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SAMPLING AND ANALYSIS OF SELECTED TOXIC SUBSTANCES

Task IA - Hexachlorobenzene

Environmental Protection Agency
Office of Toxic Substances
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Task IA - Hexachlorobenzene

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Project Officer

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NOTICE

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CONTENTS

CONTENTS (concluded)

FIGURES

FIGURES (continued)

FIGURES (concluded)

TABLES

TABLES (continued)

TABLES (continued)

TABLES (concluded)

SUMMARY

The purpose of this program is to provide sampling and analysis capabilities to EPA's Office of Toxic Substances, so that the levels of suspected toxic substances in air, water, soil, and sediment at designated locations throughout the United States may be determined. Four tasks have been assigned on this program. The first task included the sampling and analysis for hexachlorobenzene (HCB).

Methods for sampling and analyzing HCB in air, water, soil, and sediments were evaluated. A protocol was developed and approved.

Nine industrial plants were selected for sampling. The plants represent six major industries: perchloroethylene, trichloroethylene, carbon tetrachloride, chlorine, triazine herbicides, and pentachloronitrobenzene.

Of the six industries sampled, higher concentrations of HCB were associated with the production of perchloroethylene, trichloroethylene, and carbon tetrachloride. In the one plant that produced only carbon tetrachloride, HCB levels were quite low. HCB concentrations detected in samples from the pentachloronitrobenzene plant were relatively high, i.e., low micrograms per cubic meter range in air and generally over 100 $\mu g/g$ along in-plant roads. The levels of HCB associated with plants producing chlorine and triazine herbicides were very low.

Several different waste-disposal methods were used at the perchloro- and trichloroethylene plants that were sampled. The highest level of HCB was detected in air and soil at the plant using on-site landfill and open pit storage. High HCB levels were detected in loading and transfer areas at plants using off-site disposal methods. Lower levels of HCB were found at plants using on-site incineration but downwind air concentrations were elevated above background at these plants. The production of perchloro- and trichloroethylene by low temperature oxychlorination and the incineration of liquid bottom wastes resulted in a high HCB level ($\mu g/m^3$) in the air but relatively low levels in the effluent water.

The highest level of HCB found in the air on plant property was 24 $\mu g/m^3$. The HCB level in an open waste treatment pond was 306 µg/liter. The level of HCB in soil within the plant area was over 1,000 µg/g at three plants.

The maximum concentration of HCB in air sampled off plant property was 0.36 $\mu g/m^3$. A level of 3 $\mu g/m^3$ was detected at the boundary of another plant. Soil taken from a cornfield adjacent to one plant contained 1.1 µg/g, and over 3,000 µg/g were detected along a boundary road of another. HCB levels in water sampled beyond the plant property exceeded 1 µg/liter at two plants.

Samples were collected from two sewage treatment plants; negligible quantities of HCB were detected.

SECTION I

INTRODUCTION

Environmental contamination of hexachlorobenzene (HCB) has been reported internationally and nationally. Detection of HCB in human adipose tissue has been reported in Australia, Germany, and Japan.[1-3] In the United States, HCB has been detected in cattle raised in Louisiana, and sheep raised in New Mexico, Colorado, and California.[4] In addition, concentrations of HCB at 16 $\mu g/m^3$ have been detected in air samples collected by the Louisiana State Air Control Commission.[4]

On July 5, 1973, Midwest Research Institute (MRI) initiated a study to estimate the quantities and identify sources of HCB in the environment. The origin of HCB in the environment in the United States was identified as the waste materials or by-products from the production of perchloroethylene, trichloroethylene, carbon tetrachloride, chlorine, pentachloronitrobenzene and the herbicide Dacthal®. Specific industrial plants from the above industries were recommended to EPA as potential sampling sites.

On June 27, 1974, MRI project (3953-C) entitled "Sampling and Analysis of Selected Toxic Substances" was initiated. The objective of this program was to provide the EPA with sampling and analysis capabilities to determine the levels of toxic substances in air, water, soil and sediment from designated sources and ambient locations throughout the United States. The first task of this program was the sampling and analysis for HCB (Task IA) and HCBD (Task IB). Tasks II and III of this program are the sampling and analysis for ethylene dibromide, and evaluation of vinyl chloride levels in outdoor and indoor air due to the presence of PVC products, respectively. The ethylene dibromide study has been completed and reported to the Office of Toxic Substances in September 1975 under the title of: "Sampling and Analysis of Selected Toxic Substances: Task II - Ethylene Dibromide," EPA Report No. 560/6-75-001.

This report describes the Task IA of the program, i.e., the sampling and analysis for HCB as follows: experimental procedures; screening and selection of sampling sites; presampling surveys and field sampling; discussion of results, sewage treatment facilities; and summary and conclusions. Site surveys and field sampling data for individual sites, analytical data, and methods development efforts are appended to the report.

SECTION II

EXPERIMENTAL PROCEDURES

SAMPLING PROCEDURES

Water was sampled by two different methods--grab sampling and porous polymer extraction. The grab water samples were composited and stored in glass 1-gal. bottles that had been used for pesticide grade solvents. In the second technique, water was mechanically pumped through a 30 x 5.2 cm i.d. glass tube packed with 250 g Amberlite XAD-4. The Amberlite resin removed HCB quantitatively from the water stream flowing at 0.6 to 1.4 liters/min. The composited water samples and the Amberlite resin tubes were capped and stored in ice chests until ready for analysis.

Air was sampled through a 37-mm diameter, 0.8 μm pore size, millipore filter, followed by a 15-cm, 1.2-cm i.d., glass sampling tube* packed with 1 g Tenax®-GC. Air was drawn through the filter and Tenax®-GC tube with the aid of a mechanical pump. The flow rate was regulated with either an 18-gauge needle (~ 3.5 liters/min) or a 26-gauge needle (~ 0.4 liters/min). A schematic of the air sampling train is shown in Figure 1.

Soil and sediment samples from the top 2 to 5 cm were collected at designated sites. From 0.5 to 1 kg of sample was composited and stored in wide-mouth glass bottles with Teflon®-lined caps and kept in an ice chest until ready for analysis.

ANALYSIS PROCEDURES

Sample Preparation

The millipore filter and the Tenax®-GC resin for each air sample was sequentially extracted with 20, 20, and 10 ml of pesticide grade hexane using an ultrasonic bath. During the extraction, ice was added to the ultrasonic water bath to minimize evaporative loss of HCB. The hexane extracts were combined and diluted to 50.0 ml.

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* In sampling some industrial plants, two Tenax®-GC tubes were used in tandem.

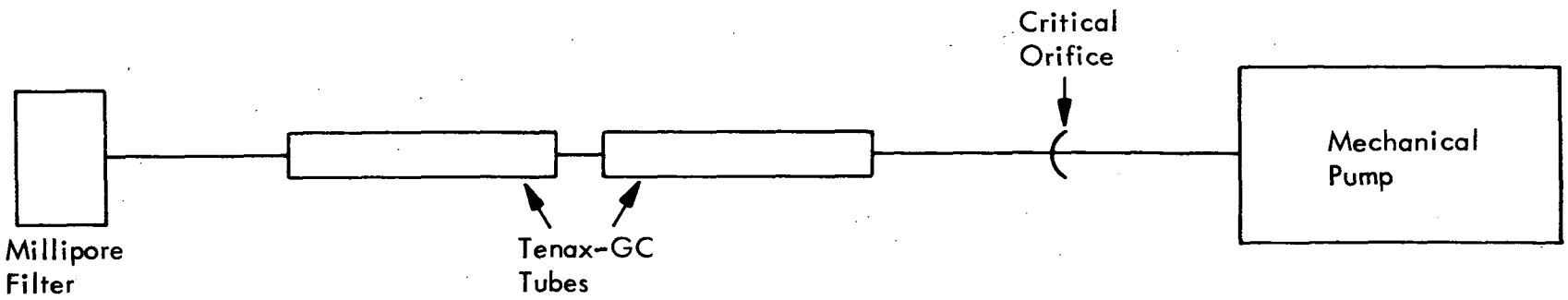

Figure 1. Air sampling train

The soil samples were first sifted on a U.S. Standard No. 18 sieve to remove stones and other foreign material. A 100-g sample was then extracted with 100 ml of n-hexane in a soxhlet apparatus overnight. The hexane extracts were transferred to 100-ml volume flasks and diluted to volume. A similar sample preparation procedure was used on the sediment samples except that the sifting step was omitted.

A 500 to 1,000-ml portion of each grab water sample was extracted sequentially with 20, 20, and 10 ml of hexane. The extracts were collected in a 50-ml volumetric flask and diluted to volume. The Amberlite XAD-4 resin was extracted with 250-ml hexane using a soxhlet apparatus. The extract was collected in a 250-ml volumetric flask and diluted to volume.

All of the extracted samples were kept in a walk-in cold room maintained at 4°C. Prior to analysis, the samples were brought to room temperature and diluted or concentrated as necessary for analysis.

Instrumentation and Conditions

A Microtek-2000R gas chromatograph equipped with an electron capture (tritium) detector was used. The output of the gas chromatograph was connected to a Hewlett-Packard 3380A integrator-recorder, which provides a printout of the chromatogram with integrated areas of individual peaks and respective retention times. A 4 ft x 1/4 in. glass column packed with 1.5% OV-17/1.95% QF-1 coated on 100/120 mesh Supelcoport® was used as the primary column for analysis. A 6 ft x 1/4 in. glass column packed with 3% XE-60 on 80/100 mesh chromosorb WHP was used to verify and differentiate HCB from α-BHC. The chromatographic operating conditions were: injector temperature, 200°C; column temperature, 150°C; detector temperature, 180°C; carrier flow rate, 100 ml/min nitrogen; purge flow rate, 90 ml/min nitrogen; and detector voltage, 10 V DC.

The instrumental limit of detection for HCB at the above mentioned conditions was 2 pg (10^{-12} g). Therefore, as an example, for any amount of air sampled, the quantity of HCB in the sample required for detection was greater than 10 ng (based on 10 μl injections of a 50-ml solution).

Calibration

A 10 ng/ml composite standard solution of HCB was used to obtain the calibration curves. The standard solution was prepared by dilution of a stock solution made up from EPA reference standards obtained from Pesticides and Toxic Substances Effects Laboratory, National Environmental Research Center, Research Triangle Park, North Carolina. Concentration ranges chosen for the calibration curve were from 10 to 60 pg, and linearity was observed.

A new calibration curve was obtained daily for the sample analysis. During the day, a known amount of the standard was injected periodically into the GC to check for changes in retention time and peak intensity.

SECTION III

SELECTION OF SAMPLING SITES

The objective of this task was to determine environmental levels of HCB by the sampling and analysis of samples from selected industrial plants. Therefore, it was important that the selected sampling locations be representative of the total industrial locations that are sources of HCB.

SELECTION CRITERIA

Selection criteria were chosen to achieve representative sampling of sites that are most likely to have detectable quantities of HCB present.

The criteria used for the selection of industrial plants for sampling are:

* Estimated quantity of HCB in industrial wastes, products, and by-products.
* Methods of production.
* Methods of waste disposal.
* Geographic location of the industrial plants.

Estimated Quantity of HCB in Industrial Wastes, Products, and By-Products

In 1974, the only active domestic producer of HCB for sale was Stauffer Chemical Company. However, industry sources report that HCB is contained in the "heavy ends" waste materials (residues) in the production of many chlorinated organic compounds, as well as in the electrolytic processes (either diaphragm or mercury cells) for chlorine gas when graphite anodes are used. An estimation of the amount of HCB produced in industrial wastes, by-products, and products is given in Table 1. As indicated in Table 1, about 90% of the HCB contamination in the environment was estimated to be from the perchloroethylene, trichloroethylene, and carbon tetrachloride industries.[5]

Table 1. ESTIMATED QUANTITIES OF HCB PRESENT IN INDUSTRIAL WASTES, BY-PRODUCTS, AND PRODUCTS IN 1972[5/]

Product	U.S. Production in 1972 (tons)	HCB (tons)
Perchloroethylene	367,400	1,313
Trichloroethylene	213,500	171
Carbon tetrachloride	498,500	150
Chlorine	9,538,000	143
Dacthal®	1,000	45
Vinyl chloride	2,545,000	13
Atrazine, propazine, simazine	56,000	3.5
Pentachloronitrobenzene	1,500	2.3
Mirex	500	0.8

Method of Production

The production method affects the quantity of HCB formed as a by-product. Therefore, the potential environmental contamination is dependent upon the production method. For example, carbon tetrachloride, perchloroethylene, and trichloroethylene are produced in several ways. If chlorine and the respective aliphatic hydrocarbons are fed into a high-temperature reactor and the products are collected by distillation, HCB is discharged as a by-product in the "heavy ends" wastes. However, if the production of carbon tetrachloride involves the reaction of chlorine with carbon disulfide, coproducts or by-products, other than reusable sulfur are greatly reduced.

