NEIC

EPA-330/2-82-001

83514

SUMMARY OF PINE RIVER RESERVOIR SEDIMENT SAMPLING SURVEY

St. Louis, Michigan
November 20-22, 1981

April 1982

National Enforcement Investigations Center, Denver

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INTRODUCTION

In June 1980* NEIC collected water and sediment samples from the Pine River to document the contamination contributed to the Pine River by previous operations of the Velsicol Chemical Corporation (VCC) at its St. Louis, Michigan facility.

At the request of Region 5, Enforcement Division, additional sediment sampling was conducted November 20 to 22, 1981 to supplement the June 1980 sampling and to more adequately define the areal and vertical distribution of total DDT, HBB, and PBB in the Pine River Reservoir. This new information was needed to support settlement negotiations between the regulatory agencies and VCC.

CONCLUSIONS

The NEIC sediment sampling data showed widespread contamination of the Pine River Reservoir by DDT, hexabromobenzene (HBB), and polybrominated biphenyl (PBB). An estimated 56,000 lb DDT, 10,000 lb HBB, and 800 lb PBB are contained in the Pine River Reservoir sediments.

The highest levels of contaminants were found in the middle basin (sampling Stations 04 to 18) of the reservoir located between the VCC Plant Site Jetty and the Mill Street Bridge [Figure 1]. Within this basin, the highest concentration of contaminants was found near the VCC plant site boundary, offshore from the former discharge points for the VCC organics production area. The maximum concentrations of total DDT, HBB, and PBB found in the segments of cores were 26,000 μg/g, 9,300 μg/g, and 330 μg/g, respectively. The lower basin (sampling Stations 19 to 23), located between

* *EPA-330/2-8-031 - Pine River Contamination Survey, St. Louis, Michigan - October 1980.*
EPA-330/2-8-030 - Investigation for Hazardous Waste Contamination, Velsicol Chemical Corporation Plant Site, St. Louis, Michigan - October 1980.

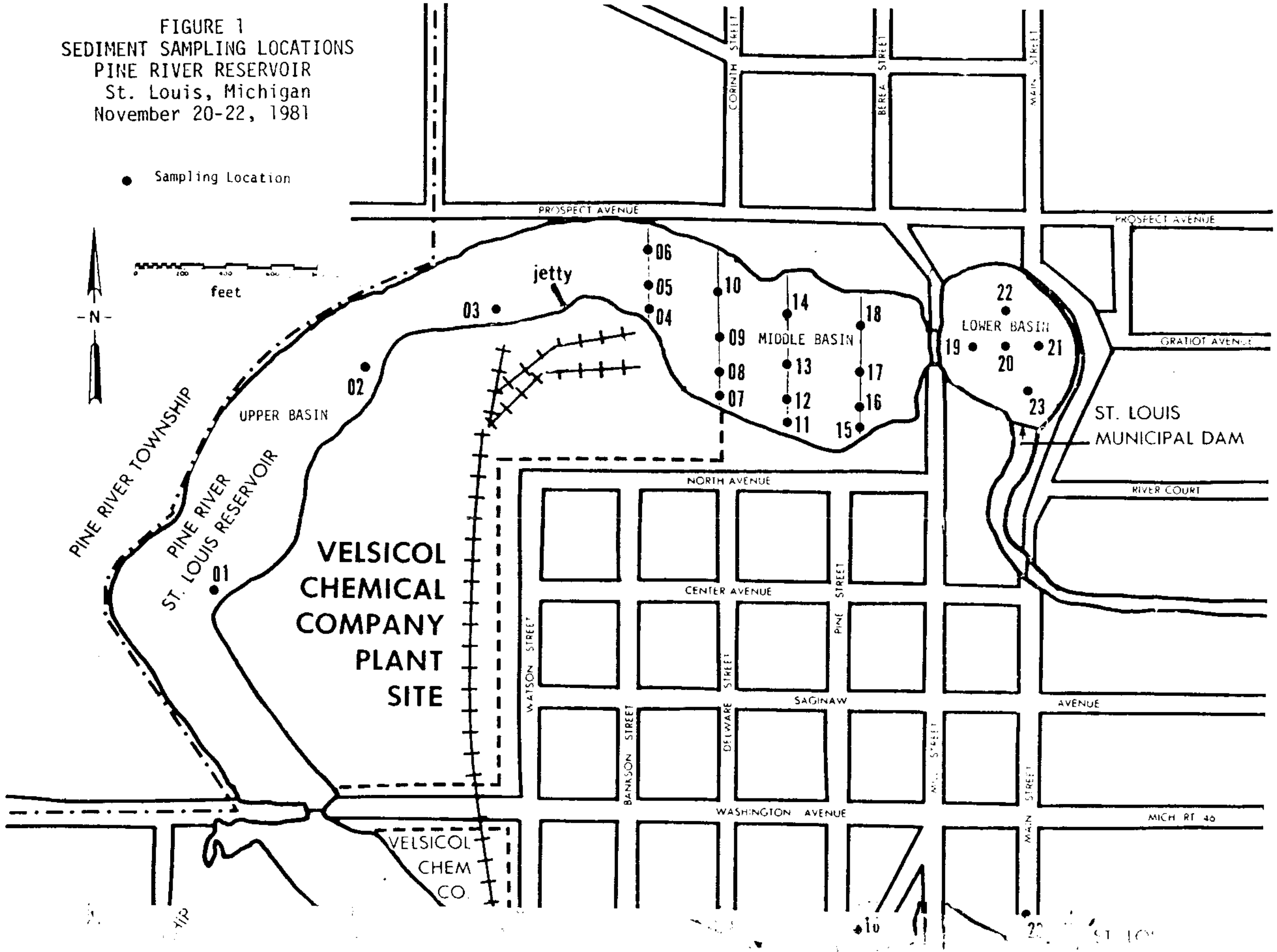

FIGURE 1
SEDIMENT SAMPLING LOCATIONS
PINE RIVER RESERVOIR
St. Louis, Michigan
November 20-22, 1981

the Mill Street Bridge and the St. Louis Dam, was found to be contaminated, but to a lesser degree than the middle basin. Maximum concentrations of total DDT, HBB, and PBB found in this area were 560 μg/g, 180 μg/g, and 5.2 μg/g, respectively. In the upper basin (sampling Stations 01, 02, and 03), located upstream of the VCC plant site jetty, the contaminant levels were lower with maximum total DDT, HBB, and PBB concentrations of 25 μg/g, 3.2 μg/g, and 2.8 μg/g, respectively.

Regardless of the areal distribution of the contaminants, the highest DDT levels were found in the deeper portions of the sediment cores with lower concentrations in the top 4 inches. High levels of DDT are also found in surface sediments (4,000 μg/g at Station 14) in areas of the reservoir where scouring had apparently exposed the older sediments. The highest levels of HBB and PBB were found in the top (0 to 16 in.) of the cores. Logically, the HBB and PBB should be more prevalent in the surface sediments, and DDT should be prevalent in the deeper sediments because HBB and PBB were produced until the mid-1970s, while DDT production ceased in 1959.

