from the mixed Aroclor standard used for instrument calibration.

7.2 On-going Calibration:

7.2.1 Chromatographic Resolution:

7.2.1.1 Chromatographic resolution should be sufficient so as to separate congeners 17 and 18 into two peaks with a valley less than the half height of congener 17. If this separation is not met, install a new GC column or adjust instrument conditions to achieve stated separation.

7.2.2 Response Factors:

7.2.2.1 The relative response factors calculated from the calibration standard will be verified on each working day by analyzing the performance standard, calculating the selected congener concentrations and comparing to their known concentration. A subset of six congeners will be used to verify the relative response factors before samples are processed. The six congeners include:

#6 and #205 - representing low level peaks in standard

#61 and #181 - representing medium level peaks in the standard
#44 and #180 - representing high level peaks in the standard.

7.2.3 After the performance standard is analyzed, calculate the amount for these six congeners and compare those values to the known concentrations by the following equation:

Percent Difference = [Amt(1)-Amt(2)]/Amt(2) x 100

Where: Amt(1)= Amount calculated for congener.
Amt(2)= Known amount for congener.

7.2.4 A percent difference greater than ±30% for the two low level peaks (#6 and #205) indicates an instrument problem or unacceptable relative response factors. A percent difference greater than ±10% for the medium level (#61 and #185) and high level (#44 and #180) peaks also indicates an instrument problem or unacceptable relative response factors. If any of the evaluation congeners fail to meet the percent difference acceptance criteria, the calibration standard must be re-analyzed and new relative response factors generated.

7.2.5 The performance standard must be analyzed again and values calculated using the new relative response factors. If the performance standard fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (i.e., GC gas leak, autosampler lines plugged, broken injector liner).

7.3 Sample Preparation:

7.3.1 Soil and Sediment Samples:

7.3.1.1 To provide guidance to the analyst, as much information about the samples received for analysis should be obtained from the generator and included in the sample log-in records. This may provide information on the expected levels of components being determined and whether dilution of the sample will be necessary.

7.3.1.2 Refer to Section 7.1 for gas chromatographic conditions before running any samples. The instrument calibration performance criteria must be met (see Section 7.2).

7.3.1.3 Sample Preparation:

7.3.1.3.1 Process the sample by first decanting water from the top of the sediment. Transfer the sample to a clean pyrex tray and remove any sticks, stones, and other foreign material, if present.

7.3.1.3.2 Allow the sample to dry at room temperature by placing in a chemical hood for 24 to 48 hours. Frequently mix the drying sample with a spatula and break up any clumps by crushing. The sample is ready for extraction when the sample has reached a free flowing consistency.

7.3.1.4 Extraction:

7.3.1.4.1 Weigh 5 to 20 grams of the air dried sample into a tared cellulose extraction thimble (Whatman 33 mm x 94 mm) and record the weight. Add a plug of silanized glass wool to the top of the thimble.

7.3.1.4.2 Add 200 mL of 1:1 mixture of Hexane and Acetone (1:1 Hexane/Acetone) to a 250 mL round bottom flask. Add several boiling chips to the round bottom flask.

7.3.1.4.3 Turn on the cooling water to the condenser units that will be used to condense the extraction solvent during the soxhlet extraction of the sample.

7.3.1.4.4 Place the sample filled thimble into the soxhlet extractor and attach the soxhlet to the 250 mL round bottom flask and condenser unit.

7.3.1.4.5 Place the 250 mL round bottom onto a heating mantle and set the controller to a value of 5 to 6. Once the solvent begins to boil, adjust the controller to achieve a solvent flushing action of once every two to three minutes.

7.3.1.4.6 Extract the sample overnight for a minimum of 16 hours. Turn the heating mantle off and allow the apparatus to cool to room temperature. Once cool, rinse the water cooled condenser with several disposable pipette volumes of hexane. Separate the condenser from the soxhlet extractor and rinse the ground glass joint with several disposable pipette volumes of hexane.

7.3.1.4.7 Move the soxhlet extractor and attached 250 mL round bottom flask into a chemical hood. Flush the remaining solvent from the soxhlet extractor by tipping the unit to facilitate the syphon action. Using forceps, remove the extraction thimble and allow residual solvent to drain into soxhlet chamber. Rinse the soxhlet chamber with several disposable pipette volumes of hexane, adding enough solvent to be able to flush the soxhlet extractor by tipping to start the siphoning action. Disconnect the soxhlet extractor from the 250 mL round bottom flask and rinse the ground glass joint with several disposable pipette volumes of hexane.

7.3.1.4.8 Add several fresh boiling chips to the round bottom flask and attach a 3-ball snyder condensing column to the round bottom flask. Prewet the snyder column with 2 mL of hexane.

7.3.1.4.9 Using a heating mantle, evaporate the solvent to approximately 5 mL. Make sure gentle boiling is taking place and that the snyder column sections are not becoming flooded with solvent. Once the extract reaches approximately 5 mL (do not go to dryness or loss of PCBs can occur), remove from heat source and quickly rinse the snyder column with 2 mL of hexane. Allow the glassware to cool to room temperature. Loosen the glass joint between the snyder column and 250 mL round bottom flask and rinse the joint into the round bottom flask with 2 mL of hexane.

7.3.1.4.10 Quantitatively transfer the sample extract to a 20 mL vial with three 2 mL hexane rinses. After the sample has been transferred, rinse the disposable transfer pipette with 0.5 mL of hexane into the 20 mL vial.

7.3.1.4.11 Evaporate the solvent with a gentle stream of purified nitrogen gas using a Pierce Reacti-therm block equipped with a 9 port syringe needle gas manifold. Gentle heating can be applied (40-50 degrees C.) to facilitate solvent evaporation. The extract should be concentrated to 0.2 to 0.3 mL, but never allow extract to go to dryness (PCB losses can occur). This concentration step removes the final traces of acetone, which needs to be removed before clean-up procedures are performed.

7.3.1.4.12 At this point the sample can be processed in either of three ways:

1) If the sample is known to be free of interferences, the sample extract volume can be adjusted to 10 mL and and screened by the GC packed column qualitative analysis to determine PCB concentration and necessary sample extract dilution or concentration for final GC capillary quantification analysis.

2) If the exact and nature of interfering co-extractables in the sample is unknown, the sample must be analyzed by the GC packed column qualitative procedure to determine appropriate clean-up procedures. The sample extract is adjusted to 10 mL and analyzed by packed column chromatography. After review of the chromatogram, the sample extract is processed using applicable clean-up techniques. After sample clean-up the extract is re-analyzed by GC packed column chromatography to determine PCB concentration and necessary extract dilution or concentration for final GC capillary quantitative analysis.

3) If prior knowledge and information exists on what types of interfering substances will be co-extracted with the PCBs, proceed directly with the necessary clean-up procedures. After sample clean-up, the extract is adjusted to 10 mL and analyzed by GC packed column chromatography to determine PCB concentration for final GC capillary quantitative analysis.

7.3.1.4.13 Refer to Section 7.4 for sample extract clean-up procedures.

7.3.1.4.14 After the sample extract has been analyzed by the GC packed column qualitative screening protocol, the sample is prepared for final analysis by capillary gas chromatography. Dilute or concentrate the sample extract so that the PCB concentration is within the established concentration range (see Section 6.13.3). Spike this sample extract with the internal standard (OCN) as close to the same level that it exists in the calibration standards (typically 0.1818 ug/mL). Place the sample into an autosampler vial and proceed with the capillary gas chromatography analysis.

7.3.1.4.15 Refer to Section 7.5 for the calculations of PCB concentration in the sample.

7.3.1.4.16 Section 8.0 outlines the necessary quality control (QC) samples to be included with each sample analysis sequence. QC samples are prepared and analyzed along with the samples being measured, and must meet defined acceptance criteria for the sample data to be valid. The QC data is tabulated or plotted on control charts and archived by the laboratory and is available for inspection by the individual requesting the analysis.

7.4 Sample Clean-up

7.4.1 Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection

have co-extracted xenobiotics and other interfering substances that must be removed before accurate quantification can take place.

7.4.2 Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. It is the experience and knowledge of the analyst combined with the sampling site history that should guide the selection of which clean-up procedures are necessary.

7.4.3 Hexane/Dimethylformamide partition:

7.4.3.1 This procedure is similar to the hexane/acetonitrile partition procedure, but affords quantitative recover of PCBs. This clean-up technique effectively removes lipids and oils from the sample extracts. When necessary, this procedure should be carried out prior to Florisil clean-up.

7.4.3.2 Quantitatively transfer the sample extract to a 30 mL vial and adjust the sample volume to 15 mL with hexane. Extract the sample four times with 5 mL of dimethylformamide saturated with hexane.

7.4.3.3 Transfer each dimethylformamide extract to a 150 mL separatory funnel. Rinse the transfer pipette with a 0.5 mL of dimethylformamide saturated with hexane into the separatory funnel. Add 100 mL of water containing 2% sodium chloride to the separatory funnel.

7.4.3.4 Extract the dimethylformamide/water mixture with 20 mL of hexane. Allow the phases to separate for 5 minutes after vigorously shaking for 30 seconds.

7.4.3.5 Decant the dimethylformamide/water phase (lower) into a 250 mL beaker. Transfer the hexane phase to a column containing 3 inches of anhydrous sodium sulfate and elute hexane containing PCBs into a 250 mL round bottom flask.

7.4.3.6 Transfer the dimethylformamide/water fraction back to the 150 mL separatory funnel and extract a second time with 20 mL of hexane. Decant the dimethylformamide/water phase back into the 250 mL beaker and add the 20 mL hexane extract to the anhydrous sodium sulfate drying column.

7.4.3.7 Repeat Section 7.4.3.6 for a total of three hexane extractions.

7.4.3.8 Rinse the separatory funnel into the sodium sulfate column with several disposable pipette volumes of hexane. Rinse the column with several disposable pipette volumes of hexane. Elute the column with an additional 50 mL of hexane to remove all the PCBs. Finally, rinse the drain tube of the column stopcock into the round bottom.

7.4.3.9 Add several boiling chips to the 250 mL round bottom flask and rinse the neck of the round bottom with several disposable pipette volumes of hexane. Attach a 3-ball snyder distilling column to the 205 mL round bottom flask and prewet the snyder column with 2 mL of hexane.