Methods of Waste Disposal

Disposal methods for "heavy ends" wastes played a role in the selection of plants for sampling. The selected plants used a variety of disposal methods including landfill, deep well, sealed lagoons, on-site incineration, and shipment of wastes to other disposal firms.

Geographic Location of the Industrial Plant

Industrial plants were selected from across the country to determine whether the potential for environmental contamination posed a national problem.

RECOMMENDED SAMPLING SITES

Using these general criteria as a guide, 10 industrial plants were selected for inclusion in this study.

These industrial plants are listed below:

Perchloroethylene

Stauffer Chemical Company	Louisville, Kentucky
Vulcan Materials Company	Wichita, Kansas

Trichloroethylene

PPG Industry, Inc.	Lake Charles, Louisiana
Diamond Shamrock Corporation	Deer Park, Texas

Carbon tetrachloride

E. I. du Pont de Nemours and Company, Inc.	Corpus Christi, Texas
Dow Chemical Company	Pittsburg, California

Chlorine

Linden Chlorine	Linden, New Jersey
Kaiser Aluminum and Chemical Corporation	Gramercy, Louisiana

Triazine herbicides (atrazine, propazine, simazine)

Ciba-Geigy Corporation	St. Gabriel, Louisiana

Pentachloronitrobenzene

Olin Corporation	McIntosh, Alabama

The geographic location and EPA region of these plants are shown in Figure 2.

During the process of selecting the sampling sites, efforts were made to select industrial plants that produce a unique product rather than a combination of several products. The efforts were successful for the two chlorine plants selected. However, plants producing low molecular weight chlorinated hydrocarbons do not generally produce a single product. All five plants that produce trichloroethylene also produce perchloroethylene. Fortunately, not all the perchloroethylene production plants produce trichloroethylene. However, these plants also produce carbon tetrachloride. The annual production capacity (1972), process technology, and latest waste disposal methods for each of the sampling sites are presented in Table 2.

The Dacthal® production facility (Diamond Shamrock Corporation) in Greens Bayou, Texas, was not selected for sampling because the waste handling and product contamination were substantially changed from pre-1972 procedures.

Vinyl chloride and mirex production facilities were omitted from this survey.

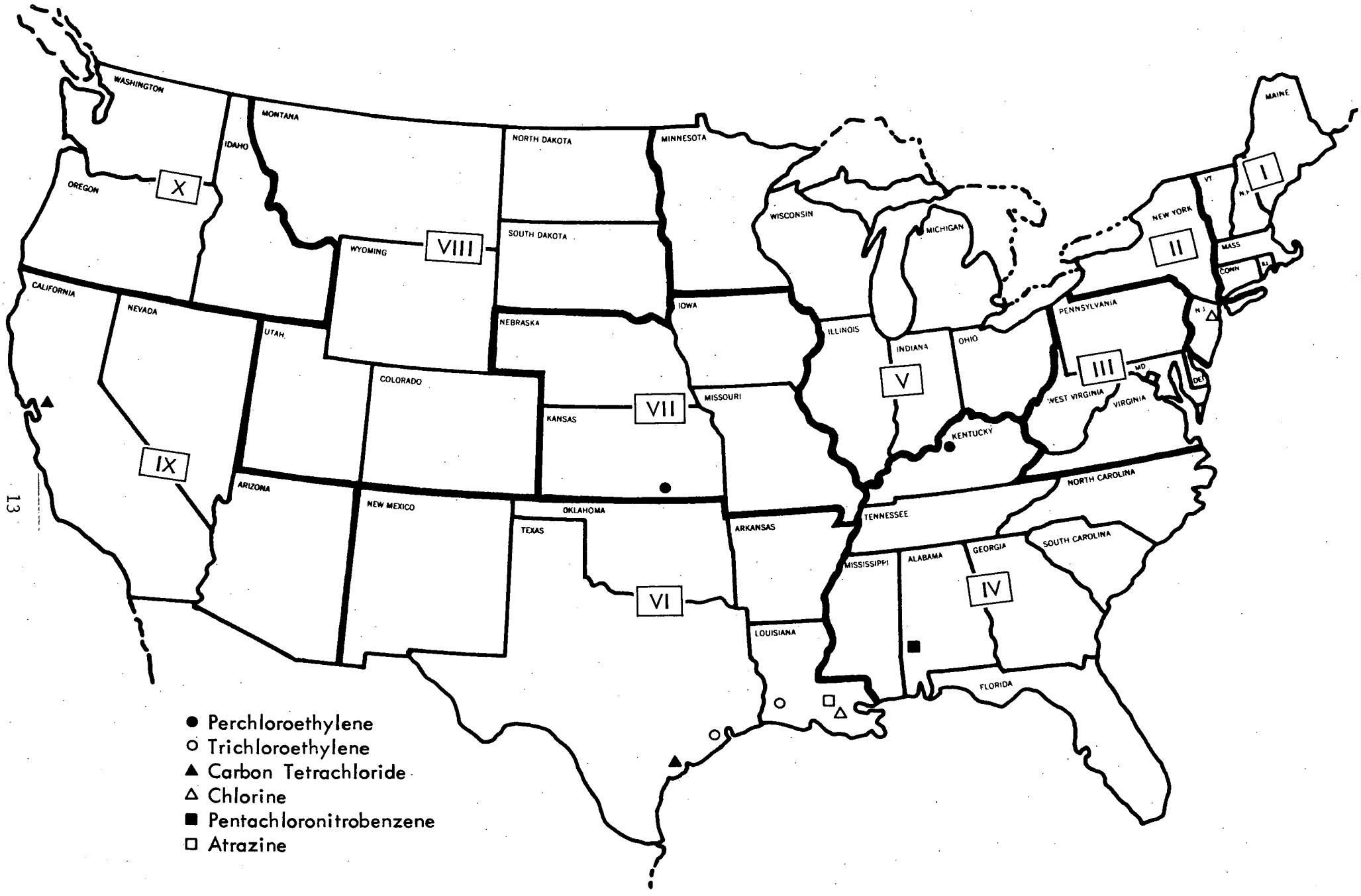

Figure 2. Geographic location of recommended sampling sites

Table 2. PRODUCTION CAPACITY, PROCESS TECHNOLOGY AND WASTE DISPOSAL AT RECOMMENDED SITES

Producers	Production sites	EPA region	Annual production capacity (10^3 tons)	Waste disposal	Process technology[a/]
Perchloroethylene					
Stauffer Chemical Company	Louisville, Kentucky	IV	35	HCB recovered for sale, remainder recycled to chlorinator	Chlorination with low molecular weight hydrocarbons, e.g., ethane, propane
Vulcan Materials Company	Wichita, Kansas	VII	25	Earth-covered groundfill	
Trichloroethylene					
PPG Industry Company	Lake Charles, Louisiana	VI	140	Incineration, landfill	Ethylene and chlorine as raw materials, under catalytic reaction at 250 to 300°C
Diamond Shamrock Corporation	Deer Park, Texas	VI	60	Ship to Rollins International for incineration	
Carbon tetrachloride					
E. I. du Pont de Nemours and Company, Inc.	Corpus Christi, Texas	VI	250	Landfill, ship to outside firm for disposal	Chlorination with methane at elevated temperature
Dow Chemical Company	Pittsburg, California	IX	23	Incineration	
Chlorine					
Linden Chlorine Company	Linden, New Jersey	II	66	Discharge to holding pond	Mercury cell; graphite electrode
Kaiser Aluminum and Chemical Corporation	Gramercy, Louisiana	VI	58	Landfill	Diaphragm cell; graphite electrode
Triazine herbicides					
Ciba-Geigy Corporation	St. Gabriel, Louisiana	VI	> 75	Still bottoms incinerated by an outside processor to extinction	Reaction of cyanuric chloride with appropriate amino hydrocarbons at elevated temperature
Pentachloronitrobenzene					
Olin Corporation	McIntosh, Alabama	IV	1.5	Stored in "blocks" covered with plastic sheet	Nitration of pentachlorobenzene or chlorination of various chloronitrobenzenes in the presence of catalyst

a/ Kirk-Othmer, Encyclopedia of Chemical Technology 2nd ed., Interscience Publishers, New York, New York (1972).

SECTION IV

PRESAMPLING SURVEYS AND FIELD SAMPLING

To plan the strategy for successful field sampling at the selected industrial plants, a presampling survey was conducted at each plant. Each presampling survey was arranged through telephone contact with the appropriate plant officials whose names were provided by the EPA project officer. Figure 3 shows the complete schedule for presampling surveys and field sampling.

PRESAMPLING SURVEYS

During the presampling site survey, a plant map was obtained. Information regarding the possible sources of HCB contamination, production technology and waste disposal techniques were obtained. In addition, the production and waste disposal sites, as well as the transportation routes were delineated. Accessible electrical outlets inside the plant were also located for possible use in air sampling. Meteorological conditions, such as wind direction and rainfall were investigated. Tentative sampling dates were agreed upon, subject to final confirmation by plant officials prior to the departure of the sampling crew from MRI.

FIELD SAMPLING

Upon the completion of a presampling site survey, the sampling strategy was planned. In general, air sampling stations were positioned upwind and at several distances downwind from the suspected source(s) of contamination. The air samplers were usually positioned 4 ft above ground. When the wind direction was uncertain, stations were positioned around the entire plant area.

Water sampling was conducted upstream and downstream of waste effluent. Storm runoff was collected when appropriate. Water samples from equilization ponds or solar ponds were collected to determine if the ponds were sources of air contamination through liquid vapor equilibrium of HCB.