METHODS

Sediment cores were collected and water and sediment depths measured at 23 sampling locations [Figure 1, Table 1]. The sediment depths were measured by penetrating the reservoir bottom with a ½ in. diameter steel rod until resistance was met. Water depth was obtained by dropping a disc (approximately 16 in. diameter) into the reservoir bottom and measuring the submerged portion of the attached cord.

The sediment samples were collected in 1-3/4 in. I.D. hexane-rinsed aluminum or galvanized steel core tubes. To facilitate sampling and handling, core tubes of 3, 4, or 6 ft in length were used. The samples were collected by slowly pushing or driving the core tubes into the sediment until a sediment layer, which was compacted hard enough to plug the end of the core tube, was reached. Care was taken to allow several inches of water to

Table 1

SEDIMENT SAMPLING LOCATIONS
PINE RIVER RESERVOIR
ST. LOUIS, MICHIGAN
November 20-22, 1981

Core Sampling	Sampling Date & Time (ft)	Depth of Water (ft)	Depth of Sediment (ft)	Core Tube Length	Station Description
01	11/20/81 1315 hrs	8	1.5	3	50 feet from the shoreline adjacent to the Velsicol Chemical Corporation (VCC) plant site, 2100 feet upstream of the VCC jetty.
02	11/20/81 1340 hrs	2	1.5	3	Same as above (01) except 900 feet upstream of the VCC jetty.
03	11/20/81 1420 hrs	4	9	4	Same as above (01) except 300 feet upstream of the VCC jetty.
04	11/20/81 1530 hrs	1	8	4	50 feet from the shoreline adjacent to the VCC plant site on the north-south transect located 1200 feet west of the Mill Street Bridge.
05	11/20/81 1545 hrs	1.5	9	6	Same as above (04) except 150 feet from the VCC shoreline.
06	11/20/81 1600 hrs	5	9	6	Same as above (04) except 300 feet from the VCC shoreline.
07	11/21/81 1125 hrs	4	6	4	50 feet from the shoreline adjacent to the VCC plant site on the north-south transect located 900 feet west of the Mill Street Bridge.
08	11/21/81 1140 hrs	3	3	3	Same as above (07) except 150 feet from the VCC shoreline.
09	11/21/81 1200 hrs	3	4.5	4	Same as above (07) except 300 feet from the VCC shoreline.
10	11/21/81 1215 hrs	3	7	6	Same as above (07) except 500 feet from the VCC shoreline
11	11/21/81 1230 hrs	5.5	4	4	50 feet from the shoreline adjacent to the VCC plant site on the north-south transect located 600 feet west of the Mill Street Bridge.
12	11/21/81 1245 hrs	5	8	6	Same as above (11) except 150 feet from the VCC shoreline.
13	11/21/81 1300 hrs	4	8	6	Same as above (11) except 300 feet from the VCC shoreline.
14	11/21/81 1335 hrs	4	2	4	Same as above (11) except 500 feet from the VCC shoreline.
15	11/21/81 1430 hrs	7	7	6	50 feet from the shoreline adjacent to the VCC plant site on the north-south transect located 300 feet west of the Mill Street Bridge.
16	11/21/81 1445 hrs	5	8	6	Same as above (15) except 150 feet from the VCC shoreline.
17	11/21/81 1500 hrs	5	8	6	Same as above (15) except 300 feet from the VCC shoreline.
18	11/21/81 1520 hrs	7	3	4	Same as above (15) except 500 feet from the VCC shoreline.
18-dup	11/21/81 1520 hrs	7	3	4	Same as above (18)
19	11/22/81 0930 hrs	8	2	3	150 feet east of the midpoint of the Mill Street Bridge.
20	11/22/81 0945 hrs	8	2	3	300 feet east of the midpoint of the Mill Street Bridge.
21	11/22/81 1000 hrs	7	4	3	450 feet east of the midpoint of the Mill Street Bridge.
21 dup	11/22/81 1000 hrs	7	4	3	Same as above (21)
22	11/22/81 1015 hrs	8	6	3	150 feet north of Station 20.
23	11/22/81 1020 hrs	8	3	3	150 feet north of the midpoint of the City of St. Louis municipal dam.

remain in the top of the core tubes to ensure that there was minimal disturbance of the surface sediment. The filled tubes were extracted from the bottom, the ends were sealed with aluminum foil, and then they were capped and frozen in an upright position.

After freezing, the 4 and 6 ft core tubes were cut in half, the open ends covered with aluminum foil, and stored in dry ice. At the NEIC laboratory, the frozen samples were extruded from the core tubes by warming the outside of the tubes with water and pushing the frozen cores from the tubes. After extrusion, each frozen core was observed and the physical characteristics recorded [Table 2]. The top 4 inches of each sediment sample was separated from the core and the remainder divided, from top to bottom, in 1 ft intervals or fractions of a foot, as applicable. All segments of each sediment core were wrapped in aluminum foil. These segments were subsequently thawed, thoroughly mixed, and analyzed for PBB, total DDT, and HBB. Complete analytical methodology is shown in Attachment A. NEIC chain-of-custody and document control procedures were followed throughout the survey.

Table 2

SEDIMENT CORE DESCRIPTIONS
PINE RIVER RESERVOIR
St. Louis, Michigan
November 20-22, 1981

Station Number	Extrusion Date and Time	Total Length (in.)	Core Segment (in.)	Visual Description*
01	12/1/81 1200 hrs	13	0-3	gray-brown flock grading into gray-brown silt
			3-10	gray-brown silt
			10-13	gray-black sludge
02	11/30/81 1400 hrs	17	0-4	brown sandy silt
			8-14	white silt (MgO_2)
			14-17	black sludge
03	12/1/81 0955 hrs	24	0-6	black sludge
			6-10	gray-white silt (MgO_2)
			10-24	white silt (MgO_2)
04	11/30/81 1425 hrs	13	0-9	dark gray-brown sandy silt
			9-13	black silt
05	11/30/81 1335 hrs	40	0-4	brown-gray floc grading into gray-brown silt
			4-13	gray-black sludge
			13-15	white silt (MgO_2)
			15-40	gray-black sludge
06	11/30/81 1440 hrs	32	0-4	gray-brown floc grading into gray-brown sandy silt
			4-13	gray sandy silt
			13-32	gray-white silt
07	11/30/81 1450 hrs	36	0-6	brown floc
			6-20	black silty sand
			20-36	gray-black sandy silt
08	11/30/81 1325 hrs	28	0-4	brown-gray floc grading into brown-gray silt
			4-28	gray-black silt
09	11/30/81 1315 hrs	34	0-34	gray-black silt
10	11/30/81 1500 hrs	24	0-8	gray sand with streaks of white silt (MgO)
			8-14	gray sand
			14-24	black sandy silt
11	11/30/81 1310 hrs	16	0-4	gray-brown floc grading into gray-black silt
			4-16	gray-black silt
12	11/30/81 1520 hrs	30	0-3	gray-brown floc grading into gray-black silt
			3-30	gray-black silt
13	11/30/81 1300 hrs	46	0-6	gray-brown floc grading into silty gray-black
			6-46	gray-black silt
14	12/1/81 0830 hrs	28	0-4	gray-brown floc grading into gray-black silty sand
			4-8	black silty sand
			8-28	gray-brown coarse sand
15	12/1/81 0915 hrs	46	0-2	black floc
			2-46	dark black sludge

Table 2 (Cont.)