7.4.3.10 Using a heating mantle, evaporate the solvent to approximately 5 mL. Make sure gentle boiling is taking place and that the snyder column sections are not becoming flooded with solvent. Once the extract reaches approximately 5 mL (do not go to dryness or loss of PCBs can occur), remove from heat source and quickly rinse the snyder column with 2 mL of hexane. Allow the glassware to cool to room temperature. Loosen the glass joint between the snyder column and 250 mL round bottom flask with 2 mL of hexane.

7.4.3.11 Quantitatively transfer the sample extract to a 15 mL vial with three 2 mL hexane rinses. After the sample has been transferred, rinse the disposable transfer pipette with 0.5 mL of hexane into the 15 mL vial.

7.4.3.12 Evaporate the solvent with a gentle stream of purified nitrogen gas using a Pierce Reacti-therm heating block equipped with a 9 port syringe needle gas manifold. Gentle heating can be applied (40-50 degrees C.) to facilitate solvent evaporation. The extract should be concentrated to 0.2 to 0.3 mL, but never allow extract to go to dryness (PCB losses can occur).

7.4.3.13 Quantitatively transfer the sample to a 10 mL volumetric flask and proceed with the packed column GC analysis.

7.4.4 Sulfuric Acid Wash:

7.4.4.1 The concentrated sulfuric acid treatment removes hydrocarbons and other colored biogenic compounds which are co-extracted with the environmental PCB residues. The sulfuric acid wash should be done first if a florisil clean-up is also being applied to the sample.

7.4.4.2 Quantitatively transfer the sample extract to a 20 mL vial and adjust the volume to approximately 5 mL.

7.4.4.3 Add 2 mL of concentrated sulfuric acid to the sample extract and shake vigorously for 30 seconds. Centrifuge samples on low speed (2) using a bench top centrifuge. Transfer the hexane upper layer to a 20 mL vial.

7.4.4.4 Wash the sulfuric acid three times with 2 to 3 mL of hexane. Shake each wash vigorously and centrifuge to aid in phase separation. Transfer all three hexane washes to the 20 mL vial.

Make sure to rinse the transfer pipette with 0.5 mL of hexane into the 20 mL vial.

7.4.4.5 Evaporate the solvent with a gentle stream of purified nitrogen gas using a Pierce Reacti-therm heating block equipped with a 9 port syringe needle gas manifold. Gentle heating can be applied (40-50 degrees C.) to facilitate solvent evaporation. The extract should be concentrated to 0.2 to 0.3 mL, but never allow extract to go to dryness (PCB losses can occur).

7.4.4.6 If the sample is to be analyzed at this point, quantitatively transfer the extract to a 10 mL volumetric and proceed with the packed column GC screening analysis.

7.4.4.7 If the sample needs further clean-up, proceed to the next clean-up procedure.

7.4.4.8 Caution must be taken in performing the sulfuric acid wash when high amounts of lipid (i.e., fish samples) or water remain in the sample extract. These types of samples may produce excessive heat due to the exothermic reaction of the sulfuric acid with these materials. The rise in sample temperature can cause sulfonation of the lower chlorinated PCB congeners and therefore losses of PCB from the sample. If high amounts of lipids and water are suspected, cool the sample in an ice bath. Also cool the concentrated sulfuric acid in an ice bath before addition to the sample. Gently shake the sample extract and keep the sample in the ice bath while treating it with the sulfuric acid.

7.4.4.9 The pesticides dieldrin and endrin are destroyed by the sulfuric acid treatment. If these pesticides are to be measured they must be fractionated (i.e., silica column) from the PCBs.

7.4.5 Florisil Adsorption Chromatography:

7.4.5.1 The florisil chromatography separates PCBs and certain pesticides (i.e., DDT and analogs) from other co-extracted, polar compounds. The procedure outlined below is for collection of the fraction that contains PCBs and certain organochlorine pesticides only. Additional fractions, eluted by stronger polarity solvent mixtures can be collected if other pesticides are to be determined.

7.4.5.2 Adjust the sample extract volume to 0.2 to 0.3 mL, so that the sample is applied to the florisil column in a small chromatographic band.

7.4.5.3 Prepare a micro florisil column in the following manner. Cut the top of a 5 mL disposable pipette at approximately 1 inch from the end of the pipette. Insert a small plug of silanized glass wool into the pipette and position at the 9 mL mark. Dry pack the 5 mL pipette with 100% deactivated Florisil to make a column of

1 mL in height. Make sure the Florisil is well settled by tapping the column with a spatula. Rinse the micro column with 10 mL of hexane and after elution of the 10 mL, rinse the outside tip of the pipette column, with 1 mL hexane. Place a 20 mL vial under the micro column for collection of the elute.

7.4.5.4 Quantitatively transfer the sample extract to the Florisil micro column and rinse the sample container with 3 successive 0.5 mL hexane volumes. Rinse the disposable transfer pipette into the micro column with 0.5 mL of hexane. Carefully rinse the micro column with 1 mL of hexane and then elute the sample to a total volume of 20 mL with hexane. After collection of the sample rinse the outside tip of the micro column into the collection vial.

7.4.5.5 Evaporate the solvent with a gentle stream of purified nitrogen gas using a Pierce Reacti-therm heating block equipped with a 9 port syringe needle gas manifold. Gentle heating can be applied (40-50 degrees C.) to facilitate solvent evaporation. The extract should be concentrated to 0.2 to 0.3 mL, but never allow extract to go to dryness (PCB losses can occur).

7.4.5.6 If the sample is to be analyzed at this point, quantitatively transfer the extract to a 10 mL volumetric and proceed with the packed column GC screening analysis.

7.4.5.7 If the sample needs further clean-up, proceed to the next clean-up procedure.

7.4.6 Sulfur Removal:

7.4.6.1 Elemental sulfur is soluble in the extraction of solvents used for sediment and soil samples. It is almost always found as an interferant in these types of samples. Large amounts of sulfur can cause the electron capture detector to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can interfere with PCB measurement by co-eluting as a chromatographic peak with certain PCB congeners.

7.4.6.2 Sulfur removal clean-up should be routinely done on soil and sediment samples due to its ubiquitous nature. The sulfur removal should be done prior to sulfuric acid and column chromatography clean-up techniques.

7.4.6.3 Quantitatively transfer the sample extract to a 20 mL vial and adjust the volume to approximately 5 mL.

7.4.6.4 Add 0.3 to 0.5 mL of elemental mercury to the sample extract and vigorously shake for 1 minute. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract.

7.4.6.5 Quantitatively transfer the sample extract to a 20 mL vial by using three successive 1.0 mL hexane rinses. Rinse the disposable transfer pipette into the vial with 0.5 mL of hexane.

7.4.6.6 The precipitated sulfur can be removed from the sample by performing a sulfuric acid clean-up or Florisil adsorption column clean-up.

7.4.6.7 After removal of the sulfur precipitate by either method listed in section 7.4.6.6, quantitatively transfer the sample extract to a 10 mL volumetric flask and proceed with the packed column GC screening analysis.

7.4.6.8 If the sample needs further clean-up, proceed to the next clean-up procedure.

7.4.7 Silica Gel Adsorption Chromatography:

7.4.7.1 Co-extracted organochlorine pesticides can interfere with PCB identification and quantification. A separation of PCBs from organochlorine pesticides can be accomplished by silica gel adsorption chromatography, allowing the analysis of both PCBs and pesticides as separate fractions.

7.4.7.2 Adjust the sample volume to 0.2 to 0.3 mL with hexane. Prepare a column of 7.5% deactivated silica gel (100-200 mesh, Grade 923, Aldrich), packed with 11.5 g of the silica gel. Rinse the column with 50 mL of methylene chloride followed by 50 mL of pentane. Rinse the stopcock drain tube with 1.0 mL of pentane. Place a 250 mL round bottom under the column to collect the sample eluate.

7.4.7.3 Quantitatively transfer the sample to the silica column using three successive 0.5 mL pentane rinses. Rinse the disposable transfer pipette into the column with 0.5 mL pentane. Rinse the column walls with several disposable pipette volumes of pentane. Elute the silica gel column with 50 mL of pentane. Rinse the stopcock drain tube with 1.0 mL of pentane. This fraction will contain the PCBs.

7.4.7.4 Place a 50 mL bottle under the silica gel column. Elute the silica gel column with 36 mL of 20% methylene chloride in pentane. This second fraction will contain the organopesticides and polychromatic hydrocarbons. This fraction can be stored in a refrigerator and analyzed at a later date if pesticide determination is requested.

7.4.7.5 To the round bottom flask containing the PCB fraction add several boiling chips and rinse the neck of the round bottom flask with several disposable pipette volumes of hexane. Attach a

3-ball snyder distilling column to the 250 mL round bottom flask and prewet the snyder column with 2 mL of hexane.

7.4.7.6 Using a heating mantle, evaporate the solvent to approximately 5 mL. Make sure gentle boiling is taking place and that the snyder column sections are not becoming flooded with solvent. Once the extract reaches approximately 5 mL (do not go to dryness or loss of PCBs can occur) , remove from heat source and quickly rinse the snyder column with 2 mL of hexane. Allow the glassware to cool to room temperature. Loosen the glass joint between the snyder column and 250 mL round bottom flask and rinse the joint into the round bottom flask with 2 mL of hexane.

7.4.7.7 Quantitatively transfer the sample extract to a 15 mL vial with three 2 mL hexane rinses. After the sample has been transferred, rinse the disposable transfer pipette with 0.5 mL of hexane into the 15 mL vial.

7.4.7.8 Evaporate the solvent with a gentle stream of nitrogen gas using a Pierce Reacti-therm heating block equipped with a 9 port syringe needle gas manifold. Gentle heating can be applied (40-50 degrees C.) to facilitate solvent evaporation. The extract should be concentrated to 0.2 to 0.3 mL, but never allow extract to go to dryness (PCB losses can occur).

7.4.7.9 If the sample is to be analyzed at this point, quantitatively transfer the extract to a 10 mL volumetric and proceed with the packed column GC screening analysis.

7.4.7.10 If the sample needs further clean-up, proceed to the next clean-up procedure.

7.5 Calculations:

7.5.1 External Standard Calibration (Packed GC):

7.5.1.1 The packed column GC screening analysis will be done by the external standard calibration technique. Calibration and sample quantification will be performed by a commercial GC software package installed on a personal computer. The GC will be standardized by using Aroclor 1242 and Aroclor 1260. These two Aroclor formulations incorporate most environmental PCBs found in sample extracts and provide a good estimate of PCB amount for final dilution or concentration for capillary analysis. A multi-level calibration curve will be developed based on 1 ppm and 10 ppm standards. Weight percent data (Webb and McCall) will be used to generate standard peak amounts.