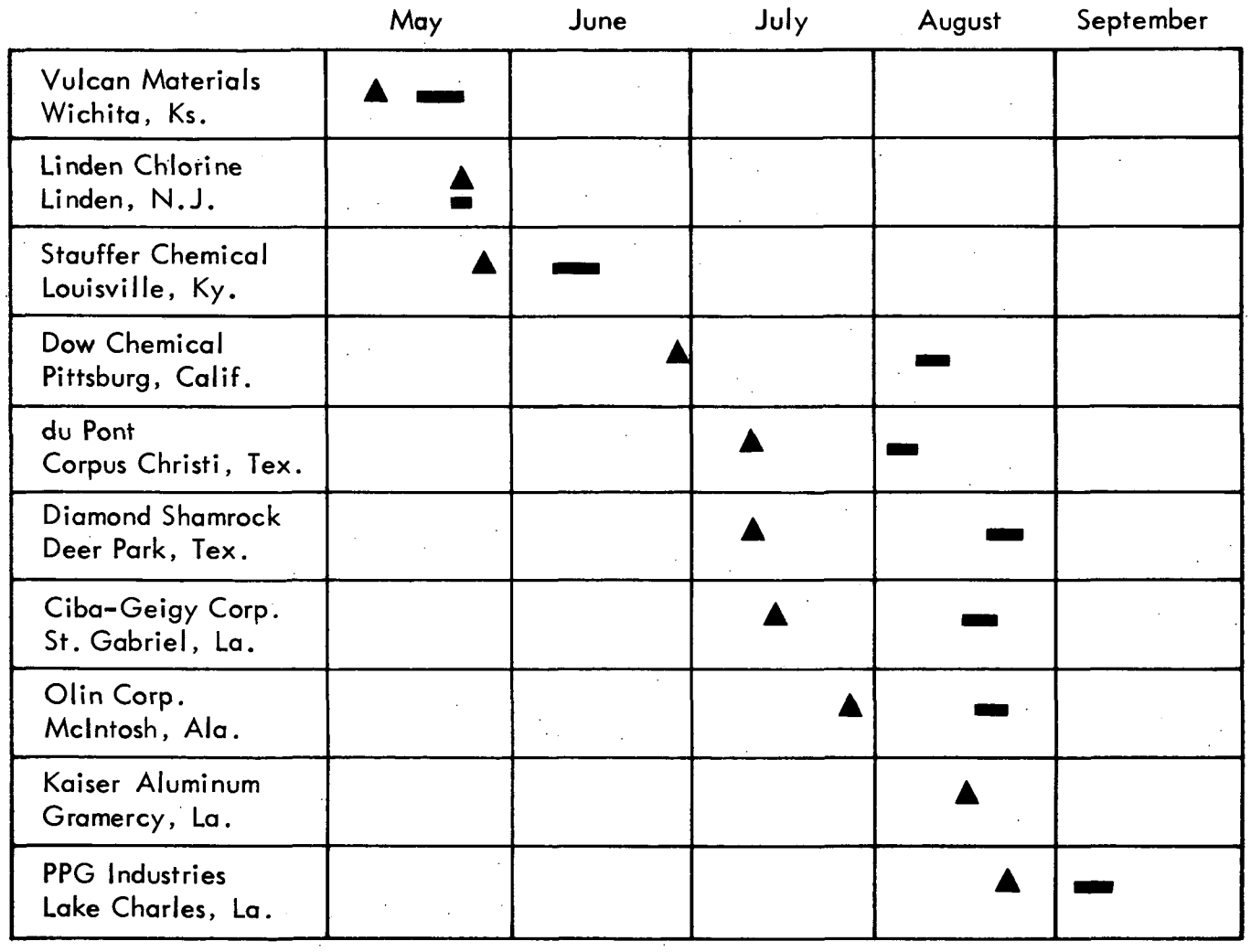

Figure 3. Presampling survey and field sampling schedule

Soil sampling was generally conducted along the plant boundaries, transportation routes, and around waste disposal and storage areas. Sediment samples were collected from streams, equilization ponds and natural solar ponds. Solids and liquid were also sampled from open disposal pits. The preparation for sampling usually was conducted 3 to 5 days prior to the sampling date. Sampling equipment was sent to the plant scheduled for sampling. Major sampling equipment included vacuum pumps, poles, rubber hoses, electrical prongs and adapters, and sampling bottles. To avoid possible breakage and contamination, the air sampling train components, i.e., the filter and the Tenax®-GC tubes, as well as the Amberlite XAD-4 sampling tubes were all hand carried to the site by the sampling crew. Generators were rented at local dealers when electrical outlets were not available in the plant.

Because of the extensive sampling involved in the first two sites, i.e., Vulcan Materials Company and Stauffer Chemical Company, a four-man crew was required. The rest of the sampling trips were conducted by two- or three-man crews. Generally, 3 days were spent on each sampling site. The total number of samples analyzed for each sampling site is presented in Table 3. A summary of air sampling parameters for each site is given in Table 4. The sites at which HCB particulate was detected are indicated. Detailed descriptions of the field sampling and presampling surveys conducted at each plant are presented in Appendix A.

Table 3. FIELD SAMPLING SUMMARY

Site	Air samples[a] (stations x train components x sampling period)	Total samples (number/type)
Vulcan	18 x 2 x 5	180 air
		10 soil
		4 water
Linden	No air samples	6 water
		3 solid
		1 soil
Stauffer	9 x 2 x 6	108 air
		5 soil
		6 water
		3 sediment
Dow	8 x 2 x 1	16 air
		3 soil
		1 water
Du Pont	8 x 2 x 1	10 air
		3 soil
		7 water
		3 sediment
Diamond Shamrock	8 x 3 x 1	24 air
		3 soil
		2 water
Ciba-Geigy	8 x 2 x 1	16 air
		4 soil
		2 water
Olin	8 x 3 x 1	24 air
		11 soil
		10 water
		1 sediment
PPG	10 x 3 x 1	30 air
		4 soil
		7 water
		3 sediment

a/ The total number of air samples consist of the number of air sampling stations times the components of the train, i.e., filter and Tenax®-GC resin times the number of sampling periods.

Table 4. SUMMARY OF AIR SAMPLING PARAMETERS

Plant	Average sampling vol. (ℓ)	Average sampling time (hr)	Rate (ℓ/min)	Particulate (HCB)
Vulcan[a]	(1) 150-200 (2) 800-1,000 (3) 4,000	(1) 1 (2) 4	0.5 0.5	Yes Yes
Stauffer[b]	450	2	3.5	Limited
Dow	4,100	20	3.5	No
Du Pont	4,200	21	3.5	No
Ciba-Geigy[c]	1,200-2,100	6-8	3.5	No
Diamond Shamrock	550	24	0.4	No
Olin[c]	1,500-2,000	9	3.5	No
PPG	1,200	24	0.4	Yes

a/ Five 4-hr periods.
b/ Six 4-hr periods (2 hr on, 2 hr off).
c/ Three 8-hr periods (each 2 to 3 hr sampling).

SECTION V

DISCUSSION OF RESULTS

Air, soil, water, and sediment samples were collected from nine recommended industrial plants whose products included perchloroethylene, trichloroethylene, carbon tetrachloride, triazine herbicides, pentachloronitrobenzene and chlorine. In general, HCB concentrations varied from a maximum, near the production and waste disposal areas, to a minimum, in the samples taken upwind of a recognizable source. However, in a few instances, HCB contamination was observed over the general plant area and a specific emission source was difficult to determine. HCB was detected as both a vapor and a particulate; the predominate form was dependent upon the production and waste disposal methods of each plant. The results from each sampling site are discussed below.

VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Field sampling at Vulcan Materials Company's perchloroethylene plant at Wichita, Kansas, was conducted on May 20, 1975. Other compounds of interest produced at this plant include carbon tetrachloride and chlorine. The samples collected were: 180 air (90 filters and 90 Tenax®-GC columns), 10 solid, and 4 water.

Air Samples

The 180 air samples were collected from three samplers which were positioned upwind, nine samplers immediately downwind of the general production and waste storage areas, and six sampling devices positioned further downwind beyond the northern plant boundary. The samplers beyond the northern plant boundary were positioned at three locations with two samplers per location at 4 and 11 ft above ground, respectively. The upwind and farthest downwind samplers were operated continuously for a 4-hr period while those closer to the general production and waste storage area were operated only for the first hour of each 4-hr period. After each 4-hr period, the filter and the Tenax®-GC column in each sampler were replaced by fresh components. The sampling

strategy was designed so that results of the analysis would elucidate: (a) the major sources and level of HCB emission, (b) the diurnal and operation-related effects of HCB emission, (c) the physical form, i.e., particulate or vapor of HCB in the plant air, and (d) the variation of HCB concentrations with respect to sampler distance above ground.

Sources and Levels of HCB Emission - The analytical data for the 180 air samples are tabulated in Table B-1 of Appendix B. Figure 4 shows the average concentrations of HCB during the 20-hr sampling period at the 18 sampling stations. It is obvious that major sources of HCB in the air are the production and waste storage ("Hex Pit") areas. In addition, other sources of HCB are indicated by the upwind air concentrations.

The HCB levels in samples immediately downwind of the production and storage areas ranged from 0.1 to 24 $\mu g/m^3$. The levels of HCB in the upwind samples and samples taken downwind beyond the northern plant boundary were similar and in the range of 0.1 to 2.1 $\mu g/m^3$.

Variation of HCB Emission with Time - The variation of HCB levels over the 20-hr sampling period is shown in Figures 5 through 7.

Figure 5 shows that the HCB levels in air samples immediately downwind of the production and waste storage area did not increase following the dumping of the "hex residue" at the "Perc Plant" or the "Hex Pit." The increase in HCB at Stations 4 and 6 occurred either prior to or significantly after dumping.

The variation of HCB levels in samples beyond the northern boundary of the plant is shown in Figure 6. Each point is the average of two samplers positioned at different heights (4 and 11 ft).

Figure 7 shows that during the entire sampling period, HCB levels were relatively high in upwind Stations 16 through 18; in fact, the 20-hr average values were greater than some of the downwind stations. Among the three stations, higher concentrations were detected at Stations 17 and 18 which were closer to the landfill than Station 16.

Physical Form of HCB - The average vapor and particulate concentrations of HCB over the 20-hr sampling period at each station are shown in Figure 8. The physical state of the HCB detected was dependent upon the sampling location. Particulate HCB was not detected in samples taken near the solar pond (Stations 7 through 9) and at the northeast corner (Stations 14 and 15), northwest corner (Stations 10 and 11) and southwest corner (Station 16) of the sampling area. At all other sampling stations, particulate HCB was approximately equal to or much greater than (Stations 4 through 6) the HCB detected as a vapor.

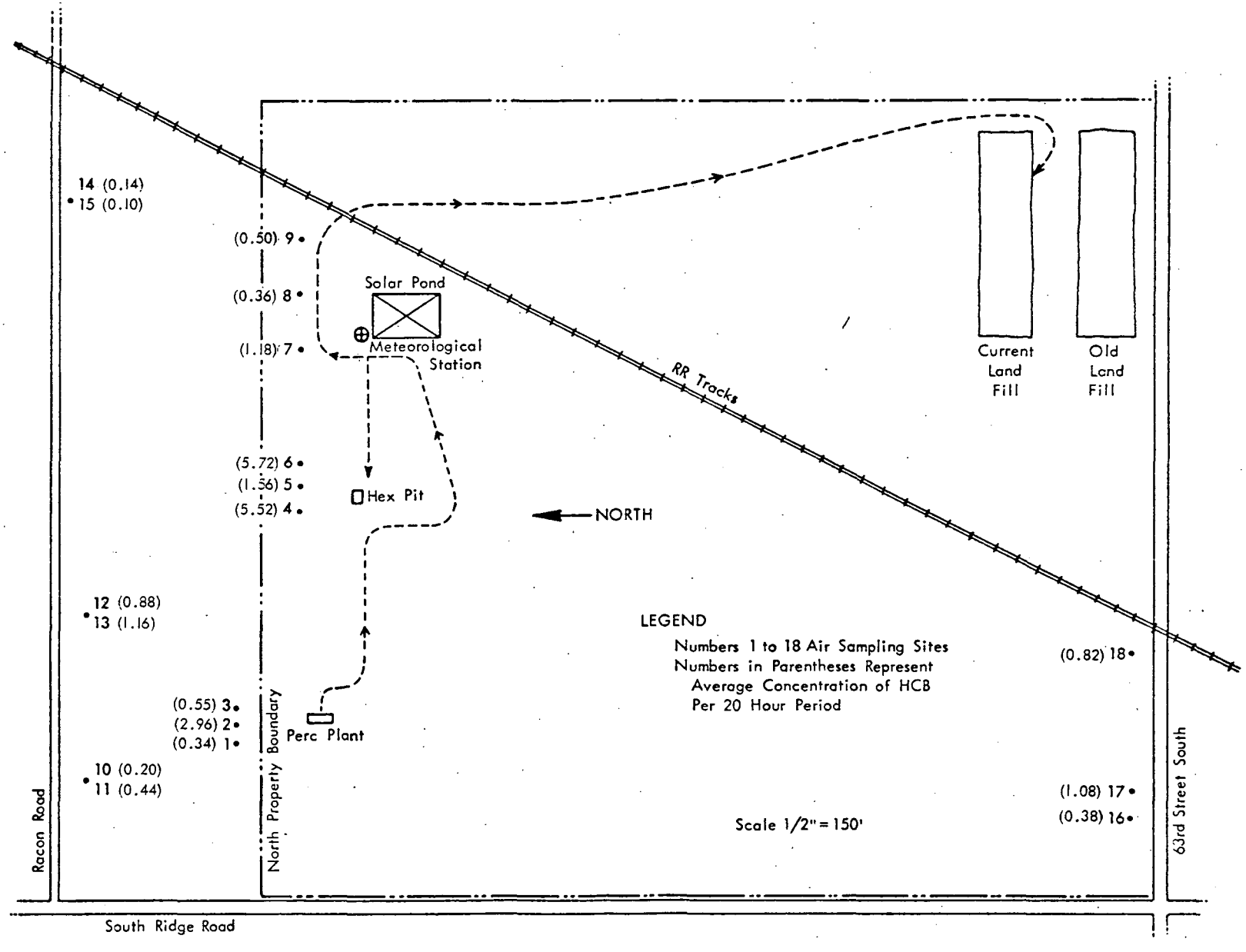

Figure 4. Average concentrations ($\mu g/m^3$) of HCB in air per 20-hr period at 18 sampling stations at Vulcan Materials Company, Wichita, Kansas