SEDIMENT CORE DESCRIPTIONS
PINE RIVER RESERVOIR
St. Louis, Michigan
November 20-22, 1981

Station Number	Extrusion Date and Time	Total Length (in.)	Core Segment (in.)	Visual Description*
16	11/30/81 1250 hrs	42	0-12	gray-brown silt
			12-42	black sludge
17	12/1/81 0935 hrs	45	0-4	brown floc
			4-9	black and gray-white silt layers
			9-40	black silt
			40-45	brown-gray clay
18	11/30/81 1220 hrs	35	0-11	gray-brown floc grading into fine gray-brown silt
			11-35	black silt
18-dup	11/30/81 1240 hrs	34	0-11	gray-brown floc grading into fine gray brown silt
			11-34	black silt
19	12/1/81 1015 hrs	22	0-3	brown-gray floc grading into black silt
			3-18	black sludge
			18-22	fine gray-brown silt
20	11/30/81 1345 hrs	27	0-5	gray-black silt
			5-19	black sludge
			19-27	light brown clay
21	12/1/81 1035 hrs	21	0-5	brown-gray floc grading into black silt
			5-13	black silt
			13-21	gray-black silt
21 dup	12/1/81 1135 hrs	14	0-4	brown-gray floc grading into black silt
			4-14	black silt
22	12/1/81 1145 hrs	23	0-6	brown-gray floc grading into black silt
			6-18	black silt
			18-23	gray-black silt
23	12/1/81 1200 hrs	34	0-3	gray-brown floc
			3-28	black silt
			28-34	gray-brown silty sand

* Descriptions are from visual observations of the outside of the frozen cores after extrusion from the core tubes.

SURVEY FINDINGS

PHYSICAL OBSERVATIONS

Total sediment depth at the sampling Stations varied between 1.5 and 9 ft [Table 1]. The deepest sediments (8 to 9 ft) were found in the extreme upper end (Stations 04, 05, and 06) and lower end (Stations 12, 13, 16, and 17) of the middle basin.

After extrusion of the sediment cores, the physical characteristics of the sediment cores were observed and recorded [Table 2]. Many of the cores (78%) contained a surface layer (1 to 6 inches) of gray-brown floc (light silty filamentous material). Nearly all the cores contained layers of varying thickness of black or gray-black sludge. A sulfurous odor was noticed in the cores which contained this black sludge. The strongest odors were from samples collected at Stations 07, 08, 15, 19, 20, and 23. Many of the cores contained layers of white sludge which, according to Company officials, is probably magnesium oxide (MgO). Both the black and white layers had a very small grain size, usually indiscernible to the naked eye.

The black and white layers of materials seen in the cores varied in thickness and depth; hence, no visual correlation of the layers between sampling stations could be made. These varying depths and thicknesses of sediment layers and the differences in total sediment depths at the sampling stations showed the expected variability in deposition rates within the reservoir.

ANALYTICAL RESULTS

Approximately 56,000 lbs DDT, 10,000 lbs HBB, and 800 lbs PBB are contained in the Pine River sediments. Less than 5% of the DDT is found in the top 4 in. of sediment, but over 13% of the HBB and 57% of the PBB was found in the top 4 in. of the cores. These loadings were calculated by integrating sediment density, contaminant concentration by depth, and the areas assigned to each of the sampling Stations [Appendix A].

The highest concentrations of total DDT, HBB, and PBB [Table 3] were found in the middle basin of the Pine River Reservoir (Stations 04-18) which extends from the VCC plant site jetty to the Mill Street Bridge [Figure 1]. Average maximum total DDT, HBB, and PBB concentrations* in the samples collected in this basin were 3,500 μg/g, 1,000 μg/g, and 42 μg/g, respectively. Wastewater from the former VCC organics production area was discharged to this portion of the basin. Within this basin, the highest total DDT concentrations were found at Stations 07 (26,000 μg/g) and 08 (8,800 μg/g). The highest concentrations of HBB were also found at Station 07 (9,300 μg/g) and 08 (2,600 μg/g). Highest PBB concentrations were found at Station 04 (330 μg/g), 10 (66 μg/g), 07 (64 μg/g), and 08 (58 μg/g). Stations 07 and 08 are the Stations nearest to the former organics wastewater discharges, and they are close to the location where the highest total DDT concentration (44,000 μg/g) was found during the June 1980 NEIC survey.** Contaminant concentrations within the middle basin generally decreased [Figures 2, 3, and 4] toward the northern shore. The old river channel is located along this shoreline.

Compared to the middle basin, the average concentrations of contaminants in the upper basin are low. Average maximum core concentrations of total DDT, HBB, and PBB of 11 μg/g, 1.8 μg/g, and 1.5 μg/g, respectively, were found in this portion of the reservoir which extends from the Washington Street Bridge to the VCC plant site jetty (Stations 01, 02, and 03).

The lower basin (Stations 19 to 23) of the reservoir between the Mill Street Bridge and the St. Louis Municipal Dam was found to be contaminated; however, contaminant concentrations were lower than the levels found in the middle basin. Average maximum core concentrations of total DDT, HBB, and PBB found in this area were 310 μg/g, 81 μg/g, and 4.2 μg/g, respectively.

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* *The average maximum concentrations were calculated by averaging the concentrations of the most contaminated segment of each core.*