7.5.1.2 The calibration curves for each calibrated PCB peak will be calculated using the following formula:

$$\text{Calibration factor} = \frac{\text{Amount (ug) of component}}{\text{Total area of analyte peak}}$$

The calibration curve will be fitted to the calculated calibration factors by a cubic equation to give the best line for all calibration points.

7.5.2 Sample Calculations (Packed GC)

7.5.2.1 The concentration of each identified PCB peak in a sample will be calculated based on the extract volume (not the sample weight or volume) to supply solution concentration values that show if the extract needs to be diluted or concentrated for final capillary GC analysis. The solution concentration of each standardized PCB peak in a sample is calculated as follows:

Concentration (ug/mL) = (Ax) x (CF)

Where: Ax=Area of peak of interest in sample
CF=Calibration factor from peak in standard.

7.5.3 Internal Standard Calibration (Capillary GC)

7.5.3.1 The capillary column GC analysis will be done by the internal calibration technique. Calibration and sample quantification will be performed by a commercial GC software package installed on a personal computer. The capillary GC will be standardized by using an Aroclor mixture that encompasses all the possible PCB congeners present in environmental samples. Refer to Section 6.13 for details on the calibration standard and Aroclor ratios.

7.5.3.2 Response factors for each separated and identified peak in the standard will be calculated using the following formula:

$$RRF = (Ax/Ais) \times (Cis/Cx)$$

Where: RRF = Relative response factor of congener(s).
Ax = Area of peak for the congener(s).
Ais = Area of peak for the internal standard.
Cx = Concentration of the congener(s).
Cis = Concentration of the internal standard.

7.5.3.3 For the 19 chromatographic peaks that will utilize response factors from tabulated data found in Mullin et at, 1984 (see Section 7.1.2.5), numbers will be manually entered into the peak calibration table used for sample quantification. All calculations will proceed exactly as outlined above for peaks that

have response factors generated from amount information contained in Appendix A.

7.5.4 Sample Calculations (Capillary GC)

7.5.4.1 The concentration of each identified PCB peak in a sample will be calculated based on the sample air dried weight.

7.5.4.2 The sample PCB concentration of each standardized PCB peak is calculated as follows:

$$\text{Concentration (ng/g)} = \frac{[(Ax)(Cis)(D)]}{[(Ais)(RRF)(Ws)]}$$

Where:
- Ax = Peak area for congener(s) being measured.
- Cis = Amount of internal standard added to sample extract
- D = Dilution factor, if sample was diluted prior to analysis.
- Ais = Peak area of added internal standard.
- RRF = Relative response factor for congener(s) being measured,as determined in Section 7.5.3.2.
- Ws = Air dried weight of sample.

The dilution factor (D) is based on a final extract volume of 1 mL. Typically, the sample is extracted with about 200 mL which is then cleaned up and concentrated to a final volume of 10 mL. If no further volume adjustment is required, then D=10. If further dilutions are required, the D=10 x (dilution #1) x (dilution #2) x...

7.5.5 Data Output and Reporting Format:

7.5.5.1 Several specialized software routines have been developed for high resolution PCB analysis to aid the researcher in understanding and organizing the complex data generated from this extremely detailed analysis. Appendix C contains examples of the sample hard copy format that will be used in reporting sample information.

8.0 Quality Control

8.1 Table 3 lists the Quality Control requirements and required recording format that will be applied to the capillary gas chromatographic analysis of PCBs in soils and sediments.

Table 3 - Quality Control Requirements

QC Sample	Reporting Format	Frequency
Lab Blank	Tabulation	Daily, or with each sample analysis sequence (up to 20 samples).
Performance check	Tabulation	Performance check sample analyzed prior to each sample analysis sequence (up to 10 samples).
Duplicate Analysis	Tabulation	One duplicate analysis per 10 field samples, or monthly if less than 10 samples/month are analyzed.
Matrix Spike	Tabulation	One matrix spike per 10 field samples.

8.1.1 Sample Records: All samples that arrive at the lab should be accompanied by a chain of custody document. The following pertinent information should be documented for all samples:

1) Unique label that identifies sample.

2) Location of sample collection site.

3) Date and time of collection.

4) Project name and/or ID number.

5) Field personnel at sampling.

6) Required analysis.

The sample information will be recorded in the Lab Log Books and each sample will be assigned a unique Lab ID Number. The sample analysis will be archived by computer using the Lab ID Number.

8.1.2 Laboratory Blank: The laboratory blank will monitor and assess if the contamination of excessive interference is occurring from laboratory solvents, reagents, and glassware used in processing samples for analysis. The laboratory blank is taken through the sample extraction and clean-up procedures to include all manipulations exposed to actual samples (required volume of solvents, concentration steps, clean-up procedures, etc.). If the laboratory blank is positive for PCB contamination, the source of the contamination must be traced down and eliminated before samples can be processed and analyzed. If non-PCB contamination occurs that interferes with PCB quantification, it too must be traced down and eliminated before proceeding with sample analysis.

The laboratory blank will consist of sandbox sand, purchased at a local hardware store, which has been baked at 250 degrees C. in a vacuum

oven for 24 to 36 hours and allowed to cool and store in glass containers. This is the same procedure used to prepare material which is supplied to EPA's contract laboratories by EMSL/Cincinnvti, for use as clean solid matrix.

8.1.2.1 Samples analyzed after a positive laboratory blank should be considered unreliable. If a laboratory blank is positive for PCBs, the source of contamination shall be located and eliminated. If the contamination occurred during the extraction procedure, the samples will require re-extraction and re-analysis. If the contamination occurred after this step, then re-extraction is not appropriate and the existing extracts will be reanalyzed. Any aliquots of the extracts (i.e., injection vials) which could have become contaminated will be discarded.

8.1.3 Performance Check Standard: As outlined in section 7.2, a performance check standard will be analyzed on each working day prior to sample analysis and at an interval of one performance standard per each sample analysis sequence (up to 10 samples). The performance check standard must meet the acceptance criteria established in Section 7.2. If the performance check standard fails to meet the acceptance criteria, the calibration standard must be analyzed and new response factors generated.

8.1.3.1 The performance check standard must be analyzed again and compared to the acceptance criteria. If the performance check standard fails to meet the acceptance criteria after re-calibration, sample analysis must not proceed until the problem is corrected. A typical analytical sequence is as follows:

1)	Performance check standard.
2)	Method blank.
3 thru 11)	Samples (including duplicates, MS).
12)	Performance check standard.
13 thru 21)	Samples (including duplicates, MS).
22)	Performance check standard.

8.1.3.2 All samples that were analyzed after the performance check standard exceeded established criteria must be re-analyzed.

8.1.4 Duplicate Analysis: Duplicate analysis of the same sample are performed to assess method precision. The percent relative standard deviation of the two measurements on the sample is calculated on total PCB concentration by the following equation:

$$RSD=(DUP1 - DUP2)/AVG \times 100$$

Where:
- RSD = Percent relative standard deviation.
- DUP1 = the greater of the measured values.
- DUP2 = the lesser of the measured values.
- AVG = average of the two analyses.

8.1.4.1 The percent relative standard deviation must be less than or equal to 25% if the concentration of PCB in the sample is greater than or equal to 0.5 ppm. The percent relative standard deviation must be less than or equal to 50% if the concentration of the PCB in the sample is less than 0.5 ppm.

8.1.5 Matrix Spike: Spiked sample matrix data are analyzed to assess analytical accuracy. Thus the sample is spiked and carried through sample analytical procedures including extraction, clean-up, and GC analysis. Matrix spike duplicates are not required for this project.

8.1.5.1 If a sample matrix previously analyzed to be free of PCB is available, spike this sample with matrix spike standard (see Section 6.16) at a concentration similar to sample concentrations. Extract and analyze this spiked sample following procedures used for actual sample analysis. Calculate the percent recovery of the matrix spike by the following:

$$P = A/T \times 100$$

Where: P=Percent recovery, %.
A=Concentration of analyte in the spiked sample aliquot.
T=Known true value of the spike concentration added to the sample aliquot.

8.1.5.2 If an uncontaminated sample is not available, the matrix spike can be performed on a sample previously analyzed. There must be sufficient sample for re-analysis as a matrix spike and the sample must be homogeneous in PCB distribution for valid data to be produced. Preferably a sample of low level should be used in this case. Spike the sample with the matrix spike standard (see Section 6.16) at a concentration similar to sample concentrations. Extract and analyze this spiked sample following procedures used for actual sample analysis. Calculate the percent recovery of the matrix spike by the following:

$$P = (A-B)/T \times 100$$

Where: P=Percent recovery, %.
A=Concentration of analyte in the spiked sample aliquot.
T=Known true value of the spike concentration added to the sample aliquot.
B=Background concentration of PCB in the unspiked sample aliquot.

8.1.5.3 The percent recovery for a spiked matrix sample must be greater than or equal to 70% and less than or equal to 130%, based on the total PCB concentration. If a matrix spike recovery sample does not meet the acceptance criteria the cause must be found and corrected. All data collected on samples after the spiked

recovery sample failed to meet the acceptance criteria should be considered unreliable and samples must be re-extracted and analyzed. If additional sample is unavailable, then the data must be flagged as such.

8.1.6 Retention Time Windows

8.1.6.1 The GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Make three analytical measurements of the standard mixture containing the compounds of interest of a minimum of a 72-hour period.

8.1.6.2 Calculate the standard deviation resulting from the variation in retention times for each component from three analytical runs.

8.1.6.3 The retention time window is defined as plus or minus three times the standard deviation of the three retention time determinations.

8.1.6.4 If the standard deviation for a particular component is zero, substitute the standard deviation of a close eluting compound found in the standard solution.

8.1.6.5 Besides using the retention time window to assign peaks for quantification, the analyst should also rely on his experience in pattern recognition of multi-residue sample analysis.

8.1.6.6 After the daily performance check standard has been analyzed, establish the daily retention time window for each analyte from the retention time of the standard just analyzed. The daily retention time window equals the absolute retention time of the performance check standard (midpoint of the window for the day) plus or minus three times the standard deviation determined in Section 8.1.6.2.