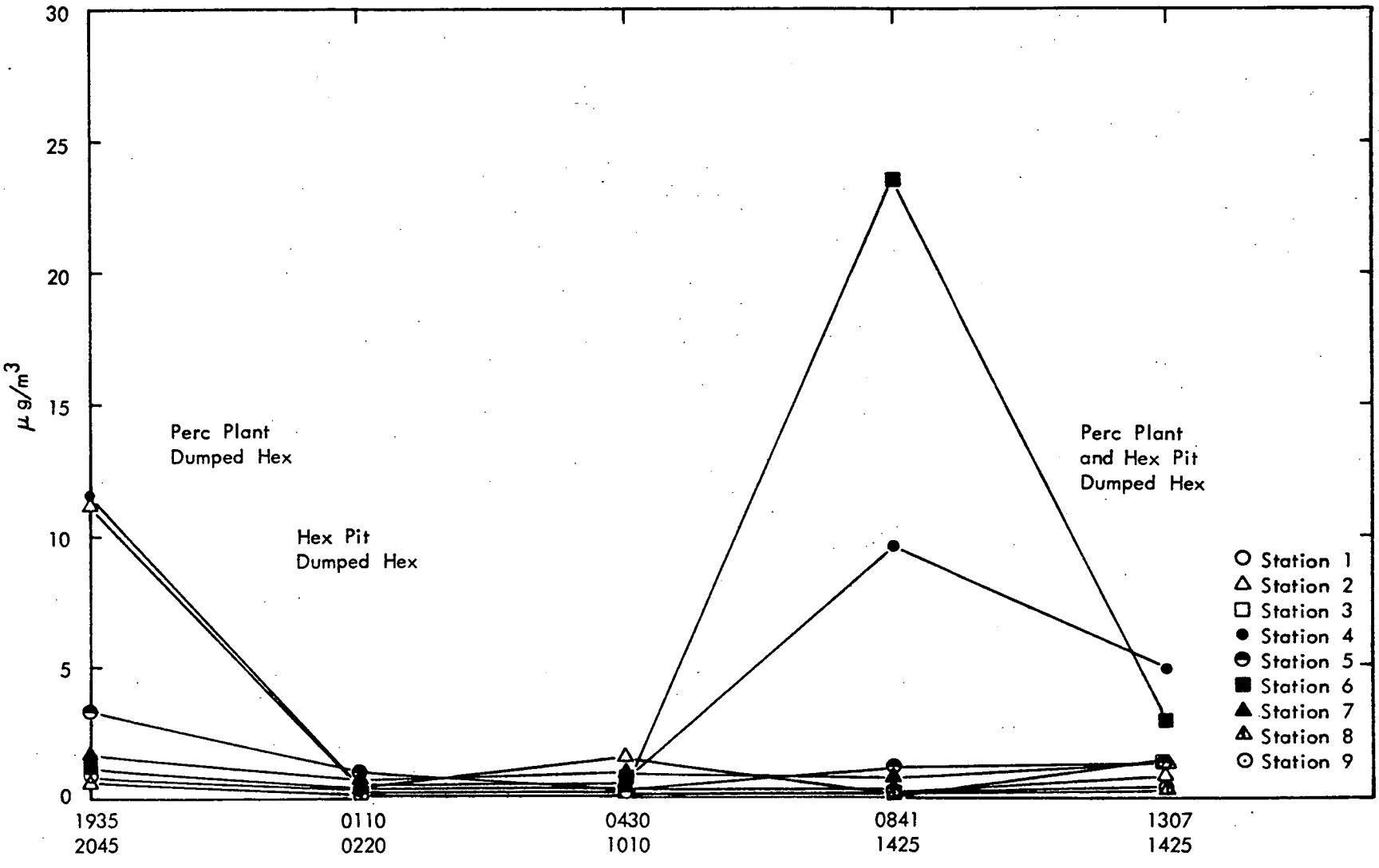

Figure 5. HCB concentration per 20-hr period at sampling stations within the plant perimeter (Vulcan)

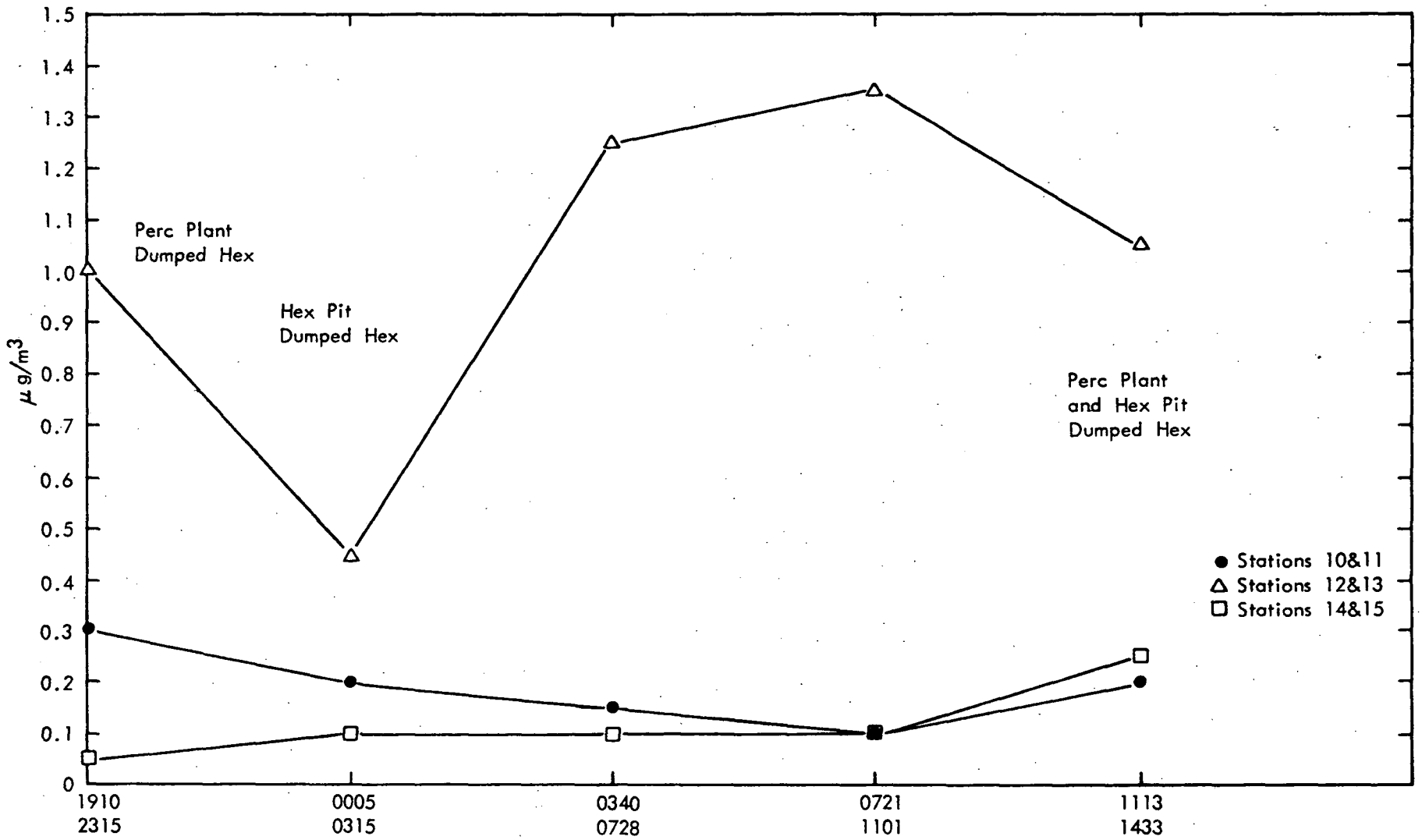

Figure 6. HCB concentration per 20-hr period, downwind stations (Vulcan)

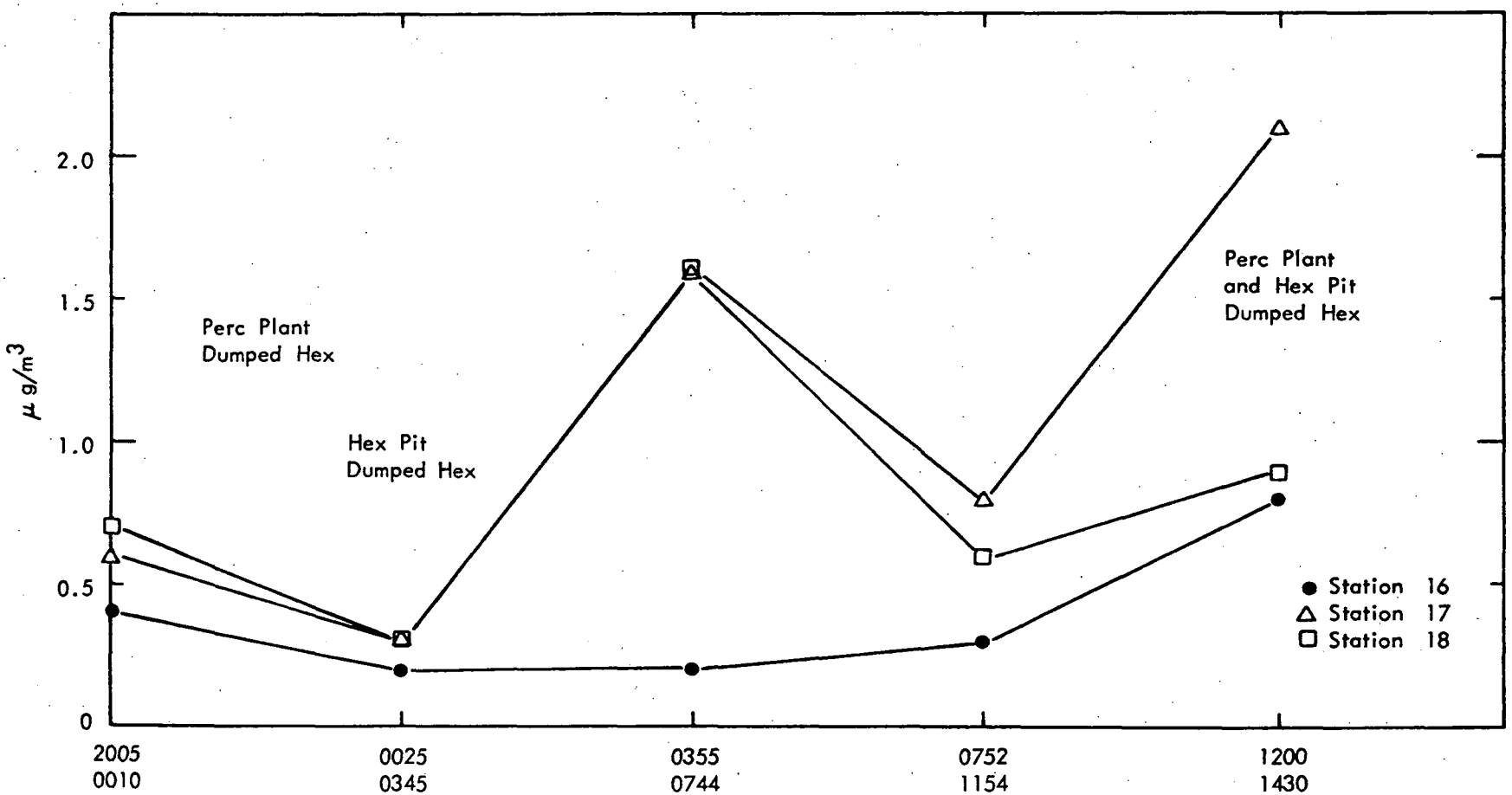

Figure 7. HCB concentration per 20-hr period, upwind stations (Vulcan)