** *EPA-330/2-80-031 - Pine River Contamination Survey, St. Louis, Michigan - October 1980.*

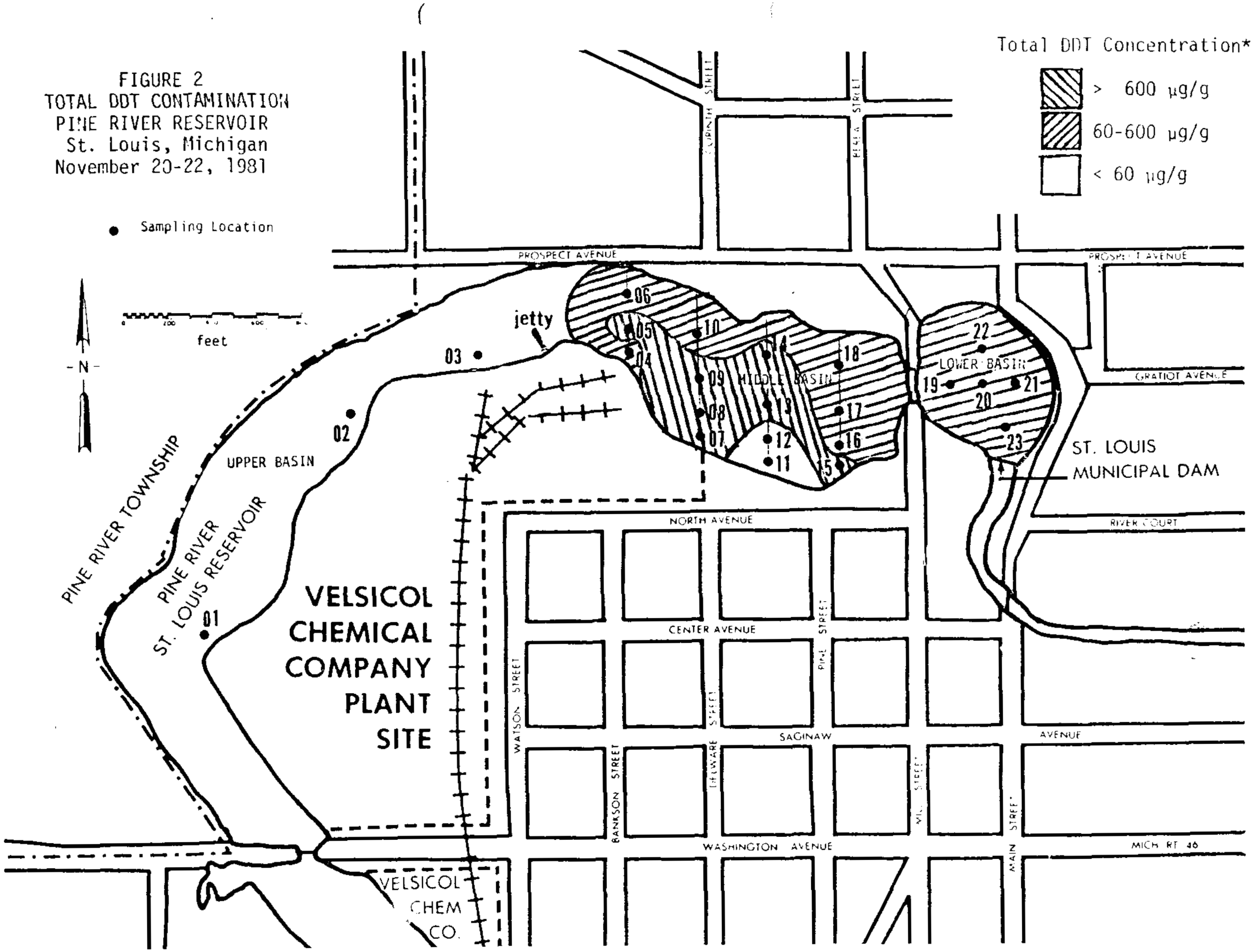
Table 3

TOTAL DDT, HBB, AND PBB CONCENTRATIONS
IN SEDIMENTS BY BASIN
PINE RIVER RESERVOIR
St. Louis, Michigan
November 20-22, 1981

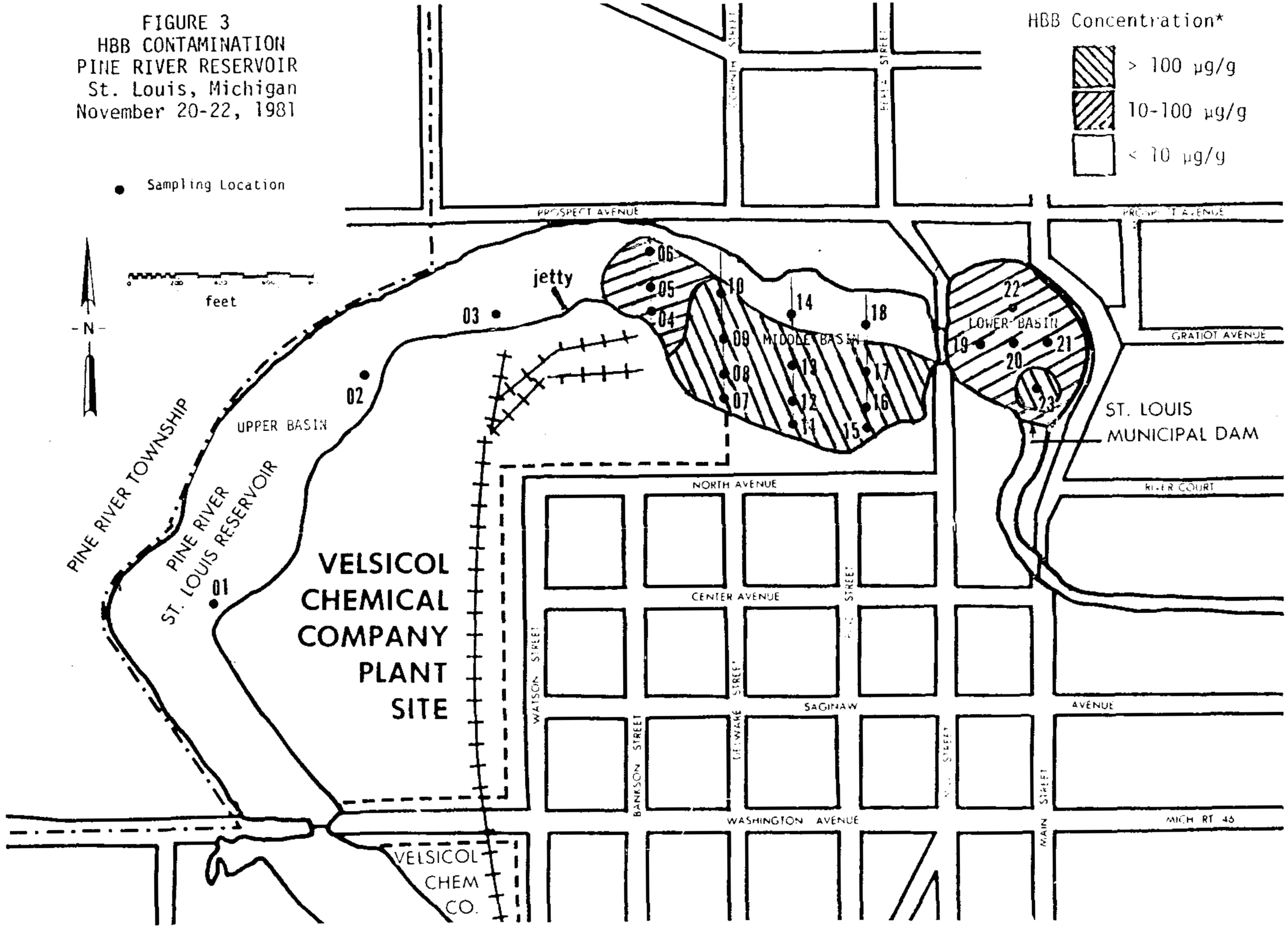
Basin	Sample Size Stations	Total DDT		HBB		PBB	
		Avg.- Maximum Concentration µg/g	Maximum Concentration µg/g	Avg.- Maximum Concentration µg/g	Maximum Concentration µg/g	Avg.- Maximum Concentration µg/g	Maximum Concentration µg/g
UPPER Stations 01-03	3	11	25	1.8	3.2	1.5	2.8
MIDDLE Stations 04-18	15	3500	26,000	1000	9300	42	330
LOWER Stations 19-23	5	300	560	81	180	4.2	5.2

* This average maximum concentration was calculated by averaging the concentrations of the most contaminated segment of each core.