8.1.7 Qualitative Compound Identification/Confirmation: The identification of specific congeners in a PCB mixture is done by comparing the retention time of each peak with the retention times of the peaks in a known standard. The standard, consisting of a specified mixture of three Aroclors (1232, 1248, and 1262), has been completely characterized by workers at EPA's Large Lakes Research Station (see Mullin, 1985). Repeated injections of the standard are used to establish an acceptable "retention time window" for each peak. It should be noted, also, that the experience of the analyst plays a significant role in the recognition of PCB patterns. The Laboratory Director of Northeast Analytical, Inc., Robert Wagner, has over 8 years of experience in performing congener-specific analysis of PCBs. Comparative studies using GC/MS and GC-HECD detector analytical techniques have confirmed the validity of this method.

Potential interferences to the PCB analysis are minimized by a number of mechanisms. These include:

1. The EC detector is largely specific to chlorinated compounds.
2. Rigorous clean-up steps are performed to remove specific interferants, including pesticides, sulfur, lipids and oils, hydrocarbons and biogenic matter. The analyst will strive to produce an extract which is free of interferences.

9.0 References

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Appendix A

Congener Composition of Aroclor Calibration Mixture (6.1 ppm)

(Aroclors 1232, 1248, 1262 in a ratio of 25:18:18)

Peak Number	RRT	Amount (ng/mL)	Congener ID
1	0.3243	430	001
2	0.3536	*	002
3	0.3540	260	003
4	0.3685	28	004, 010
5	0.3887	22	007, 009
6	0.3941	42	006
7	0.3980	500	005, 008
8	0.4102	*	014
9	0.4106	10	019
10	0.4133	*	030
11	0.4220	*	011
12	0.4245	9.2	012, 013
13	0.4269	130	015, 018
14	0.4285	74	017
15	0.4339	8.8	024, 027
16	0.4389	131	016, 032
17	0.4436	*	023
18	0.4485	*	034, 054
19	0.4495	1.8	029
20	0.4517	23	026
21	0.4530	10	025
22	0.4564	166	031
23	0.4573	214	028, 050
24	0.4631	168.5	020, 021, 033, 053
25	0.4670	116.7	022, 051
26	0.4707	27	045
27	0.4746	*	036
28	0.4753	14	046
29	0.4790	*	039
30	0.4798	129.1	043, 052, 073
31	0.4825	90	049
32	0.4844	50	047
33	0.4854	40	048, 075
34	0.4897	*	062, 065
35	0.4915	*	035
36	0.4915	150	044, 104
37	0.4936	88	037, 042, 059
38	0.4990	163	041, 064, 071, 072
39	0.5026	*	068
40	0.5041	*	096
41	0.5031	33	040
42	0.5085	*	057, 103
43	0.5094	5	067, 100

Peak Number	RRT	Amount (ng/mL)	Congener ID
44	0.5119	7.4	058, 063
45	0.5145	81	074, 094
46	0.5164	210	061, 070, 076
47	0.5182	272	066, 093, 095
48	0.5227	14	055, 091, 098
49	0.5274	180	056, 060
50	0.5308	43	084, 092, 155
51	0.5324	3	089
52	0.5346	48	090, 101
53	0.5337	23	099
54	0.5421	1.8	112, 119, 150
55	0.5437	3.6	083, 109
56	0.5472	19	086, 097, 152
57	0.5500	33.2	081, 087, 111, 115
58	0.5528	21	085, 116
59	0.5549	14	136
60	0.5570	71	077, 110
61	0.5630	*	154
62	0.5637	13	082
63	0.5688	57	151
64	0.5714	22	124, 135
65	0.5727	2.23	144
66	0.5739	3.3	107, 108, 147
67	0.5770	*	123
68	0.5775	145	106, 118, 149
69	0.5833	*	139, 140
70	0.5858	8.5	114, 134, 143
71	0.5875	0.91	122, 131, 133, 142
72	0.5945	16	146, 161
73	0.5975	68.04	105, 132
74	0.6001	147.96	153
75	0.6100	*	168
76	0.6106	52	141
77	0.6121	54.6	179
78	0.6163	2.5	130
79	0.6193	13.88	137, 176
80	0.6237	98	138, 160, 163, 164
81	0.6274	12	158
82	0.6315	3	129
83	0.6379	34	178
84	0.6430	*	166
85	0.6439	6	175
86	0.6468	150	182, 187
87	0.6497	4.7	128
88	0.6529	77	183
89	0.6583	1.1	167
90	0.6651	22	185
91	0.6724	110	174, 181
92	0.6778	57	177
93	0.6838	36.9	156, 171

Peak Number	RRT	Amount (ng/mL)	Congener ID
94	0.6889	3.31	202
95	0.6919	*	157
96	0.6928	1.273	173
97	0.6996	20.697	200, 204
98	0.7045	19.2	172, 192
99	0.7104	2.18	197
100	0.7141	240	180
101	0.7190	14	193
102	0.7251	4.5	191
103	0.7323	10	199
104	0.7554	91.1	170
105	0.7611	29.9	190
106	0.7677	0.4	169
107	0.7779	6.7	198
108	0.7826	150	201
109	0.7036	170	196,203
110	0.8171	1.8	189
111	0.8484	55.9	195
112	0.8583	24.9	208
113	0.8774	4.8	207
114	0.9058	69	194
115	0.9241	4	205
116	1.0271	42	206
117	1.0838	0.95	209

RRT= Relative Retention Time to internal standard OCN= 1.0000

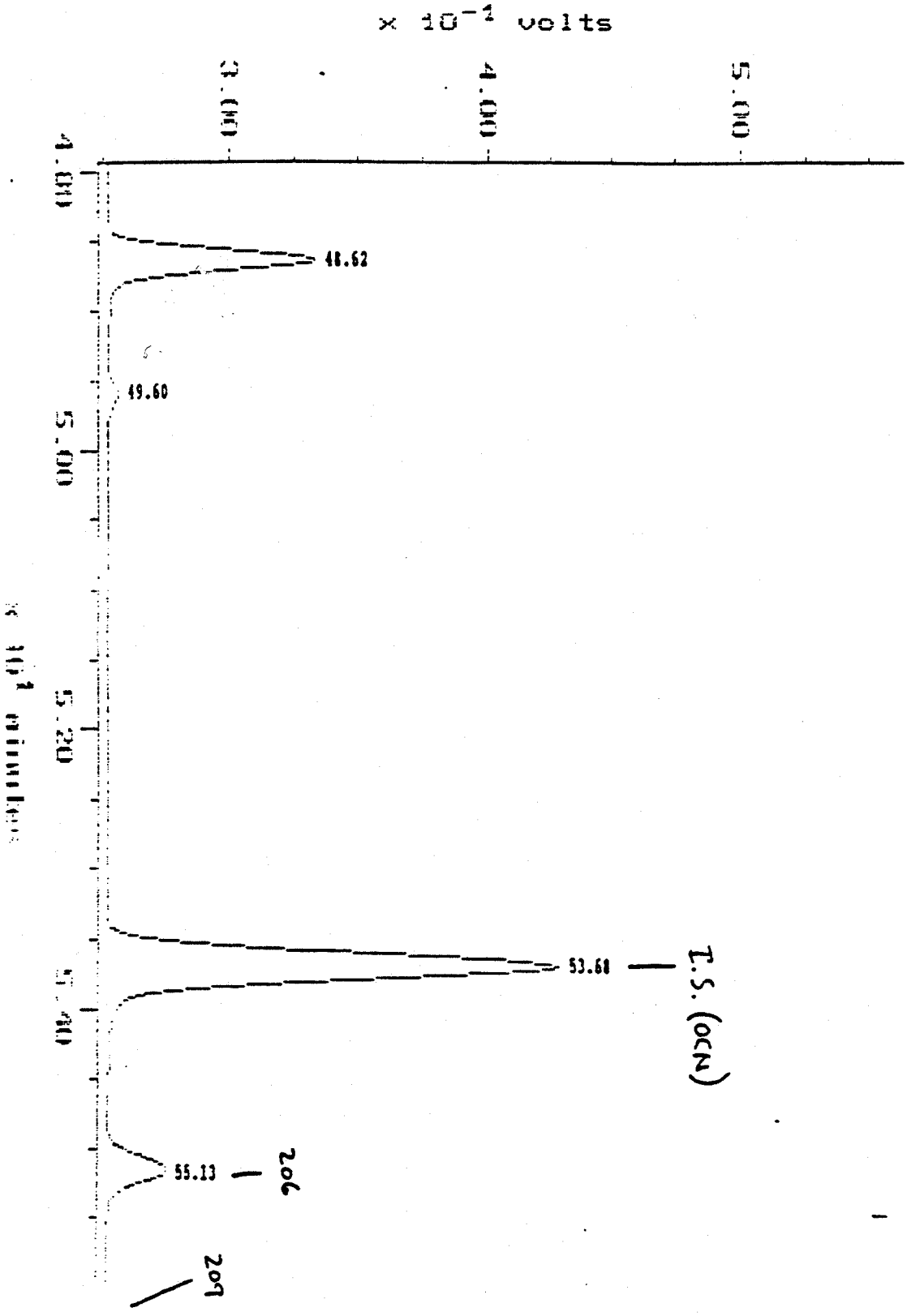
* Refer to Section 7.125 for description of peak quantification for congeners with no amount values.

Appendix B

DB-1 Capillary GC Chromatogram of Aroclor Mixture

(Aroclors 1232, 1248, 1262 in a ratio of 25:18:18)

Sample: 1232/1248/1262 Channel: detector 1 Filename: A324862
Acquired: 27-JUN-89 13:11 Method: C:\MAZ\GC1\118A Operator: RZW



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Appendix C

Format of sample reports and specialized data handling and data reduction reports.