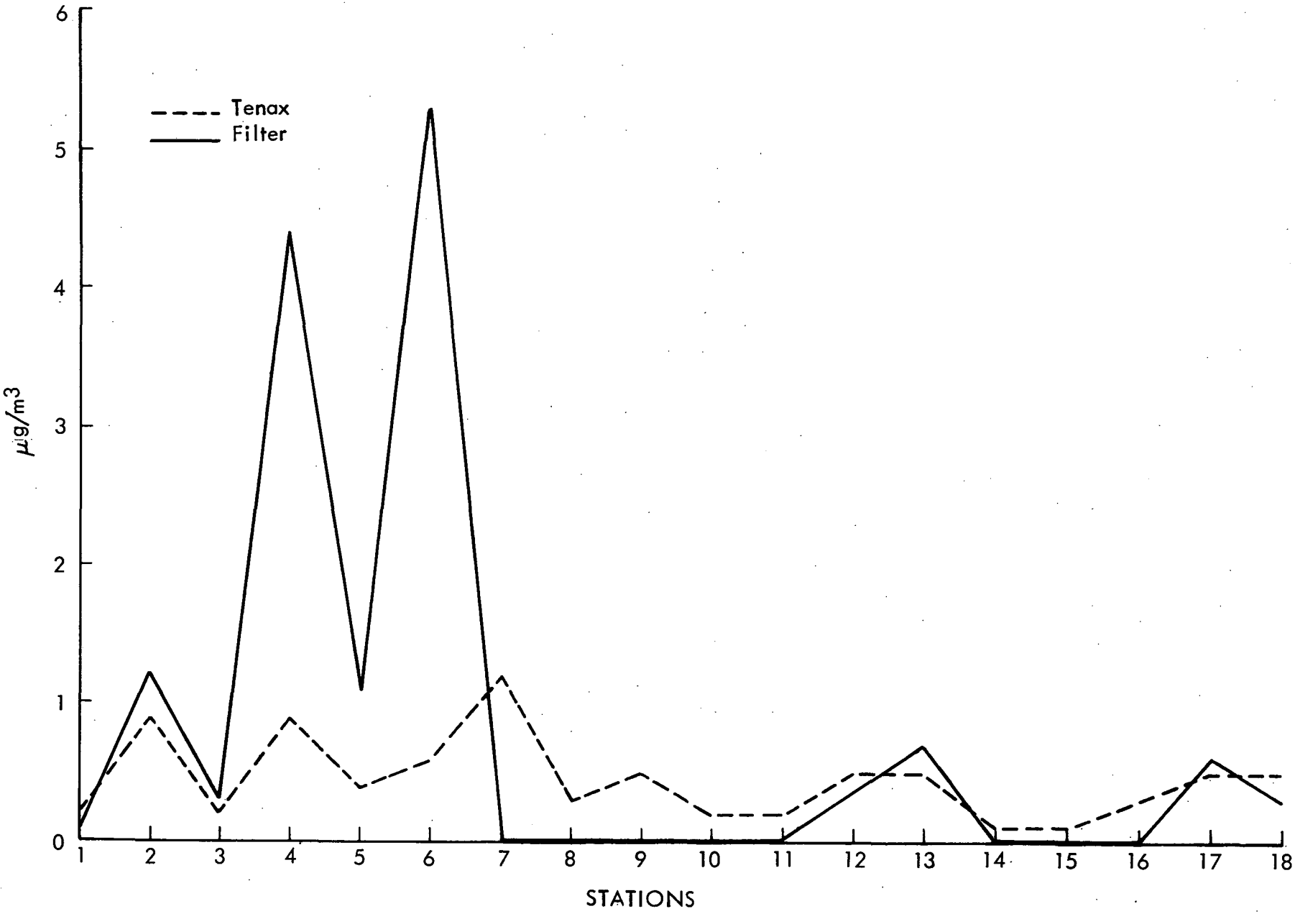

Figure 8. Average vapor and particulate concentrations of HCB in air per 20-hr period at 18 sampling stations (Vulcan)

The variation of HCB levels over the 20-hr period in samples taken near the "Hex Pit" was due almost entirely to variation in particulate levels. Figure 9 shows the HCB vapor concentration at Station 4 was relatively constant, near the 1 $\mu g/m^3$ level, whereas the particulate level fluctuated from 0 to 10 $\mu g/m^3$. Particulate HCB was not observed during the 0110 to 0220 and 0450 to 0550 sampling periods when vehicular activity was probably low; particulate levels were 5 $\mu g/m^3$ or higher during the periods of 1945 to 2045, 0902 to 1010, and 1307 to 1425. Figure 10 shows a similar, but less pronounced pattern for upwind Station 18; the HCB vapor concentration ranged from approximately 0.3 to 0.7 $\mu g/m^3$ while the particulate HCB varied from 0 to 1.2 $\mu g/m^3$.

All samples showed a relatively constant HCB vapor concentration during the entire sampling and fluctuating HCB particulate concentration (when detected) that probably reflects the activity in the immediate area of the sampling stations. A slight increase in HCB vapor concentration during the warmer sampling periods was observed at some sampling stations (Figure 10). This increase was not apparent until the particulate and vapor concentrations were considered separately.

HCB Concentration Versus Sampler Height - A comparison of HCB concentrations at 4 and 11 ft for five time periods is shown in Table 5. The results indicate that the differences in levels at the two heights were not significant.

Precision of Sampling and Analysis - If it is assumed that the HCB levels were the same at the two sampler heights (4 and 11 ft), the results from each paired station, i.e., 10 and 11, 12 and 13, and 14 and 15, at each of the five sampling periods can be considered as duplicates and a pooled relative standard deviation (PRSD)* determined. The PRSD calculated from these data indicates the overall precision of the air sampling and analysis methods including sample collection, storage, recovery, and analysis. The PRSD is 17%. The PRSD is based on 13 duplicates. Two duplicate measurements were not included in the calculations: one pair was near the detection limit (< 0.1 $\mu g/m^3$) and the other station had a high particulate level and was considered an outlier. The quantities of vaporous HCB detected in this station pair (Stations 12 and 13) were approximately equal.

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* The PRSD was calculated as follows:

$$S = \sqrt{\sum_{i=1}^{n} (X-\bar{X})^2/0.889}$$

$$RSD = S/\bar{X} \times 100$$

$$PRSD = \sqrt{\sum_{i=1}^{n} RSDi^2/n}$$

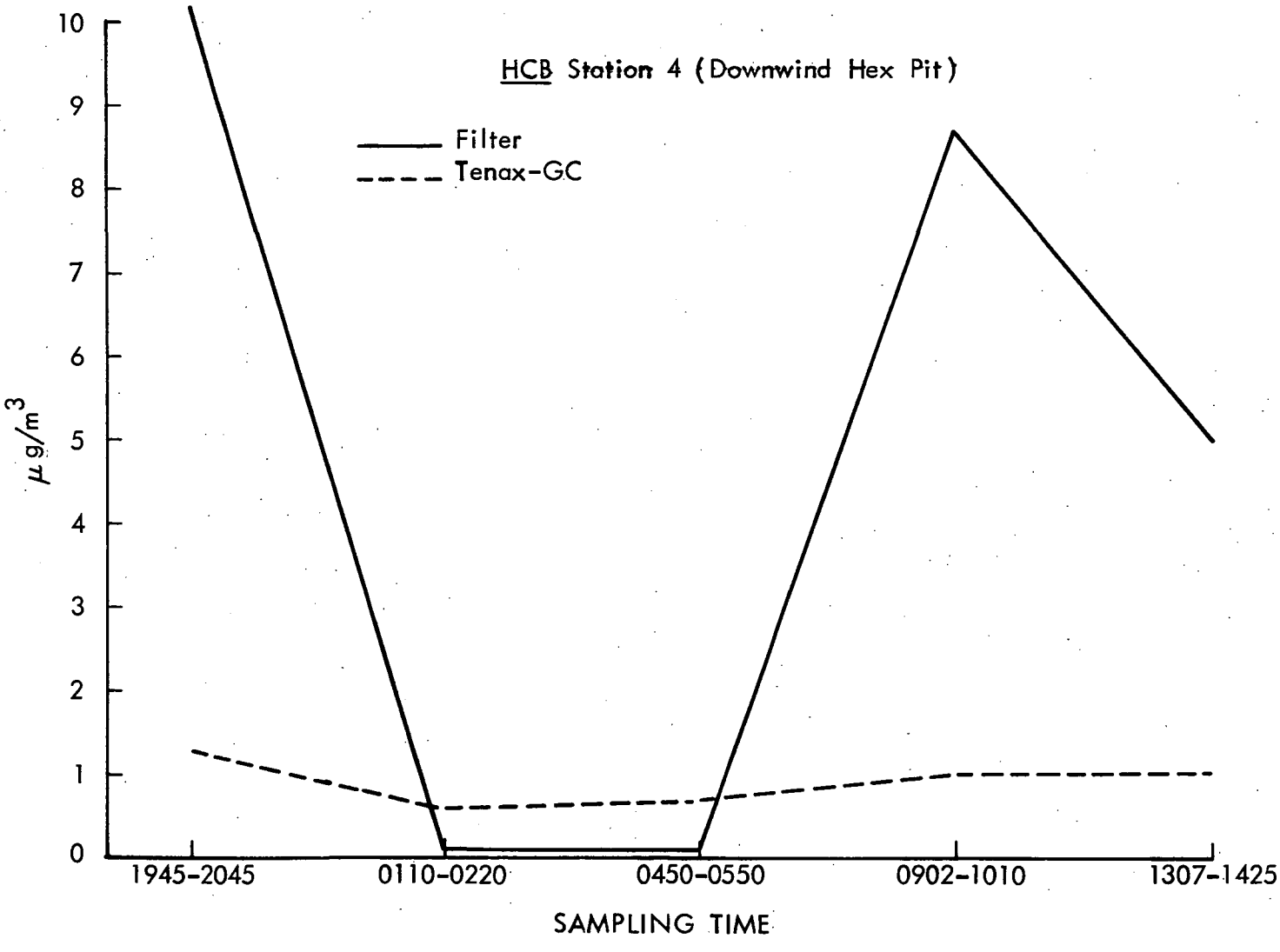

Figure 9. Variation in vapor and particulate HCB levels at Station 4 (Vulcan)