FIGURE 2
TOTAL DDT CONTAMINATION
PINE RIVER RESERVOIR
St. Louis, Michigan
November 20-22, 1981

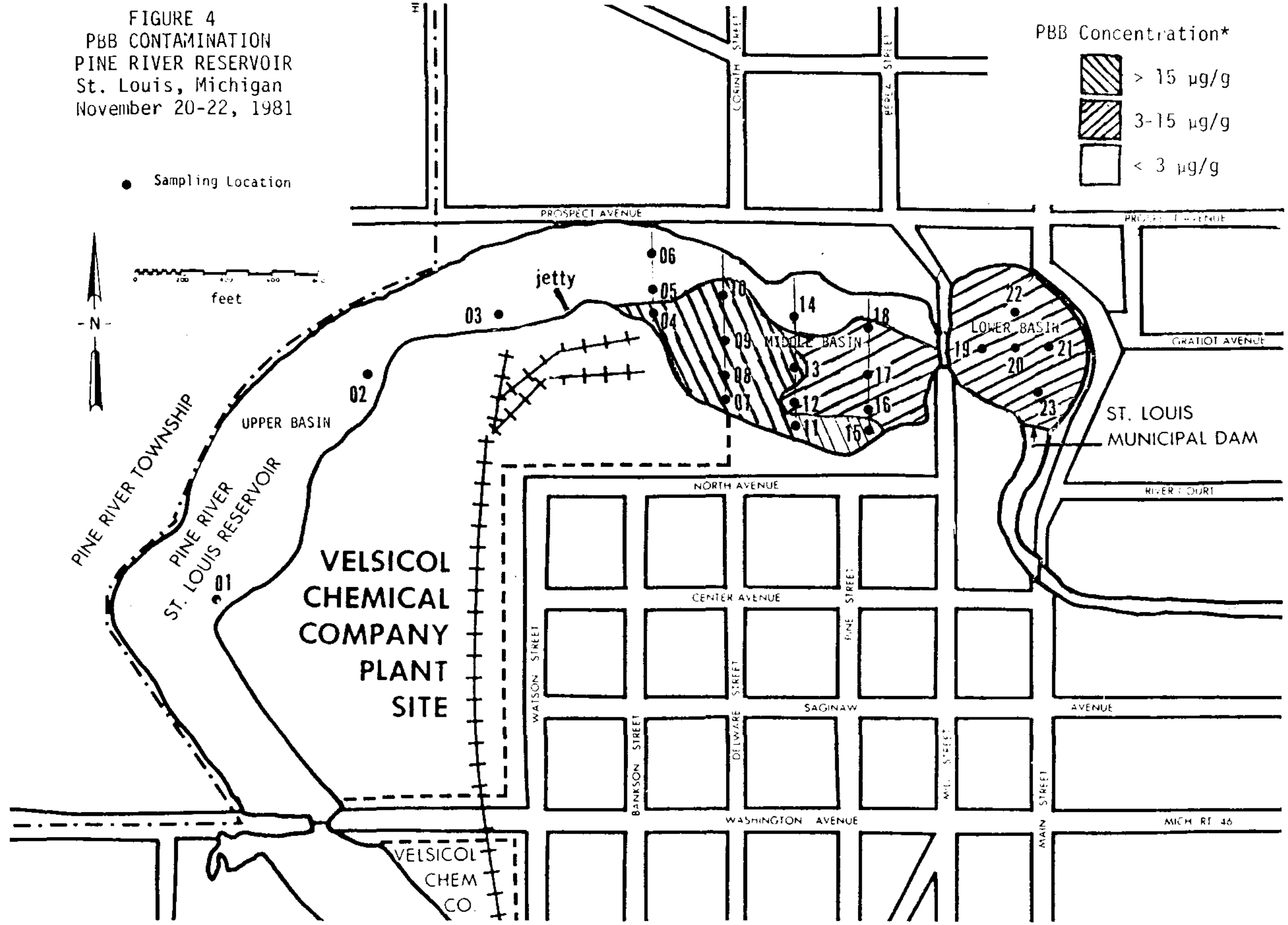


* Based on maximum concentrations found in individual sediment cores



* Based on maximum concentrations found in individual sediment cores

FIGURE 4
PBB CONTAMINATION
PINE RIVER RESERVOIR
St. Louis, Michigan
November 20-22, 1981



* Based on maximum concentrations found in individual sediment cores

A concentration profile by depth [Table 4] was developed by summarizing the data from each of 5 specified depths (0 to 4 in., 4 to 16 in., 16 to 28 in., 28 to 40 in., and 40 in. and below) from the 20 sampling Stations in the middle and lower basins (Stations 04 to 23).* This profile showed the highest average total DDT concentrations were found to be in the deeper sediment layers (16 to 28 in. and 28 to 40 in. layers). An average total DDT concentration of 960 µg/g was found in the 16 to 28 in. segments of the sediment cores, and 1,000 µg/g was found in the 18 to 40 in. segments.

Conversely, lower concentrations of total DDT were generally found in the surface portion (0 to 4 in.) of the sediment cores. One station (14), however, had a surface sediment total DDT concentration of 4,000 µg/g. This high concentration is probably due to the deposition and erosion patterns in the reservoir. Station 14 is located at the edge of the main river channel which runs along the north side of the reservoir. The velocity of the currents would be expected to be higher in this area of the reservoir during high flow periods, increasing scouring and exposing the older and deeper layers of sediment which contain higher total DDT levels. The analyses of the three cores which were greater than 40 in. in length showed very low concentrations of contaminants below the 40 in. level.

Maximum HBB and PBB concentrations were generally found to be in the upper sediments (0 to 16 in.). A maximum HBB concentration of 9,300 µg/g was found in the 4 to 16 in. layer at Station 07, and a maximum PBB concentration of 330 µg/g was found in the 0 to 4 in. layer at Station 04.

Complete analytical data for all the sediment samples, as well as the analytical methods and associated quality control data, can be seen in Appendix B.