MAXIMA 820 CUSTOM REPORT

Printed: 27-JUL-1989 15:31:58

SAMPLE SEDIMENT, SECTION 617
#7 in Method: HIGH RES AROCLOR QUANT.
ACQUIRED: 20-JUL-1989 10:34
Rate: 5.0 points/sec
Duration: 70.000 minutes
Operator: REW

TYPE: UNKN
Instrument: GC2
FILENAME: 89001
Index: Disk

Dilution: 4.167
Amount: 0.500

DETECTOR: detector 1

ID#	PK#	RETENTION TIME (minutes)	PEAK NAME (chlorine no.)	Peak Area	AMOUNT SOULTION (ug/ml)	SAMPLE AMOUNT (ug/g)
2	4	17.48	2 (1)	16822.5	0.052630*	0.43862*
5	7	19.89	5 (4,10)	94056.9	0.164038!	1.36709!
6	9	20.88	6 (7,9)	4289.9	0.001725	0.01438
7	10	21.24	7 (6)	9915.7	0.004179	0.03483
8	11	21.45	8 (5,8)	45470.5	0.020305	0.16922
10	12	22.13	10 (19)	59987.6	0.027093	0.22579
14	13	23.02	14 (15,18)	21477.3	0.011548	0.09624
15	14	23.09	15 (17)	13372.2	0.005714	0.04762
16	15	23.38	16 (24,27)	62691.7	0.013882	0.11569
17	16	23.66	17 (16,32)	31481.6	0.007599	0.06333
19	17	24.06	19 (34,54)	5020.4	0.001119*	0.00933*
21	18	24.34	21 (26)	27177.8	0.005058	0.04215
22	19	24.42	22 (25)	21079.9	0.004588	0.03824
23	20	24.60	23 (31)	34627.1	0.006847	0.05706
24	21	24.64	24 (28,50)	37269.8	0.009023	0.07520
25	22	24.99	25(20,21,33,53)	37185.8	0.008428	0.07024
26	23	25.16	26 (22,51)	28349.4	0.003843	0.03203
27	24	25.37	27 (45)	4313.7	0.001029	0.00858
29	25	25.61	29 (46)	3220.1	0.001429	0.01191
31	26	25.86	31 (52,73)	54018.5	0.015520	0.12935
32	27	26.01	32 (49)	71918.7	0.015636	0.13031
33	28	26.10	33 (47)	98758.1	0.017085	0.14239
37	29	26.49	37 (44,104)	20673.3	0.003803	0.03169
38	30	26.60	38 (37,42,59)	23516.2	0.005290	0.04409
39	32	26.89	39(41,64,71,72)	37428.5	0.004803	0.04003
41	33	27.03	41 (96)	8237.6	0.002223*	0.01853*
42	34	27.11	42 (40)	2516.7	0.000407	0.00339
43	35	27.31	43 (57,103)	11719.7	0.002115*	0.01763*
44	36	27.49	44 (67,100)	10848.3	0.001987*	0.01656*
45	37	27.59	45 (58,63)	3222.5	0.000525*	0.00437*
46	38	27.72	46 (74,94)	13739.7	0.001839	0.01533
48	40	27.97	48 (66,93,95)	35057.2	0.005087	0.04239
49	41	28.17	49 (55,91,98)	38865.8	0.007035	0.05863
50	42	28.42	50 (56,60)	10320.4	0.001376	0.01146
51	43	28.62	51 (84,92,155)	61168.2	0.010703	0.08920
53	44	28.82	53 (90,101)	119458.8	0.026868	0.22392
54	45	28.98	54 (99)	66138.0	0.011244	0.09371
55	46	29.23	55(112,119,150)	19896.4	0.002236*	0.01863*
56	47	29.31	56 (83,109)	8803.6	0.001829	0.01525
57	48	29.50	57 (86,97,152)	18519.2	0.002503	0.02086
58	49	29.66	58 (87,111,115)	27694.5	0.003021	0.02518
59	50	29.81	59 (85,116)	32043.9	0.002879	0.02399
60	99	54.22	I.S. (OCN)	4566128.9	0.202000	
TOTAL				20278027.1	0.49676	4.14000

MAXIMA 820 CUSTOM REPORT

Printed: 27-JUL-1989 16:35:48

SAMPLE: SEDIMENT, SECTION 617
87 in Method: HIGH RES AROCLOR QUANT.
ACQUIRED: 20-JUL-1989 10:34
Rate: 5.0 points/sec
Duration: 70.000 minutes
Operator: REW

TYPE: UNKN
Instrument: GC2
FILENAME: 89001
Index: Disk

Dilution: 4.167
Amount: 0.500

DETECTOR: detector 1

ID#	PK#	RETENTION TIME (minutes)	PEAK NAME (chlorine no.)	Peak Area	AMOUNT SOULTION (ug/ml)	SAMPLE AMOUNT (ug/g)
1	51	29.92	60 (136)	49734.7	0.010919	0.09100
2	52	30.04	61 (77,110))	124172.2	0.016898	0.14083
3	53	30.30	62 (154)	13529.7	0.002472*	0.02060*
4	54	30.40	63 (82)	4784.6	0.000469	0.00391
5	55	30.69	64 (151)	131715.2	0.020118	0.16767
6	56	30.82	65 (124,135)	64105.8	0.007402	0.06169
8	57	30.96	67(107,108,147)	98570.7	0.014373	0.11979
10	58	31.17	69(106,118,149)	571825.7	0.088853	0.74050
12	59	31.58	71(114,134,143)	20598.9	0.002379	0.01983
13	60	31.79	72(122,31,33,42	15905.4	0.001914*	0.01595*
14	61	32.09	73 (146,161)	92894.3	0.013138	0.10949
15	62	32.26	74 (105,132)	118195.4	0.009147	0.07623
16	63	32.39	75 (153)	326473.3	0.075866	0.63227
18	64	32.97	77 (141)	117796.2	0.007936	0.06614
21	66	33.36	80 (137)	40671.6	0.006651	0.05543
22	67	33.44	81 (176)	32099.3	0.002073*	0.01728*
23	68	33.69	82(138,160,3,4)	404734.7	0.060371	0.50313
24	69	33.88	83 (158)	43052.2	0.003625	0.03021
25	70	34.11	84 (129)	7992.3	0.000879	0.00733
26	71	34.45	85 (178)	54400.3	0.007313	0.06095
28	72	34.78	87 (175)	12024.6	0.002160*	0.01800*
29	73	34.94	88 (182,187)	240682.9	0.035214	0.29347
30	74	35.10	89 (128)	39935.1	0.002761	0.02301
31	75	35.27	90 (183)	134422.3	0.018141	0.15118
32	76	35.57	91 (167)	14693.5	0.002[illegible]7	0.02231
33	77	35.94	92 (185)	46459.0	0.003893	0.03245
34	78	36.34	93 (174,181)	247191.1	0.029164	0.24305
35	79	36.64	94 (177)	143527.1	0.016853	0.14046
36	80	36.97	95 (156,171)	107088.6	0.009579	0.07983
38	81	37.24	97 (157)	20714.4	0.003228	0.02683
39	82	37.45	98 (173)	5934.7	0.000200*	0.00166*
40	83	37.83	99 (200,204)	15595.0	0.001545*	0.01288*
41	84	38.09	100 (172,192)	57179.2	0.005783	0.04820
43	85	38.62	102 (180)	567316.0	0.060907	0.50760
44	86	38.89	103 (193)	32653.3	0.001589*	0.01324*
45	87	39.23	104 (191)	13896.5	0.000639*	0.00533*
46	88	39.62	105 (199)	22381.4	0.001327*	0.01106*
47	89	40.88	106 (170)	316544.6	0.023393	0.19496
48	90	41.18	107 (190)	73528.3	0.004853	0.04045
49	92	42.11	108 (198)	10419.1	0.000479*	0.00400*
50	93	42.37	109 (201)	147378.5	0.011801	0.09835
51	94	42.95	110 (196,203)	193149.8	0.014378	0.11982
52	95	44.24	111 (189)	11566.8	0.000379*	0.00316*
53	96	45.96	112 (195)	82128.2	0.005304	0.04420
56	97	49.10	115 (194)	195965.6	0.010702	0.08919
57	98	50.10	116 (205)	7874.8	0.000275*	0.00229*
58	99	54.22	I.S. (OCN)	4566128.9	0.202000	
TOTAL				9776276.4	0.620012*	5.15719*

NORTHEAST ANALYTICAL, INC.

301 MOTT STREET
SCHENECTADY, NY 12305
(518) 346-4592

PCB CONGENER AMOUNT and NANOMOLE REPORT

NEA FILE NAME: 89001.MOL

CUSTOMER: J. EPA
SAMPLE DESCRIPTION: SEDIMENT, SECTION 617
COMMENT: SAMPLE SHOWED ENVIRONMENTAL ALTERATION

TYPE FOR MIXED PEAK DECONVOLUTION= S

PEAK NO.	MOLECULAR WT.	AMOUNT	Nanomoles/g(ml) Sample
2	188.70	0.43862	2.32443
5	223.10	1.36709	6.12770
6	223.10	0.01438	0.06446
7	223.10	0.03483	0.15612
8	223.10	0.16922	0.75849
10	257.50	0.22579	0.87685
14	249.00	0.09624	0.38651
15	257.50	0.04762	0.18493
16	257.50	0.11569	0.44928
17	257.50	0.06333	0.24594
19	267.90	0.00933	0.03483
21	257.50	0.04215	0.16369
22	257.50	0.03824	0.14850
23	257.50	0.05706	0.22159
24	257.50	0.07520	0.29204
25	259.50	0.07024	0.27067
26	258.70	0.03203	0.12381
27	292.00	0.00858	0.02938
29	292.00	0.01191	0.04079
31	292.00	0.12935	0.44298
32	292.00	0.13031	0.44627
33	292.00	0.14239	0.48764
37	292.00	0.03169	0.10853
38	272.40	0.04409	0.16186
39	292.00	0.04003	0.13709
41	326.40	0.01853	0.05677
42	292.00	0.00339	0.01161
43	298.90	0.01763	0.05898
44	298.90	0.01656	0.05540
45	292.00	0.00437	0.01497
46	292.00	0.01533	0.05250
48	293.50	0.04239	0.14443
49	324.70	0.05863	0.18057
50	292.00	0.01146	0.03926

53	326.40	0.22392	0.68603
54	326.40	0.09371	0.28710
55	326.40	0.01863	0.05708
56	326.40	0.01525	0.04672
57	326.40	0.02086	0.06391
58	326.40	0.02518	0.07714
59	326.40	0.02399	0.07350
60	360.90	0.09100	0.25215
61	315.80	0.14083	0.44595
62	360.90	0.02060	0.05708
63	326.40	0.00391	0.01198
64	360.90	0.16767	0.46459
65	350.50	0.06169	0.17601
67	336.80	0.11979	0.35567
69	337.50	0.74050	2.19407
71	347.80	0.01983	0.05702
72	336.80	0.01595	0.04736
73	360.90	0.10949	0.30338
74	347.80	0.07623	0.21918
75	360.90	0.63227	1.75193
77	360.90	0.06614	0.18326
80	360.90	0.05543	0.15359
81	395.30	0.01728	0.04371
82	360.90	0.50313	1.39410
83	360.90	0.03021	0.08371
84	360.90	0.00733	0.02031
85	395.30	0.06095	0.15419
87	395.30	0.01800	0.04554
88	395.30	0.29347	0.74240
89	360.90	0.02301	0.06376
90	395.30	0.15118	0.38244
91	360.90	0.02231	0.06182
92	394.30	0.03245	0.08230
93	394.30	0.24305	0.61641
94	394.30	0.14046	0.35623
95	382.20	0.07983	0.20887
97	360.90	0.02683	0.07434
98	395.30	0.00166	0.00420
99	429.80	0.01288	0.02997
100	395.30	0.04820	0.12193
102	395.30	0.50760	1.28409
103	395.30	0.01324	0.03349
104	395.30	0.00533	0.01348
105	429.80	0.01106	0.02573
106	395.30	0.19496	0.49320
107	395.30	0.04045	0.10233
108	429.80	0.00400	0.00931
109	429.80	0.09835	0.22883
110	429.80	0.11982	0.27878
111	395.30	0.00316	0.00799
112	429.80	0.04420	0.10284
115	429.80	0.08919	0.20752
116	429.80	0.00229	0.00533

NORTHEAST ANALYTICAL, INC.