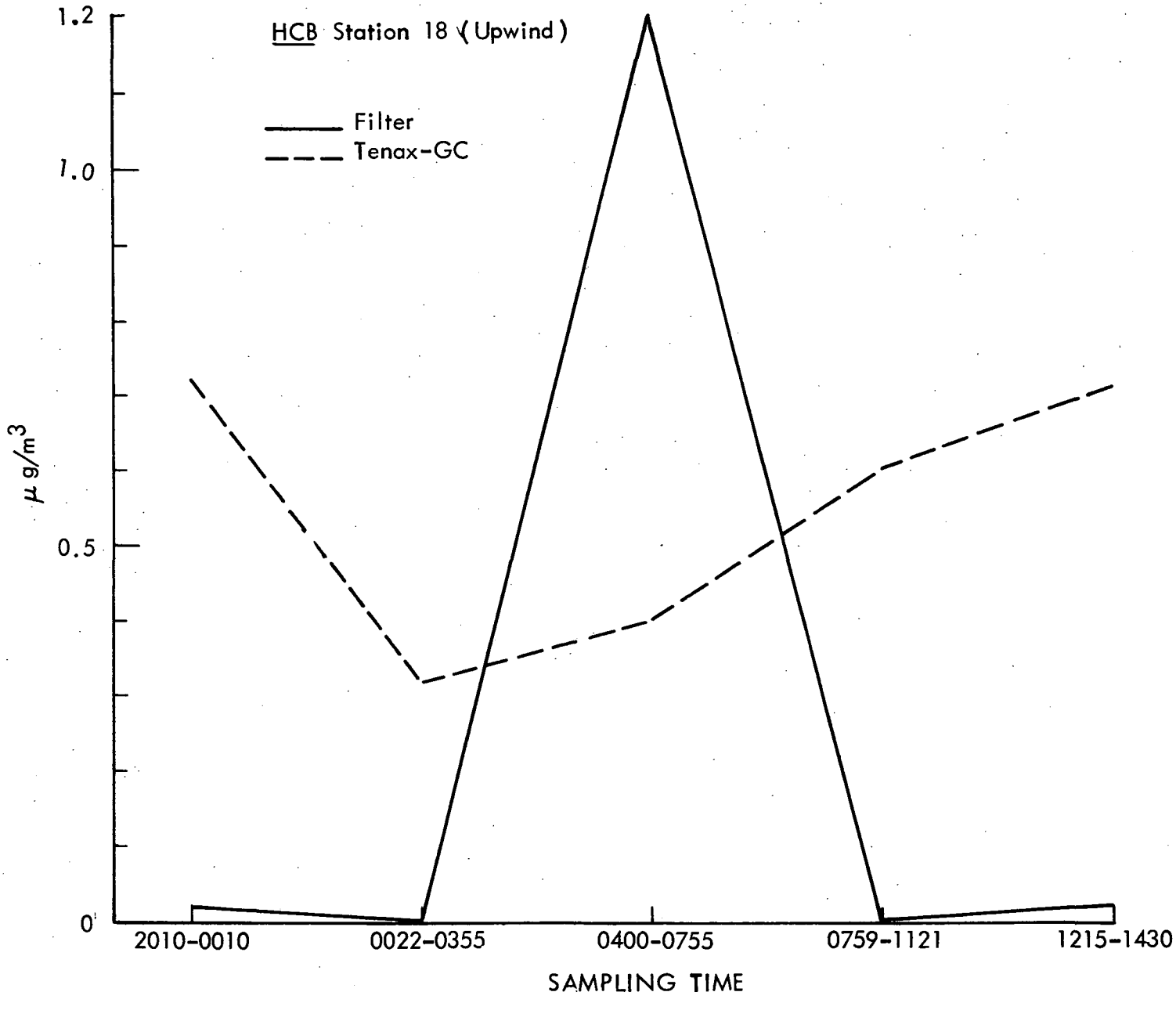

Figure 10. Variation in vapor and particulate HCB levels at Station 18 (Vulcan)

Table 5. COMPARISON OF HCB LEVELS AT 4 AND 11 FT FOR FIVE TIME PERIODS

Station pairs	HCB, $\mu g/m^3$ 4 ft	11 ft
10 and 11	0.3	0.3
	0.2	0.2
	0.2	0.2
	0.1	0.1
	0.2	0.2
12 and 13	1.0	1.0
	0.4	0.5
	1.2	1.3
	1.4	1.3
	0.4	1.7
14 and 15	0.1	0.03
	0.1	0.1
	0.1	0.1
	0.1	0.1
	0.3	0.2

Soil Samples

The nine soil samples and one "Hex Pit" solid sample were collected at the following locations: along the plant boundaries, transportation routes, landfill, the "Hex Pit" area and the production area. These sites were selected to determine HCB soil levels associated with production, waste disposal and transportation of wastes for disposal. Samples were also taken upwind and in adjacent agricultural fields to determine background concentrations in areas in the vicinity of the plant.

The results of the analysis of the nine soil and one solid samples, listed in Table 6, indicate that HCB soil concentrations were in the 1 to 1,500 ppm range, with the exception of the "Hex Pit" soil and the "Hex Pit" solids. HCB was 5% in the "Hex Pit" soil* and 21% in the "Hex Pit" solids.

Of the four plant boundaries, the highest level of HCB, 126 ppm, was observed in soil from the southern boundary (S-8). HCB levels ranged from 1.1 to 1.3 ppm in samples taken along the other boundaries.

The observation of relatively high concentrations of HCB in the southern boundary soil sample (S-8) could very well be the source of high concentrations of HCB in the air samples collected at Air Sampling Stations 16 through 18. The high concentration in the soil samples upwind of the production area could be due to the nearby landfill. This rationale is supported by the presence of over 1,000 ppm HCB in the S-5 sample, which indicates the landfill is a source of surface contamination in the immediate area.

The soil on the route to the "Hex Pit" (S-2) and the soil from the "Hex Pit" to the landfill (S-4) contained over 100 ppm HCB. HCB concentrations found in the water layer covering the "Hex Pit" were also relatively high. The results of the water analysis are presented below.

Water Samples

Two samples were taken from Cowskin Creek (Figure A-3) which receives water from the sanitary sewer system and plant heat exchangers. Samples were taken from the "Hex Pit" and solar pond to determine their contribution to HCB levels in air and into the deep well which receives water from the solar pond.

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* Taken from a 10-ft radius of the "Hex Pit."

Table 6. HCB CONCENTRATIONS IN SOIL AND SEDIMENT FROM VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Samples[a/]	Sample weight (g)	Concentration (µg/g) HCB
S-2	42.8	109
S-3	2.5	5%
S-4	48.4	157
S-5	38.7	1,453
S-6	40.5	5.6
S-7	29.6	1.3
S-8	35.6	126
S-9	43.5	1.2
S-10	34.2	1.1
"Hex Pit" solids	0.95	21%
Control		ND

a/ S-2 - Route from "Perc Plant" to "Hex Pit."
S-3 - "Hex Pit."
S-4 - Route from "Hex Pit" to landfill.
S-5 - Landfill (60 yards north of 63rd Street and 0.4 miles east of Ridge Road.
S-6 - Landfill (180 yards north of 63rd Street and 0.4 miles east of Ridge Road.
S-7 - Landfill (Ridge Road to telephone pole).
S-8 - Upwind.
S-9 - Downwind.
S-10 - Western boundary (cornfield).
Control - Soxhlet apparatus.

The results of the water analyses are shown in Table 7. The HCB levels were relatively low--parts per trillion in the Cowskin Creek water.

A high concentration (306 ppb) in the "Hex Pit" water was expected since this water covers the "hex residues" dumped in the pit. This water is likely to be saturated with HCB. The concentration in the solar pond was over two orders of magnitude lower than that in the "Hex Pit" water. The source of HCB in the solar pond water could be from leaching of the soil in the plant area or from vapor or airborne particulate from the neighboring "Hex Pit."

Plant Summary

The results of the analysis of all air, soil, and water samples indicate that the "Hex Pit" is the source of the highest levels of HCB. HCB is present in particulate and vapor form in air samples taken from within the plant area. The HCB detected in the downwind air samples beyond the plant perimeter was present mainly as a vapor. There appears to be a relatively constant air concentration of HCB vapor of approximately 0.1 to 1.0 $\mu g/m^3$ even in the upwind air samples. This background level of HCB in air may be due to the HCB present in the general plant area soil and landfill. The variation in HCB levels over the sampling period was due primarily to variations in HCB detected in particulates. HCB concentrations in soil (excluding the "Hex Pit" area) ranged from 1 to 1,500 ppm. The water samples taken beyond the plant area from Cowskin Creek contained very low levels of HCB (parts per trillion range). Downstream levels of HCB were twice as high as upstream levels.

LINDEN CHLORINE PLANT, LINDEN, NEW JERSEY

The survey of Linden Chlorine Plant was conducted on May 29, 1975. During the intended presampling survey, it was apparent that an extended air sampling plan was not warranted. Therefore, sediment, water, and soil samples were taken during the survey.

The Linden Plant was selected as a tentative sampling site because graphite electrodes were used in the production of chlorine and the plant produced a single product. During our visit we learned that graphite electrodes had been phased out at the end of March 1975.

Seven water and four solid samples were analyzed for HCB. The results are listed in Table 8. HCB was detected in the spent brine at 0.34 μg/liter. Based on the instrumental detection limit for HCB, 2 pg (2×10^{-12} g); the volume of water extracted, 1 liter; and the final extract volume, 50 ml; the minimum detectable concentration of HCB in water was 10 parts per trillion.

Table 7. HCB CONCENTRATIONS IN WATER FROM VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Samples	Volume sampled (liter)	Concentration (µg/ℓ) HCB
"Hex Pit" water	0.315	306
Solar Pond	0.335	0.7
Upstream (Cowskin Creek)	323	0.009
Downstream (Cowskin Creek)	365	0.018

ND - None detected.

Table 8. HCB CONCENTRATIONS IN WATER AND SOLIDS FROM LINDEN CHLORINE COMPANY, LINDEN, NEW JERSEY

Sample	Concentration (µg/ℓ) HCB
Water	
Holding pond, inlet	ND
Holding pond, outlet	ND
GAF weir, upstream of Cl_2 plant	ND
Waste stream, downstream of Cl_2 plant	ND
Process water	ND
Tap water	ND
Spent brine water	0.34
Solid	**Concentration (µg/g)**
Holding pond, settled and suspended	0.1
Dredged solids adjacent to holding pond	0.6
Waste stream, downstream of Cl_2 plant	7.6
Soil, around one of the cell buildings	1.7

All four solid samples contained HCB (from 0.1 to 7.6 μg/g). The highest level was observed in the waste stream sludge, downstream of the plant. The HCB level in soil outside a cell building was 1.7 μg/g.

Due to the complexity, i.e., large number of peaks, of these chromatograms, selected samples were fortified with standards to confirm the presence of HCB. In addition, the samples were prepared and analyzed in duplicate or in triplicate.

Plant Summary

Air samples were not taken at the Linden Chlorine Plant because graphite electrodes had been phased out prior to our sampling. HCB was detected in spent brine. The highest concentration of HCB (7.6 μg/g) was found in the sludge taken from the waste downstream of the plant. The levels detected in the water and solid samples indicate this plant is not a current source of significant quantities of HCB.

STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Field sampling at Stauffer Chemical Company's perchloroethylene plant at Louisville, Kentucky, was conducted on June 12, 1975. A total of 108 air, (54 filters and 54 Tenax®-GC columns) 5 soil, 3 sediment, and 6 water samples was collected. The results of the analysis of these samples are discussed below.

Air Samples

The 108 air samples were collected from nine samplers which were positioned at nine locations surrounding the plant. The positioning of downwind samplers was limited by a flood wall along the Ohio River.

Sampling at these nine locations was divided into six 4-hr periods and all samplers were operated 2 hr of each 4-hr period. After each 4-hr period, the filters and the Tenax®-GC columns in the samplers were replaced. The sampling was conducted so that the analytical results would indicate (a) the sources and levels of HCB, (b) the diurnal and plant operational effects, if any, and (c) the physical form of HCB in the air.

Sources and Levels of HCB Emission - The results of the analyses are listed in Table B-2 of Appendix B. The filters collected at the same sampling station at various sampling times were combined to form one sample to reduce the analysis time. The combined filter analysis is still indicative of the specific form of the two substances present in the plant air. The average HCB concentration per 24-hr period at each sampling station is shown in Figure 11.