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* *Sediment cores were divided into two to five segments depending on each core's length.*

Table 4

TOTAL DDT, HBB, AND PBB CONCENTRATIONS[1]
IN SEDIMENTS BY BASIN
PINE RIVER RESERVOIR
St. Louis, Michigan
November 20-22, 1981

Core Seg. Depth (in.)	Sample Size Stations	Total DDT		HBB		PBB	
		Avg.[2]-Maximum Concentration µg/g	Maximum Concentration µg/g	Avg.[2]-Maximum Concentration µg/g	Maximum Concentration µg/g	Avg.[2]-Maximum Concentration µg/g	Maximum Concentration µg/g
0-4	19	34	4,000	61	2,600	8.6	330
4-16	19	159	2,900	255	9,300	7.4	66
16-28	17	957	26,000	5.8	100	0	1
28-40	9[3]	1014	4,800	0.13	44	0	0
40-46	3	3.3	9.5	0	0	0	0

1 Dry weight concentrations
2 This average was derived by comparing the mean concentration of the entire set (Stations 04-23) at the various depths with individual values, dismissing values which were more than 2 standard deviations from the mean, and calculating a new average with the remaining values. This average is not the true average but an expected average with depth. The high values dismissed are valid concentrations although not typical for the majority of the cores.
3 A sample size of 8 was used to derive the average concentrations for HBB.

APPENDIX A

TOTAL DDT, HBB, AND PBB LOADING APPROXIMATIONS

TOTAL DDT, HBB, AND PBB LOADING APPROXIMATIONS*
PINE RIVER RESERVOIR
St. Louis, Michigan

Sample Number*	% H_2O	Sediment Density lbs/ft^3	Area ft^3	Total DDT Loading lbs	HBB Loading lbs	PBB Loading lbs
01-1	64.3	80.6	-	-	-	-
01-2	63.9	81.0	-	-	-	-
02-1	55.4	87.3	185,760	12.1	2.41	2.41
02-2	48.2	93.6	"	63.0	9.91	6.84
03-1	64.9	80.1	216,000	3.44	1.94	1.70
03-2	64.1	82.9	"	125	-	-
03-3	51.8	90.2	"	235	-	-
04-1	34.8	107.2	54,000	340	18.9	415
04-2	45.2	96.5	"	343	257	65.7
05-1	63.2	81.3	36,000	5.03	33.0	0.34
05-2	65.3	79.9	"	24.0	6.19	-
05-3	68.1	78.1	"	888	-	-
05-4	61.6	82.4	"	-	-	-
06-1	57.1	85.8	54,000	1.71	7.95	1.46
06-2	43.7	98.8	"	10.5	84.1	1.68
06-3	60.5	83.3	"	13.9	1.63	-
06-4	51.9	90.2	"	-	-	-
07-1	72.0	75.8	18,000	12.7	-	1.78
07-2	71.5	76.0	"	148	3626	25.0
07-3	61.3	82.7	"	14980	57.6	-
07-4	63.4	81.2	"	2.51	0.53	-
08-1	70.3	76.7	50,400	13.8	994	22.2
08-2	70.8	76.5	"	3265	2027	-
08-3	63.2	81.3	"	14940	-	-
09-1	65.6	79.8	54,000	16.3	69.2	8.90
09-2	68.3	78.0	"	801	32	24
09-3	59.6	84.0	"	143	-	-
09-4	62.4	81.8	"	9.13	-	-
10-1	68.0	78.2	57,600	2.79	26.4	6.73
10-2	64.1	80.7	"	751	734	110
10-3	56.3	86.6	"	17.0	30.5	-
11-1	68.5	77.9	25,200	3.5	20.0	1.0
11-2	65.6	79.8	"	22.8	422	14.5
12-1	68.7	77.8	32,400	3.94	23.7	1.31
12-2	63.5	81.2	"	15.4	163	7.68
12-3	55.9	86.9	"	-	-	-
13-1	67.1	78.7	61,200	7.92	23.2	2.22
13-2	67.7	78.3	"	24.8	774	38.7
13-3	67.4	78.5	"	345	43.9	1.57
13-4	62.2	82.0	"	9105	-	-
13-5	55.2	87.4	"	18	-	-
14-1	66.5	79.1	64,000	2289	3.26	1.55
14-2	13.5	140.0	"	243	-	-
14-3	15.1	137.0	"	-	-	-
15-1	71.9	75.8	12,960	1.93	7.36	0.37
15-2	68.9	77.7	"	6.58	207	5.01
15-3	67.1	78.7	"	1804	18	-
15-4	58.8	84.5	"	1940	19.9	-

APPENDIX B

COMPLETE ANALYTICAL DATA AND METHODS
AND ASSOCIATED QUALITY CONTROL DATA

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
BUILDING 53, BOX 25227, DENVER FEDERAL CENTER
DENVER, COLORADO 80225

TO Russell Forba, Project Coordinator

DATE January 14, 1982

FROM Charles P. Rzeszutko, Chemist
THRU: Dean F. Hill, Chief
Pesticide and Toxic Substances Branch

SUBJECT Project #624, Pine River, St. Louis, MI

Attached you will find the results of analysis for all core samples received for Project #624. All cores were analyzed for DDT and related compounds, hexabromobenzene (HBB), and polybrominated biphenyls (PBB). Methodology and quality control data are provided. Results previously reported on December 16, 1981, are included in this report.

If you have any questions, please contact me at your convenience.

cc: R. Laidlaw
T. Meiggs
Attachments

All samples were screened for compounds of interest by packed column electron-capture gas chromatography. Based on the screening results, high-level samples were simply diluted, whereas low-level samples were cleaned up by column chromatography. All samples requiring dilutions were diluted to the appropriate levels with hexane. All samples that did not require a dilution were cleaned up using Florisil column chromatography. The sample extract was placed on the head of a glass column packed with 20 grams of activated Florisil and eluted with 200 mℓs of 6% ethyl ether in hexane. The fraction was concentrated to less than 25 mℓs using a Kuderna-Danish evaporation apparatus and further concentrated to an appropriate volume using a nitrogen blowdown apparatus.

All sample extracts were analyzed by fused silica capillary column electron-capture gas chromatography using a 15 meter DB-5 column and a 25 meter OV-101 column. All extracts were analyzed for (1) Total DDT's, which included o,p'-DDE, p,p'-DDE, o,p'-DDD, p,p'-DDD, o,p'-DDT, p,p'-DDT; (2) hexabromobenzene (HBB); and (3) polybrominated biphenyls (PBB).

Several of the sample extracts were also analyzed for these compounds by gas chromatography/mass-spectroscopy (GC/MS).

NOTE: The following samples were extracted in the same manner as described above with the exception that a 10g sample size was weighed-out with 5 g of anhydrous Na_2SO_4 and extracted with 20 mℓs of 50% acetone in hexane for the first extraction: 09-1, 09-2, 09-3, 11-1, 11-2, 13-1, 13-2, 13-3, 16-1, 16-2, 16-3, 18-1, 18-2, 18-3, 18-1 Dup., 18-2 Dup., and 18-3 Dup.

RESULTS

The results of analysis by fused silica capillary column electron capture gas chromatography are given in Table 1. All results were calculated on a dry weight basis. Also listed in this table are the results for the percent moisture determinations for each core section. The results of analysis by GC/MS for several selective samples are listed in Table 2. A list of the gas chromatographic conditions are provided in Table 3.