301 NOTT STREET
SCHENECTADY, NY 12305
(518) 346-4592

CONGENER WEIGHT and MOLE REPORT

NEA FILE NAME: 89001.MOL

CUSTOMER: J. EPA
SAMPLE DESCRIPTION: SEDIMENT, SECTION 617
COMMENT: SAMPLE SHOWED ENVIRONMENTAL ALTERATION

TYPE FOR MIXED PEAK DECONVOLUTION= S

PEAK#	RET. TIME	T-CL:O-CL	IUPAC#	RRT	CONGENERS	WEIGHT %	MOLE %
2	17.48	1:1	001	.1544	2	4.716	7.543
5	19.89	2:2	004 010	.2245	22' ; 26	14.697	19.885
6	20.88	2:1	007 009	.2566	24 ; 25	0.155	0.209
7	21.24	2:1	006	.2709	23'	0.374	0.507
8	21.45	2:1	005 008	.2785	23 ; 24'	1.819	2.461
10	22.13	3:3	019	.3045	22'6	2.427	2.845
14	23.02	3:2 2:0	018 015	.3387	22'5 ; 44'	1.035	1.254
15	23.09	3:2	017	.3398	22'4	0.512	0.600
16	23.38	3:2	024 027	.3508	236 ; 23'6	1.244	1.458
17	23.66	3:2	016 032	.3625	22'3 ; 24'6	0.681	0.798
19	24.06	3:1 4:4	034 054	.3800	2'35 ; 22'66'	0.100	0.113
21	24.34	3:1	026	.3911	23'5	0.453	0.531
22	24.42	3:1	025	.3937	23'4	0.411	0.482
23	24.60	3:1	031	.4024	24'5	0.613	0.719
24	24.64	3:1 4:3	028 050	.4031	244' ; 22'46	0.808	0.948
25	24.99	3:1 4:3	021 033	.4170	233' ; 234 ; 22'56'	0.755	0.878
26	25.16	3:1 4:3	022 051	.4267	234' ; 22'46'	0.344	0.402
27	25.37	4:3	045	.4334	22'36	0.092	0.095
29	25.61	4:3	046	.4450	22'36'	0.128	0.132
31	25.86	4:2	052 073	.4554	22'55' ; 23'5'6	1.391	1.438
32	26.01	4:2	049	.4610	22'45	1.401	1.448
33	26.10	4:2	047	.4639	22'44'	1.531	1.582
37	26.49	5:4 4:2	104 044	.4832	22'466' ; 22'35'	0.341	0.352
38	26.60	3:0 4:2	037 042	.4870	344' ; 22'34' ; 233'6	0.474	0.525
39	26.89	4:2	064 071	.4990	23'34 ; 234'6 ; 23'4'6 +	0.430	0.445
41	27.03	5:4	096	.5057	22'366'	0.199	0.184
42	27.11	4:2	040	.5102	22'33'	0.036	0.038
43	27.31	5:3 4:1	103 057	.5155	22'45'6 ; 233'5	0.190	0.191
44	27.49	5:3 4:1	100 067	.5212	22'44'6 ; 23'4'5	0.178	0.180
45	27.59	4:1	058 063	.5267	233'5' ; 234'5	0.047	0.049
46	27.72	4:1 5:3	074 094	.5340	244'5 ; 22'356'	0.165	0.170
48	27.97	4:1 5:3	066 095	.5447	23'44' ; 22'356 ; 22'35'6	0.456	0.469
49	28.17	5:3 4:1	091 098	.5549	22'34'6 ; 22'3'46 ; 233'4	0.630	0.586
50	28.42	4:1	056 060	.5676	233'4' ; 2344'	0.123	0.127

53	28.82	5:2	101	090	.5814	22'34'5 ; 22'455'	2.407	2.226
54	28.98	5:2	099		.5880	22'44'5	1.007	0.932
55	29.23	6:4 5:2	150	112	.5969	22'34'66' ; 233'56 ; 23'44'6	0.200	0.185
56	29.31	5:2	083	109	.6029	22'33'5 ; 233'46	0.164	0.152
57	29.50	6:4 5:2	152	097	.6062	22'3566' ; 22'345 ; 22'3'45	0.224	0.207
58	29.66	5:2	087	111	.6175	22'345' ; 233'55' ; 2344'6	0.271	0.250
59	29.81	5:2	085	116	.6224	22'344' ; 23456?	0.258	0.239
60	29.92	6:4	136		.6257	22'33'66'	0.978	0.818
61	30.04	4:0 5:2	077	110	.6295	33'44' ; 233'4'6	1.514	1.447
62	30.30	6:3	154		.6349	22'44'56'	0.221	0.185
63	30.40	5:2	082		.6453	22'33'4	0.042	0.039
64	30.69	6:3	151		.6499	22'355'6	1.803	1.508
65	30.82	6:3 5:1	135	124	.6563	22'33'56' ; 2'344'5	0.663	0.571
67	30.96	5:1 6:3	107	108	.6628	233'4'5 ; 233'45' ; 22'34'56	1.288	1.154
69	31.17	6:3 5:1	149	118	.6672	22'34'5'6 ; 23'44'5 ; 233'45	7.961	7.120
71	31.58	6:3 5:1	134	143	.6796	22'33'56' ; 22'3456'; 2344'5	0.213	0.185
72	31.79	5:1 6:3	122	131	.6871	2'33'45; 22'33'46;22'33'55'+	0.171	0.154
73	32.09	6:2	146	161	.6955	22'34'55' ; 233'45'6	1.177	0.984
74	32.26	6:3 5:1	132	105	.7035	22'33'46' ; 233'44'	0.820	0.711
75	32.39	6:2	153		.7036	22'44'55'	6.797	5.685
77	32.97	6:2	141		.7203	22'3455'	0.711	0.595
80	33.36	6:2	137		.7329	22'344'5	0.596	0.498
81	33.44	7:4	176		.7305	22'33'466'	0.186	0.142
82	33.69	6:2	138	163	.7403	22'344'5' ; 233'4'56 ; +2	5.409	4.524
83	33.88	6:2	158		.7429	233'44'6	0.325	0.272
84	34.11	6:2	129		.7501	22'33'45	0.079	0.066
85	34.45	7:3	178		.7537	22'33'55'6	0.655	0.500
87	34.78	7:3	175		.7611	22'33'45'6	0.194	0.148
88	34.94	7:3	187	182	.7653	22'34'55'6 ; 22'344'56'	3.155	2.409
89	35.10	6:2	128		.7761	22'33'44'	0.247	0.207
90	35.27	7:3	183		.7720	22'344'5'6	1.625	1.241
91	35.57	6:1	167		.7814	23'44'55'	0.240	0.201
92	35.94	7:3	185		.7848	22'3455'6	0.349	0.267
93	36.34	7:3	174	181	.7965	22'33'456' ; 22'344'56	2.613	2.000
94	36.64	7:3	177		.8031	22'33'4'56	1.510	1.156
95	36.97	7:3 6:1	171	156	.8105	22'33'44'6 ; 233'44'5	0.858	0.678
97	37.24	6:1	157		.8184	233'44'5'	0.288	0.241
98	37.45	7:3	173		.8152	22'33'456	0.018	0.014
99	37.83	8:4	200	204	.8197	22'33'45'66' ; 22'344'566'	0.138	0.097
100	38.09	7:2	172	192	.8278	22'33'455' ; 233'455'6	0.518	0.396
102	38.62	7:2	180		.8362	22'344'55'	5.457	4.167
103	38.89	7:2	193		.8397	233'4'55'6	0.142	0.109
104	39.23	7:2	191		.8447	233'44'5'6	0.057	0.044
105	39.62	8:4	199		.8494	22'33'4566'	0.119	0.084
106	40.88	7:2	170		.8740	22'33'44'5	2.096	1.600
107	41.18	7:2	190		.8740	233'44'56	0.435	0.332
108	42.11	8:3	198		.8845	22'33'455'6	0.043	0.030
109	42.37	8:3	201		.8875	22'33'4'55'6	1.057	0.743
110	42.95	8:3	196	203	.8935	22'33'44'5'6 ; 22'344'55'6	1.288	0.905
111	44.24	7:1	189		.9142	233'44'55'	0.034	0.026
112	45.96	8:3	195		.9321	22'33'44'56	0.475	0.334
115	49.10	8:2	194		.9620	22'33'44'55'	0.959	0.673
116	50.10	8:2	205		.9678	233'44'55'6	0.025	0.017

CONCENTRATION = 9.302

TOTAL MICROMOLES = 0.0308

AVERAGE MOLECULAR WEIGHT = 301.8

NUMBER OF CALIBRATED PEAKS FOUND= 88

NORTHEAST ANALYTICAL, INC.