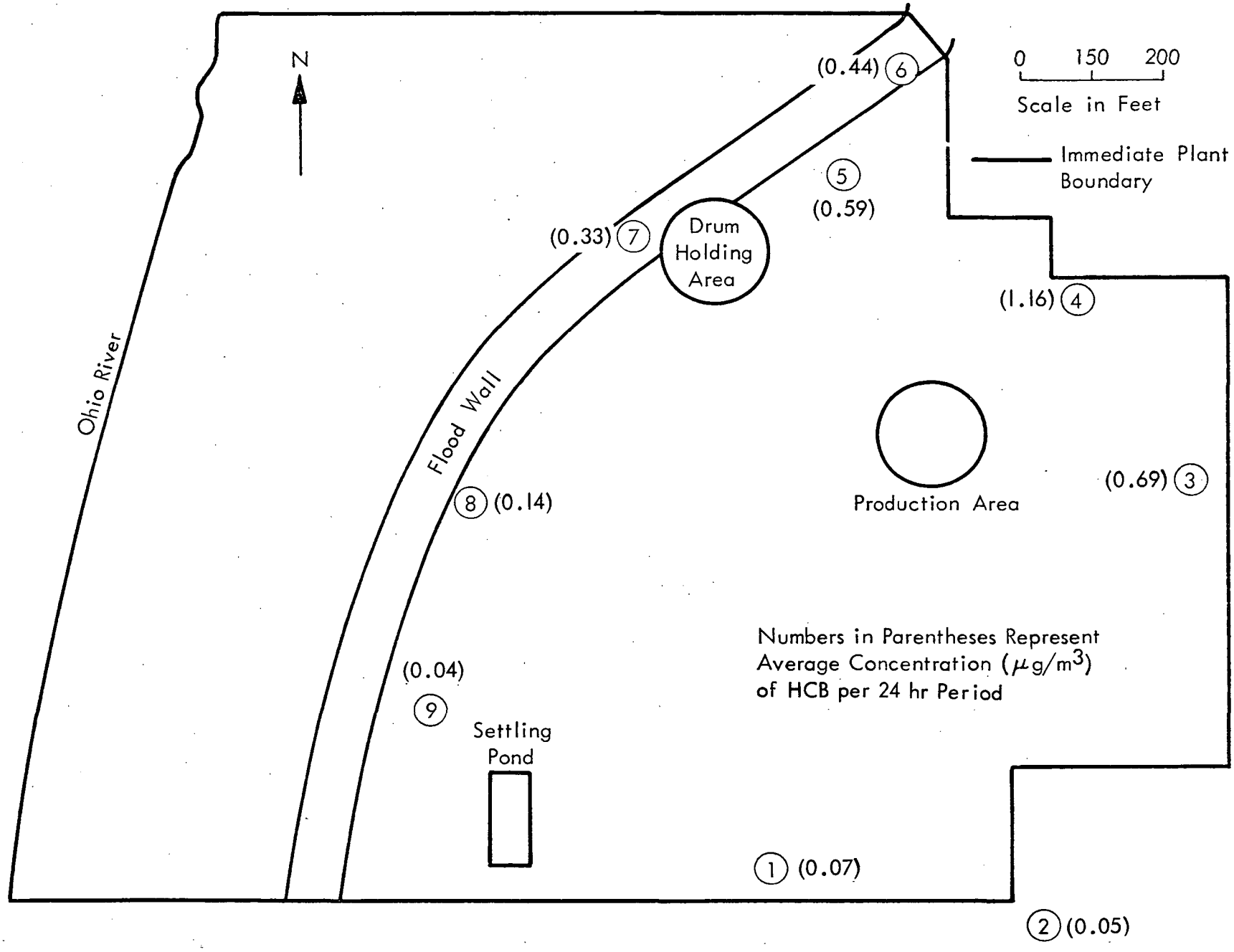

Figure 11. Average concentrations of HCB in air at nine sampling stations at Stauffer Chemical Company, Louisville, Kentucky

The 24-hr average concentrations on the filter and in Tenax®-GC resin at each sampling station are shown in Figure 12. The average HCB concentration in upwind samples (Stations 1 and 2) were 0.07 and 0.05 $\mu g/m^3$, respectively, while downwind samples ranged from 0.04 to 1.2 $\mu g/m^3$. Of the downwind stations, the highest level of HCB was observed at Station 4 which was located downwind from the perchloroethylene-carbon tetrachloride plant. The level of HCB was somewhat lower at Station 6, which was further downwind from the perchloroethylene-carbon tetrachloride plant. Stations 3 and 7 also show significant levels of HCB.

Variation of HCB Emission with Time - HCB levels detected during the 24-hr sampling period are plotted versus sampling time for each of the nine sampling stations in Figure 13.

Higher levels of HCB were observed during the first two sampling periods (1000 to 1800 hr) which may be indicative of a diurnal effect.

The removal of the solid waste drums from the plant area occurred during the latter part of the first sampling period and early part of the second (1400 to 1500 hr). HCB levels were uniformly high during this period. The exact time when the drums were removed from the drum loading area and transported off-site was not known.

Physical Form of HCB - There is some contribution to the total HCB levels from particulates collected on the filters at Stations 3 through 6. Surprisingly, HCB was not detected on the filters at Station 7, which was near the drum loading area. Although particulate HCB was not detected, settled particulates could contribute to the HCB level through the solid-vapor equilibrium of HCB-contaminated particulates. HCB has a boiling point of 230°C, but it co-distills with water vapor at low temperature and readily sublimes. It is apparent from Figure 12 that the major portion of HCB in the air was in the vapor state, since all the stations showed higher levels in the Tenax®-GC resin than on the filters.

Soil and Sediment Samples

Two soil samples were collected along the plant boundaries, one along the waste transportation route, and two from near waste handling areas; three sediment samples were taken, two from the Ohio River and one from the holding pond.

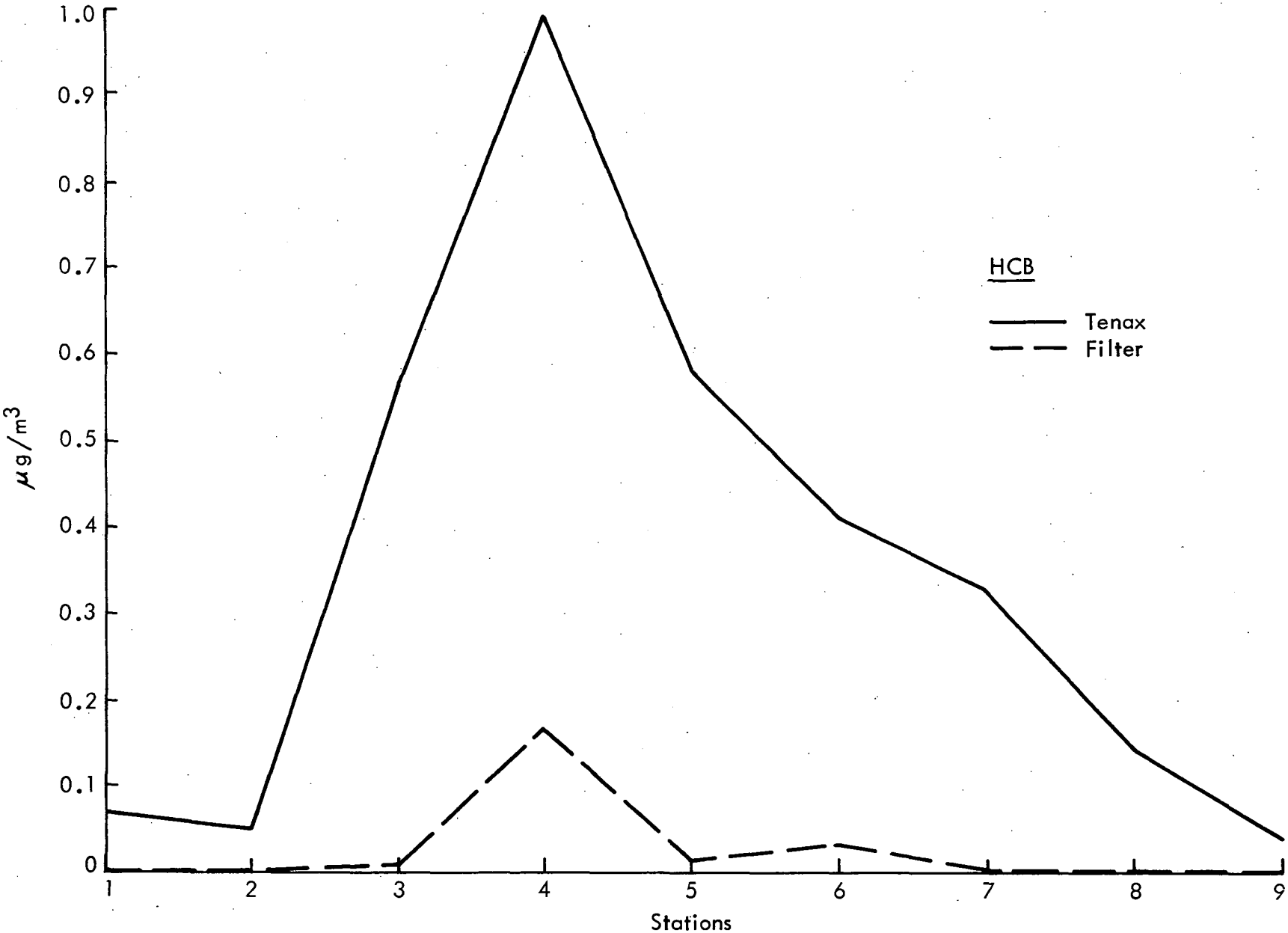

Figure 12. Average concentration of HCB in vapor and particulate at nine sampling stations (Stauffer)

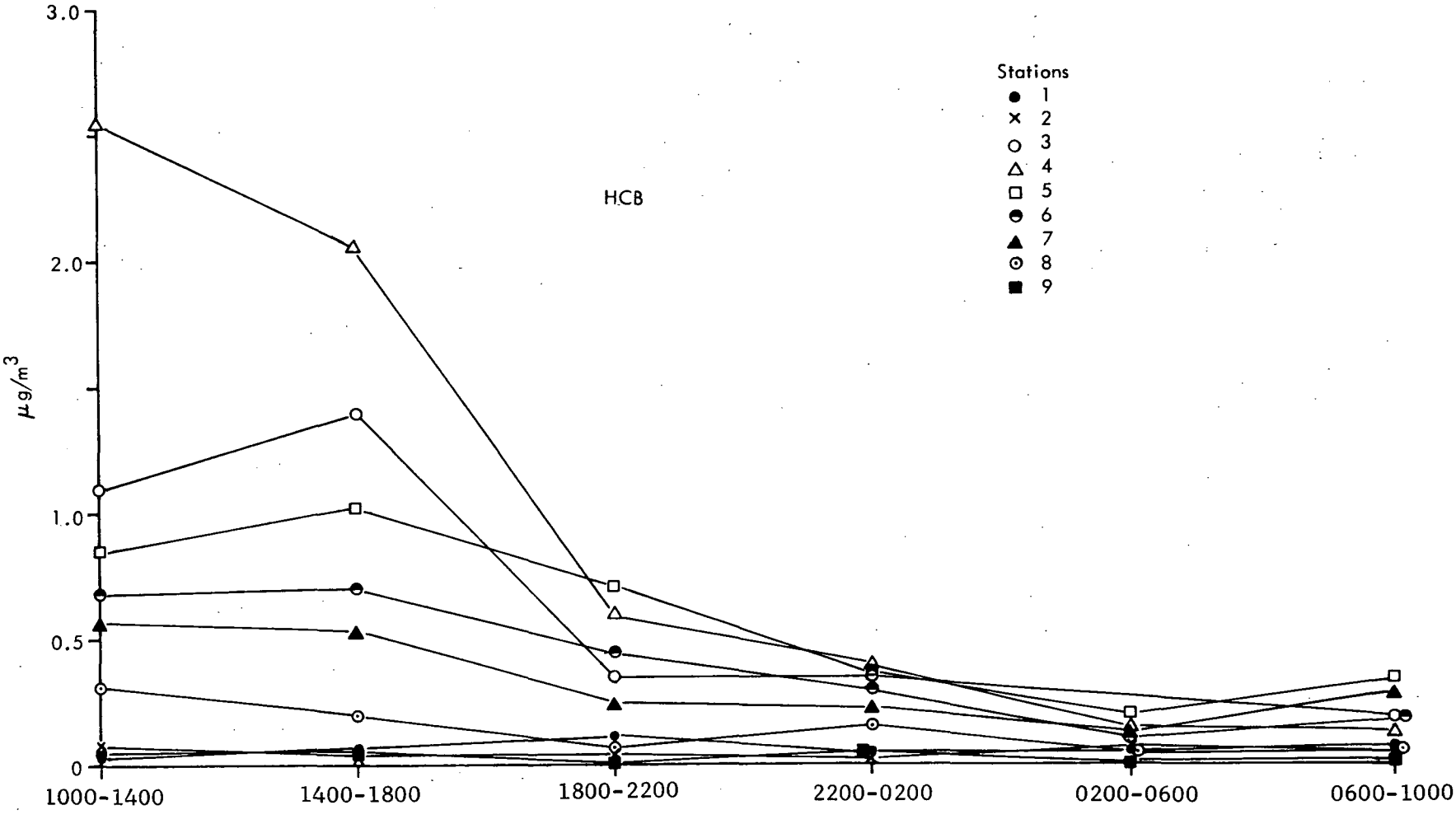

Figure 13. HCB concentration per 24-hr period at sampling stations around the plant (Stauffer)

The results, shown in Table 9, indicate that HCB soil concentrations were generally in the low parts per million range with the exception of the soil sample around the drum loading area (S-3), which contained 5,700 ppm HCB.