Table 1

RESULTS

Sample #	% Moisture	Concentrations (μg/g)[1] DDE o,p'	DDE p,p'	DDD o,p'	DDD p,p'	DDT o,p'	DDT p,p'	Total DDT and Analogues	HBB	PBB
01-1	64.3	ND[2,6]	0.07	0.08	0.10	ND[3]	0.18	0.43	3.2	2.8
01-2	63.9	ND[6]	0.10	0.12	0.13	ND[3]	ND	0.35	0.42	0.46
02-1	55.4	ND[3]	0.31	0.22	0.58	0.10	3.8	5.0	1.0	1.0
02-2	48.2	0.06	0.48	0.22	0.58	0.36	5.3	7.0	1.1	0.76
03-1	64.9	ND[3]	0.32	0.22	0.57	ND[3]	0.61	1.7	0.96	0.84
03-2	61.1	ND[4]	1.1	4.2	12	ND[5]	1.2	18	ND[7]	ND[7]
03-3	51.8	0.12	1.0	4.8	12	0.49	6.4	25	ND[4]	ND[4]
04-1	34.8	0.27	2.2	12	20	32	200	270	15	330
04-2	45.2	ND[4]	7.2	32	32	4.8	47	120	90	23
05-1	63.2	0.06	0.48	0.64	1.5	0.11	11	14	92	0.94
05-2	65.3	1.2	2.8	4.2	8.8	0.22	7.2	24	6.2	ND[5]
05-3	68.1	2.2	5.3	22	58	110	790	990	ND[5]	ND[5]
05-4	61.6	ND[6]	ND[6]	ND[3]	ND[3]	ND[3]	ND[3]	-	ND[5]	ND[5]
06-1	57.1	ND[3]	0.18	0.32	1.1	0.19	0.84	2.6	12	2.2
06-2	43.7	0.08	0.34	0.62	0.66	0.21	1.6	3.5	28	0.56
06-3	60.5	0.24	0.68	2.1	2.6	ND[4]	2.2	7.8	0.92	ND[5]
06-4	51.9	ND[3]	ND[3]	ND[4]	ND[4]	ND[4]	ND[4]	-	ND[5]	ND[5]
07-1	72.0	0.31	1.8	9.7	24	4.6	64	100	ND[5]	14
07-2	71.5	3.8	9.7	7.0	220	ND[8]	74	380	9300	64
07-3	61.3	130	160	3400	3700	7300	11000	26000	100	ND[9]
07-4	63.4	0.06	0.05	0.68	0.85	1.1	2.0	4.7	1.0	ND[5]
08-1	70.3	ND[3]	2.1	6.0	17	ND[10]	11	36	2600	58
08-2	70.8	ND[3]	22	700	1100	140	890	2900	1800	ND[5]
08-3	63.2	21	47	2000	2200	1400	3200	8800	ND[5]	ND[5]
09-1	65.6	0.12	0.64	2.3	6.5	1.6	22	33	140	18
09-2	68.3	ND[3]	4.5	180	260	32	130	600	24	18
09-3	59.6	ND[3]	ND[3]	27	26	6.5	18	78	ND[5]	ND[5]
09-4	62.4	ND[3]	ND[3]	2.5	1.6	0.39	1.0	5.5	ND[5]	ND[5]

Table 1 (Cont'd)

Sample #	% Moisture	Concentrations (μg/g)[1] DDE		DDD		DDT		Total DDT and Analogues	HBB	PBB
		o,p'	p,p'	o,p'	p,p'	o,p'	p,p'			
18-1	64.0	0.10	0.29	4.5	6.2	1.8	7.8	21	4.3	1.2
18-2	31.3	ND[3]	ND3	0.12	0.24	ND[4]	ND[4]	0.36	0.58	ND[5]
18-3	61.6	1.0	1.4	48	50	1.2	19	120	ND[5]	ND[5]
18-1 Dup.	66.3	0.10	0.27	3.4	5.6	0.81	4.0	14	5.0	1.3
18-2 Dup.	30.2	ND[3]	ND[3]	0.16	0.51	ND[4]	3.2	3.9	0.81	6.8
18-3 Dup.	56.9	0.56	1.1	28	34	14	64	140	ND[5]	ND[5]
19-1	56.5	0.08	0.28	2.3	5.0	0.21	3.4	11	34	3.0
19-2	70.5	1.0	1.6	40	35	ND[10]	10	88	42	4.8
19-3	34.8	ND[6]	ND[6]	ND[3]	ND[3]	ND[3]	ND[3]	-	ND[4]	ND[4]
20-1	61.8	0.11	0.38	1.7	3.9	0.18	6.7	1.3	82	3.4
20-2	68.6	4.0	6.0	170	160	12	210	560	28	ND[5]
20-3	18.4	ND[3]	ND[3]	0.50	04.6	ND[4]	0.10	1.1	ND[5]	ND[5]
21-1	66.6	0.06	0.26	2.0	4.2	0.16	2.7	9.4	54	3.4
21-2	69.4	2.6	3.0	88	84	3.1	18	200	32	ND[7]
21-3	51.9	ND[6]	ND[6]	ND[3]	ND[3]	ND[3]	ND[3]	-	ND[4]	ND[4]
21-1 Dup.	67.9	0.06	0.26	2.0	4.2	0.20	2.3	9.0	52	3.3
21-2 Dup.	70.0	2.2	2.6	78	70	ND[7]	2.3	160	39	ND[7]
22-1	70.3	0.06	0.32	2.3	4.8	ND[4]	1.1	8.6	35	2.1
22-2	72.3	4.5	5.2	180	170	ND[7]	7.0	370	48	5.2
22-3	64.9	0.19	0.30	5.1	9.6	ND[4]	0.74	16	ND[5]	ND[5]
23-1	66.6	0.08	0.36	1.6	3.4	0.22	2.0	7.7	180	4.4
23-2	67.6	0.70	1.8	13	32	1.0	38	87	22	ND[5]
23-3	67.1	3.8	4.0	150	140	ND[5]	9.7	310	ND[7]	ND[7]
23-4	46.6	ND[6]	ND[6]	ND[3]	ND[3]	ND[3]	ND[3]	-	ND[4]	ND[4]

Table 2

GC/MS CONFIRMATION RESULTS

Compound	Detection Limit	Concentration (μg/g)[1]				
		#05-3	#08-1	#08-2	#08-3	#15-4
o,p'-DDE	10	ND[2]	ND	ND	22	19
p,p'-DDE	20	ND	ND	ND	38	25
o,p'-DDD	50	ND	ND	680	1900	820
p,p'-DDD	50	ND	ND	1100	2200	970
o,p'-DDT	50	93	ND	170	1300	210
p,p'-DDT	50	720	ND	780	3600	480
Hexabromobenzene	100	ND	2500	1800	ND	ND
Polybrominated Biphenyl	600	ND	ND	ND	ND	ND