301 NOTT STREET
SCHENECTADY, NY 12305
(518) 346-4592

PCB SUMMARY REPORT

NEA FILE NAME: 89001.HOM

CUSTOMER: J. EPA
SAMPLE DESCRIPTION: SEDIMENT, SECTION 617
COMMENT: SAMPLE SHOWED ENVIRONMENTAL ALTERATION

Total PCBs in Sample= 9.30

PCB Homolog Distribution

Homolog Series	Percent in Sample
Mono	4.72
Di	17.30
Tri	9.31
Tetra	6.73
Penta	14.97
Hexa	23.29
Hepta	19.58
Octa	4.10
Nona	0.00
Deca	0.00

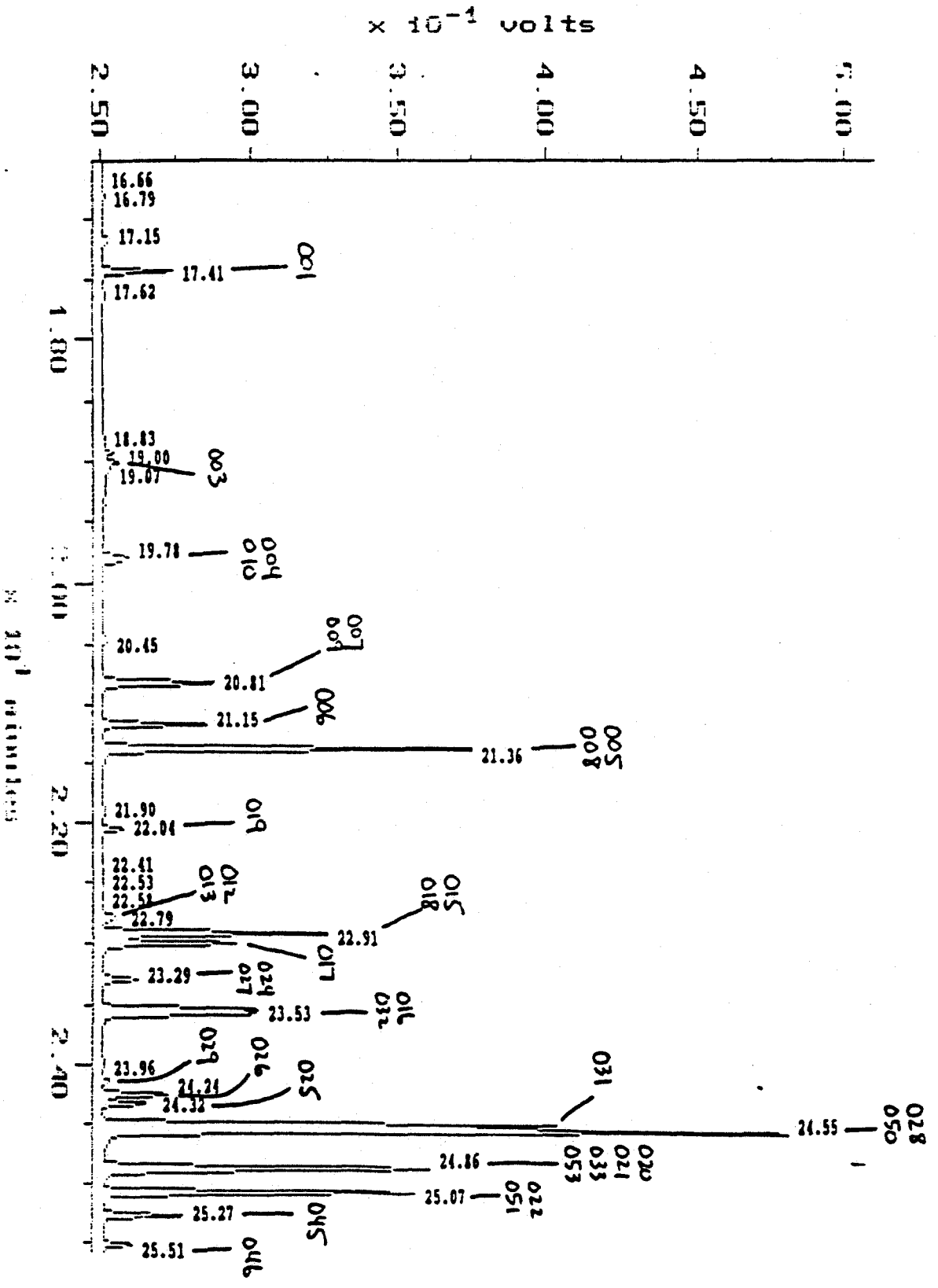
Ortho Cl / biphenyl Residue = 1.99

Meta + Para Cl / biphenyl Residue = 2.30

TOTAL Cl / biphenyl Residue = 4.29

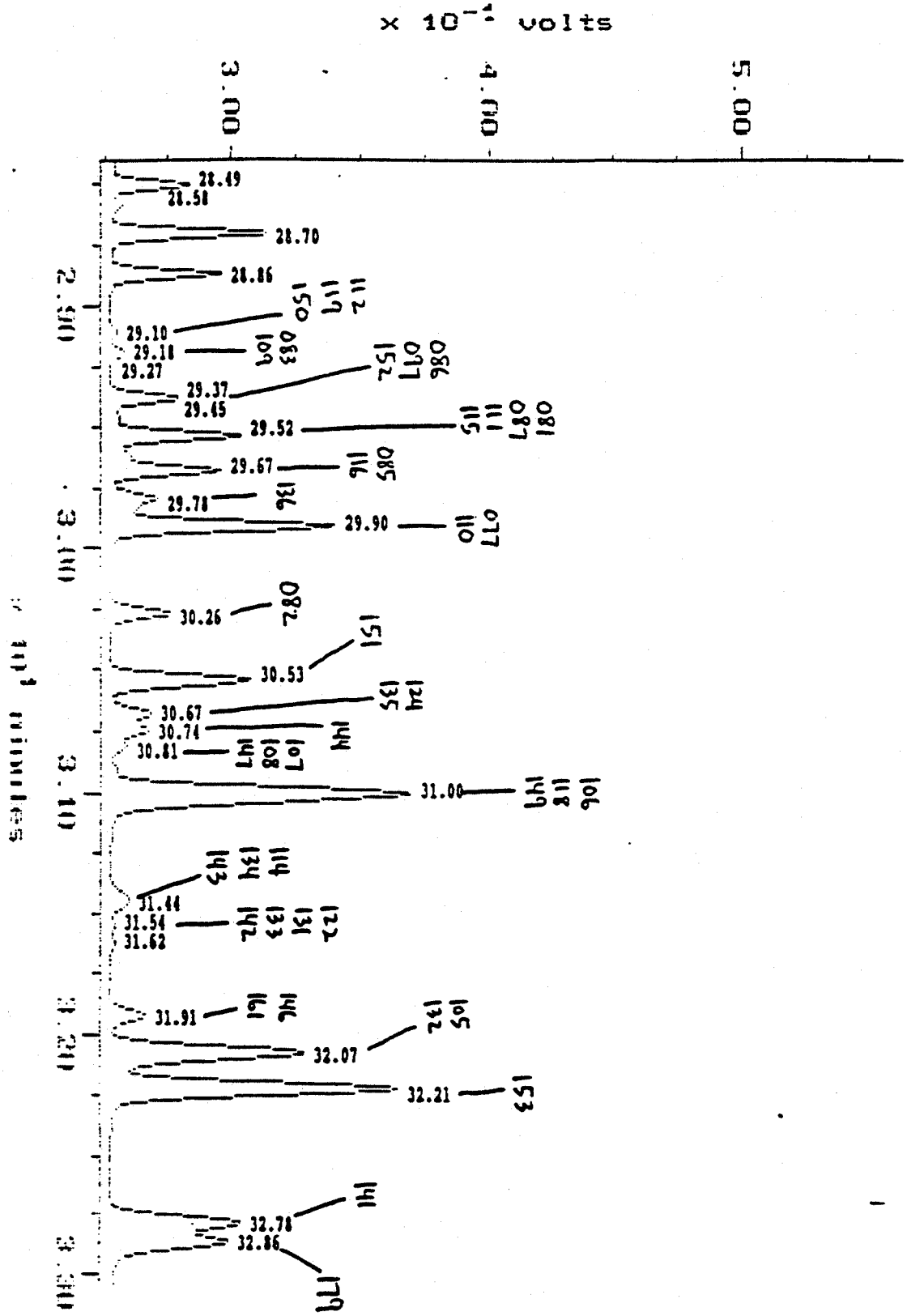
TABLE OF CONTENTS

Sample: 1232/1248/1262 Channel: detector 1 Filename: 1324862
Acquired: 27-JUN-89 13:11 Method: C:\MAX\GC1\1181 Operator: REM



312450

Sample: 1232/1248/1262 Channel: detector 1 Filename: A324862
Acquired: 27-JUN-89 13:11 Method: C:\MAX\GC1\1181A Operator: EW