(1) dry weight basis
(2) ND = none detected

Table 4

QUALITY CONTROL SUMMARY

	#07-3			#08-3			#12-1			#16-3		
	1	2	RPD[1]	1	2	RPD[1]	1	2	RPD[1]	1	2	RPD[1]
Duplicate Analyses												
o,p'-DDE	130	120	8	21	25	9	0.10	0.08	22	0.66	0.74	11
p,p'-DDE	160	160	0	47	59	23	0.39	0.48	21	0.80	0.84	5
o,p'-DDD	3400	3700	8	2000	2400	18	2.9	3.0	3	18	22	10
p,p'-DDD	3700	5000	30	2200	2600	17	6.4	7.2	12	20	24	18
o,p'-DDT	7300	8600	16	1400	2200	44	1.1	0.70	44	2.4	2.6	8
p,p'-DDT	11000	12000	9	3200	4600	36	3.9	6.4	48	12	14	15
HBB	100	100	0	ND	ND	-	90	78	14	ND	ND	-
PBB	ND[2]	ND	-	ND	ND	-	5.0	4.0	22	ND	ND	-

(1) RPD = Relative Percent Difference
(2) ND = none detected

Table 5

SPIKE RECOVERIES

Compound	Amount Added (μg)	% Recovery			
		#10-1	#17-2	#18-2	#20-3
o,p'-DDE	2.5	84	104	95	84
p,p'-DDE	2.5	96	108	97	84
o,p'-DDD	2.5	76	108	99	84
p,p'-DDD	2.5	60	100	100	87
o,p'-DDT	2.5	80	104	92	75
p,p'-DDT	2.5	NA[1]	108	94	71
HBB	2.5	NA	160	118	87
PBB	2.5	NA	180	104	90

(1) NA = not applicable

AIR POLLU[illegible]ON CONTROL DISTRICT -- COUNTY [illegible] LOS ANGELES

TEST NO. C-1895 PAGE 7

SAMPLING STATION Sulfur Unit No. 1 DATE 1-20-73

GAS VELOCITY DATA

TIME	POINT	VEL. HEAD IN. H_2O	TEMP. °F	VELOCITY FT/SEC	VEL. HEAD IN. H_2O	TEMP. °F	VELOCITY FT/SEC	VEL. HEAD IN. H_2O	TEMP. °F	VELOCITY FT/SEC
[illegible]	1	.1[illegible]	130	27.[illegible]	.2[illegible]		27.[illegible]	[illegible]		[illegible]
	2	.17		29.0	.17		29.0	.17		[illegible]
	3	.18		29.9	.18		29.9	.18		[illegible]
	4	.15		29.9	.20		31.5	[illegible]		[illegible]
	5	.22		33.0	.23		33.8	.22		[illegible]
	6	.25		34.5	.25		35.2	[illegible]		[illegible]
	7	.26		35.9	.26		35.0	.27		35.6
	8	.23		33.8	.21		32.2	.23		33.[illegible]
	9	.1[illegible]		25.3	.12		24.4	[illegible]		[illegible]
	10	.17		29.0	.16		28.2	.16		[illegible]
	11	.18		29.9	.16		28.2	.18		[illegible]
	12	.20		31.5	.18		29.9	.19		30.[illegible]
	13	.22		33.0	.22		33.0	.21		32.[illegible]
	14	.26		35.9	.26		35.9	.25		35.2
	15	.27		36.6	.28		37.3	.27		36.6
	16	.26		35.9	.24		34.5	.24		34.5
10:38			130							
	Avg			32.0			31.6			31.[illegible]

Static + .19" H_2O

A. INDICATED VELOCITY (TRAVERSE) FT/SEC 31.8

B. INDICATED VELOCITY (REFERENCE PT.) FT/SEC

C. FLUE FACTOR, A/B 1.00

D. PITOT CORRECTION FACTOR 1.00

E. GAS DENSITY CORRECTION FACTOR 1.02 (a)

F. GAS PRESSURE IN STACK, IN. HG. ABS. 30.15

G. GAS PRESS. CORREC. FACTOR, $\sqrt{29.9/F}$ .996

H. CORRECTED VELOCITY, AxDxExG, FT/SEC 32.4

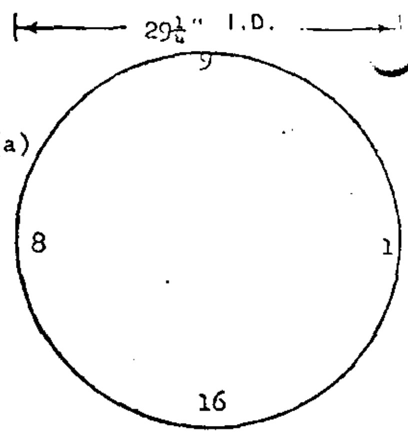
OR BxCxDxExG, FT/SEC

J. AREA OF FLUE, SQ. FT. 4.67

K. AVERAGE FLUE TEMPERATURE, °F 130

L. FLOW RATE, HxJx60, CFM 9080

M. FLOW RATE, (F/29.9)x520xL/(K+460), SCFM 8100



(a) Assumed value based upon gas composition info. in permit application.

R-1 60

AIR POLLUTION CONTROL DISTRICT -- COUNTY OF LOS ANGELES

TEST NO. C-1895 PAGE 8

SAMPLING STATION Sulfur Unit No. 1 DATE 1-10-7[illegible]

WATER VAPOR AND GAS DENSITY CALCULATIONS

PERCENT WATER VAPOR IN GASES

	NaOH Train	$ZnCO_3$ Train
A. GAS PRESSURE AT METER, IN. HG (ABSOLUTE)	29.15	29.15
B. VAPOR PRESSURE OF WATER AT IMPINGER TEMP., IN. HG	.469	.[illegible]
C. GAS VOLUME METERED, SCF	15.68	15.74
D. WATER VAPOR METERED, C X B/A, SCF	.25	.24
E. WATER VAPOR CONDENSED, VAPOR VOLUME, SCF	2.27	1.81
F. TOTAL WATER VAPOR IN GAS SAMPLE, D + E, SCF	2.52	2.05
G. TOTAL GAS VOLUME SAMPLED, C + E, SCF	17.95	17.55
H. PER CENT WATER VAPOR IN GAS SAMPLE, 100 X F/G	14	12

GAS DENSITY CORRECTION FACTOR

COMPONENT	VOLUME PER CENT/100 x	MOISTURE CORRECTION 1 − H/100 x	MOL. WT. =	WEIGHT PER MOLE OF STACK GAS
WATER		1.0	18.0	
CARBON DIOXIDE	DRY BASIS		44.0	
CARBON MONOXIDE	DRY BASIS		28.0	
OXYGEN	DRY BASIS		32.0	
NITROGEN & INERTS	DRY BASIS		28.2	

J. MOLECULAR WEIGHT OF STACK GAS ____

K. DENSITY OF GAS REFERRED TO AIR = J/28.95 = ____

L. GAS DENSITY CORRECTION FACTOR = $\sqrt{1.00/K}$ = ____

60048