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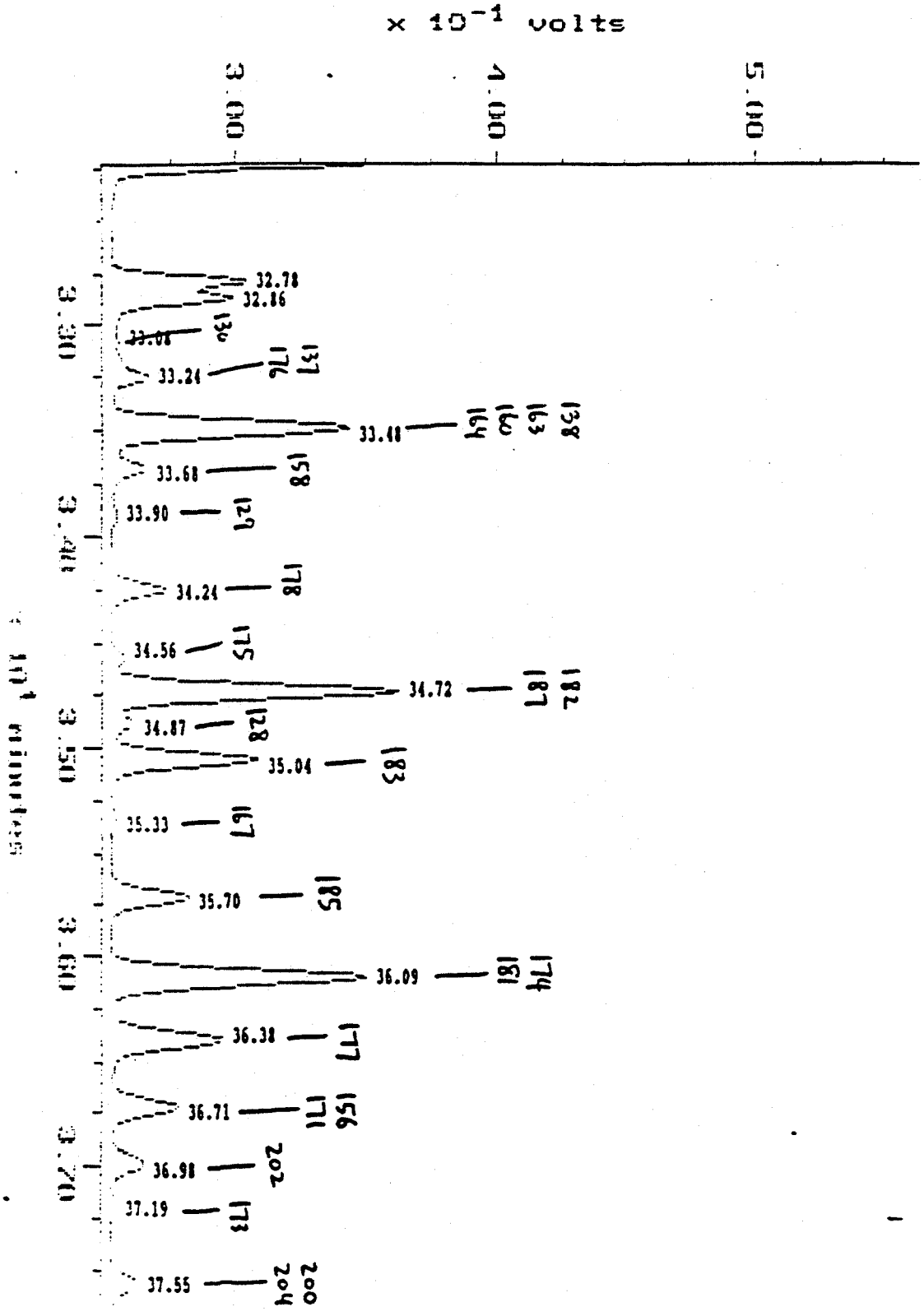
Acquired: 27-JUN-89 13:11 Method: C:\MAX\GC1\118A Operator: REW



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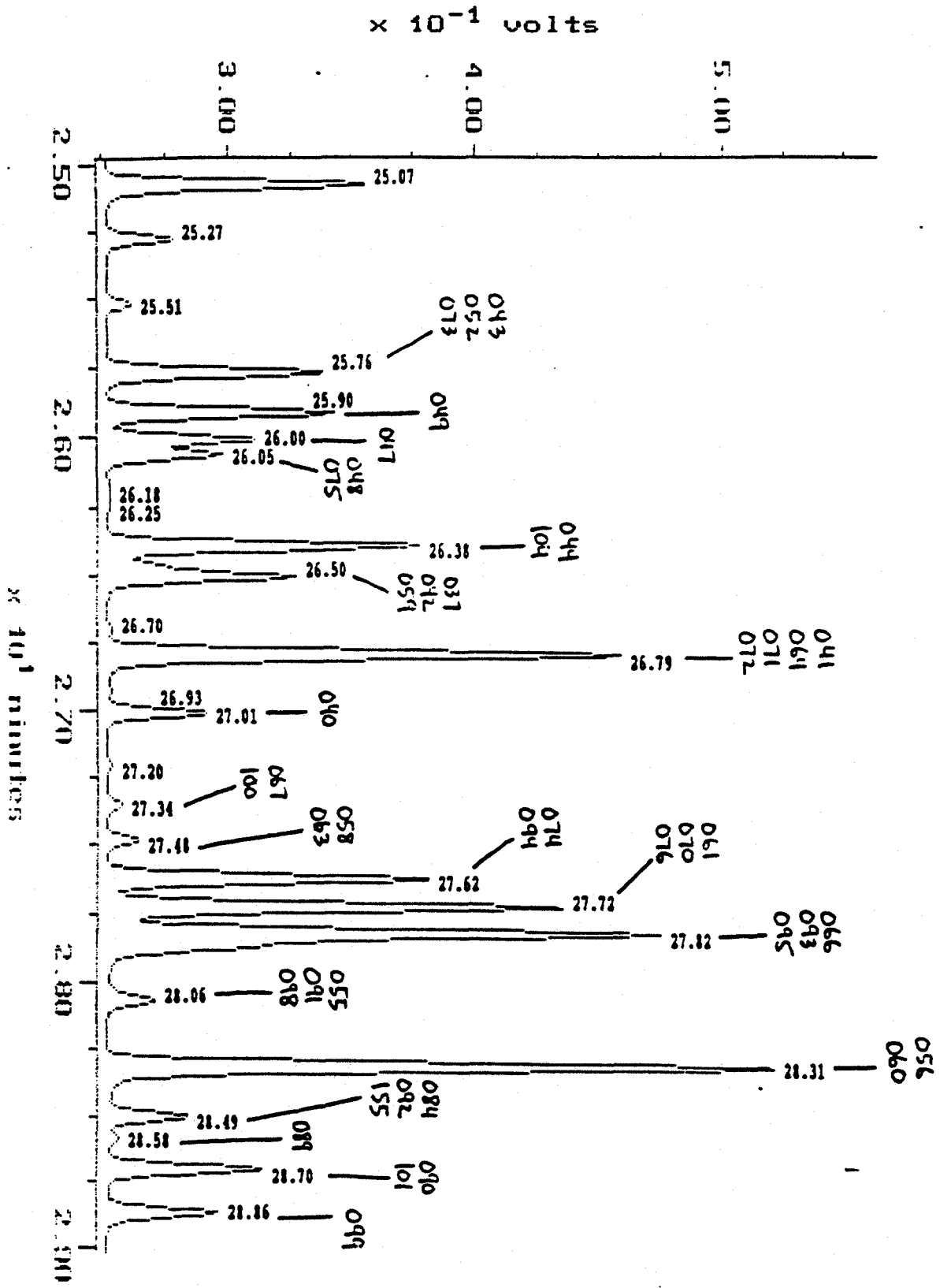
Acquired: 27-JUN-89 13:11 Method: C:\HAZ\GC1\118A Operator: RSW



312453

Sample: 1232/1248/1262 Channel: detector 1 Filename: A324862
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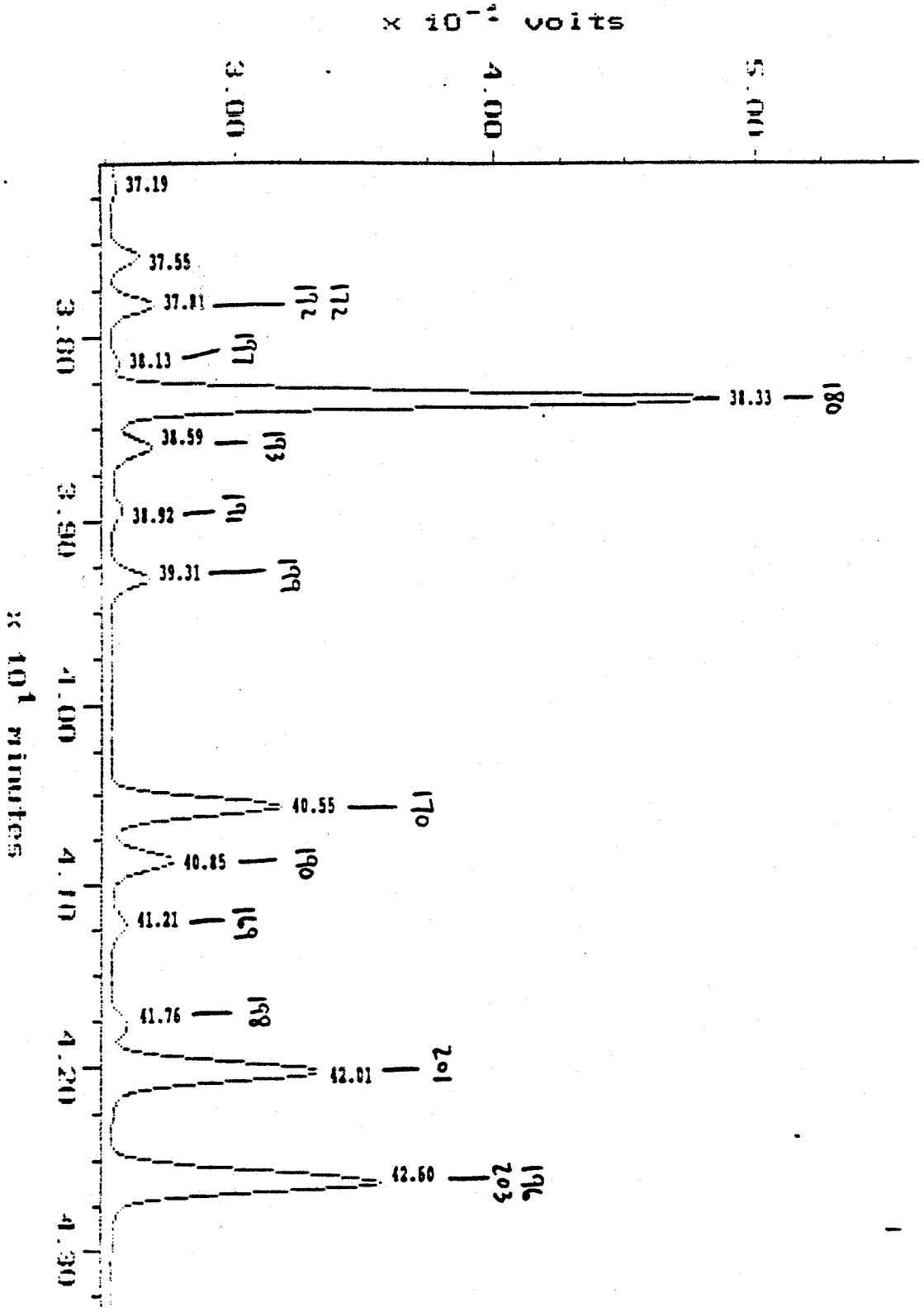

Sample: 1232/1248/1262 Channel: detector 1 Filename: A324162
Acquired: 27-JUN-89 13:11 Method: C:\MAX\GC1\1181A Operator: RBN