The soil concentrations followed the same general pattern as the air concentrations, i.e., the upwind sample had the least amount of HCB, 0.25 μg/g; the downwind sample (northern plant boundary) contained 4.75 μg/g. Other soil samples--the settling pond, main road, and northern plant boundary--show HCB levels from 5 to 20 times greater than levels found in the upwind samples.

Of the three sediment samples analyzed, the settling pond sample contained the highest level of HCB, 284 μg/g. The downstream HCB concentration was higher (0.05 μg/g) than the upstream concentration (0.008 μg/g). However, both concentrations are near the detection limit where the relative error in analysis is high.

Water Samples

Six water samples were collected--one from the plant well and five from the settling pond. The results are listed in Table 10. HCB was present in the plant well water below the parts per billion level, i.e., 0.2 μg/liter. The HCB concentration in the settling pond (grab) after treatment, i.e., the sample collected at the outlet, was half the HCB concentration in the sample taken from the inlet (grab). However, data from the 24-hr composite sample (collected by Stauffer) indicate that almost all HCB was eliminated after treatment. The XAD-4 results agree quite closely with the results obtained for the outlet grab sample.

Generally, the 24-hr composite sample is more representative. However, in the samples analyzed here, it is very likely that some HCB was lost during sampling for the 24-hr composite. The agreement between results obtained for the Amberlite XAD-4 resin and the grab sample substantiate their validity.

Plant Summary

The results of the analyses of all air samples indicate that the carbon tetrachloride-perchloroethylene plant is the major source of HCB in the general plant area. Slightly elevated levels of HCB were also observed on the eastern and western boundaries of the plant. The source of this contamination is very likely from vapor-solid equilibrium of the particulate "fall out" accumulated on the soil. The background level of

Table 9. HCB CONCENTRATIONS IN SOIL AND SEDIMENT FROM STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Samples	Concentration (µg/g) HCB
Soil	
S-1 - Upwind (southern plant boundary)	0.25
S-2 - Plant road	2.25
S-3 - Drum loading area	5,700
S-4 - Downwind (northern plant boundary)	4.75
S-5 - Settling pond area	1.3
Sediment	
R-1 - Settling pond	284
R-2 - Ohio River (upstream)	0.008
R-3 - Ohio River (downstream)	0.055

Table 10. HCB CONCENTRATION IN WATER FROM STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Samples	Concentration (µg/ℓ) HCB
Plant well water	0.2
Settling pond inlet (grab)	15
Settling pond outlet (grab)	7
Settling pond inlet (24-hr composite)	35
Settling pound outlet (24-hr composite)	0.7
Settling pond outlet (Amberlite XAD-4)	7

HCB in the air was 0.06 $\mu g/m^3$. Average concentrations of HCB on the millipore filter versus the Tenax®-GC resin indicate that the major portion of HCB in air was in the form of vapor rather than particulate. The HCB concentration was highest in soil near the "drum loading area"--5,700 $\mu g/g$. This level indicates a localized contamination from solid waste handling. Otherwise, HCB concentrations from 0.25 to < 5 $\mu g/g$ were observed in other soil samples around the plant. A slight buildup of HCB levels was observed in samples taken along the waste transportation route. Sediment samples from the settling pond showed high levels of HCB, 284 $\mu g/g$, but levels observed in samples taken from the Ohio River were insignificant. Results for grab samples collected at the settling pond outlet show excellent agreement with samples collected through Amberlite XAD-4 resin; 7 μg/liter of HCB for both.

DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA

Field sampling at Dow Chemical Company's carbon tetrachloride-perchloroethylene plant at Pittsburg, California, was conducted on August 7, 1975. A total of 24 air, including 8 filters and 16 Tenax®-GC columns, 3 soil, and 1 water sample was collected.

Air Samples

The 24 air samples were collected from 8 samplers, 2 of which were placed upwind at the western plant boundary, 3 at the midplant area, immediately downwind from the production and incinerator areas, and 3 farther downwind at the eastern plant boundary. Because no significant diurnal effects were observed at the Vulcan and Stauffer plants where air sampling was conducted in discrete 4-hr periods, the sampling at Dow was conducted for an integrated 24-hr period. Each sampling train was set up with two Tenax®-GC columns in tanden to check for possible breakthrough of HCB. The samplers were positioned so that results of the analysis would indicate (a) the sources and levels of HCB emission, (b) the physical form of HCB, and (c) the efficiency of HCB collection.

Sources and Levels of HCB Emission - The results are presented in Table B-3 of Appendix B. The average HCB concentration at each station is shown in Figure 14. The upwind (Stations 1 and 2) concentration was 0.02 $\mu g/m^3$, while downwind concentrations ranged from 0.02 to 0.08 $\mu g/m^3$. The recorded wind direction during the sampling was primarily from the west.

The increase in HCB concentration observed for Stations 3 and 4 indicates the production area which includes the thermal oxidizer (incinerator) is a source of HCB emission. Elevated HCB levels did not extend beyond the plant boundary. HCB levels for the upwind Stations 1 and 2 are about the same as those at downwind Stations 6 through 8. The results indicate there is no widespread contamination of the plant area.

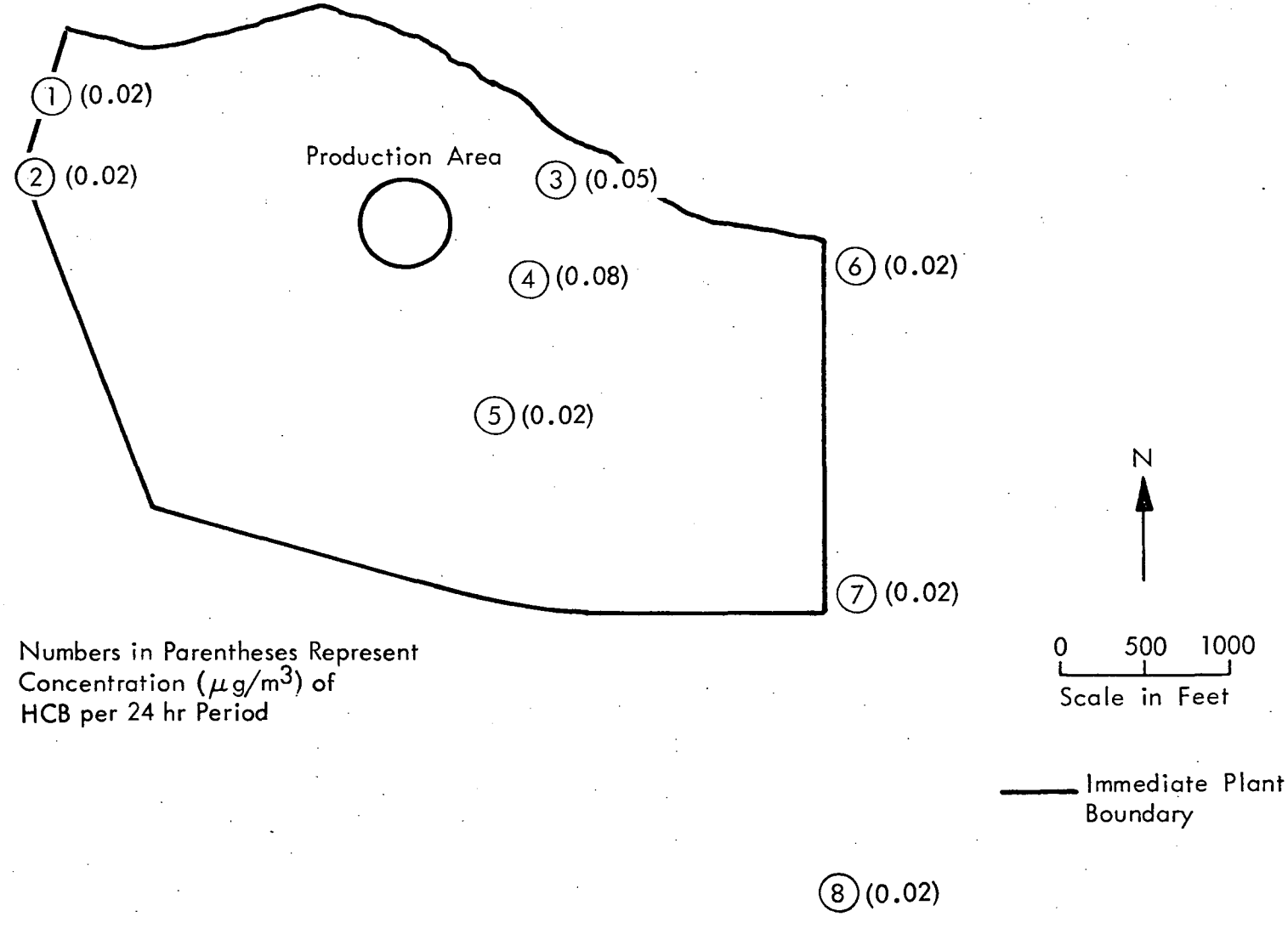

Figure 14. Average concentrations of HCB in air at eight sampling stations at Dow Chemical Company, Pittsburg, California

Physical Form of HCB - HCB was not detected on any of the filter samples. Since the instrumental limit of detection was 2 pg (2×10^{-12} g) for HCB, for an average of 4,000 liters of air sampled, the quantity of HCB collected in the form of particulates on each millipore filter was less than 10 ng. Therefore, HCB was present in the vapor form only.

HCB was not found in the back-up Tenax®-GC tube, indicating that it does not migrate through the Tenax®-GC resin.

Soil Samples

Three soil samples were collected along the eastern, western, and southern plant boundaries. The highest level (2.6 μg/g) was observed in soil from the southern boundary (Table 11). Without additional information on plant activities that might involve transportation of chlorinated hydrocarbons, we cannot explain the distribution of HCB in the soil samples. The relative concentrations do not coincide with wind direction.

Water Sample

One water sample was collected from the New York slough at the northwest corner of the plant. However, the sample was lost when the bottle containing the water was broken during shipping.

Plant Summary

The results of the analysis of air samples from the Dow plant indicate that the chlorinated hydrocarbon plant area which includes the carbon tetrachloride, tri- and perchloroethylene plants is a source of HCB. Relatively low levels of HCB, 0.02 $\mu g/m^3$, were detected in air at the eastern and western boundaries of the plant. Midplant HCB levels were slightly elevated over background. The absence of HCB on the millipore filter indicates that HCB was in the form of vapor rather than particulate in the plant air. The concentration of HCB was highest in the soil collected along the southern plant boundary. HCB concentrations of 0.22 μg/g or less were observed in the soils collected from the western and eastern plant boundaries.

E. I. DU PONT DE NEMOURS AND COMPANY, INC., CORPUS CHRISTI, TEXAS

Field sampling of Du Pont's carbon tetrachloride plant at Corpus Christi, Texas, was conducted on August 3, 1975. Because of generator failure, only five air sampling stations were in operation, which resulted in the collection of a total of 15 air samples. The generator failure limited the location as well as the number of samples taken. In addition to the air samples, 6 soil and sediment, and 7 water samples were collected.

Table 11. HCB CONCENTRATIONS IN SOIL FROM DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA

Samples	Concentration (μg/g) HCB
Western plant boundary	0.22
Eastern plant boundary	0.014
Southern plant boundary	2.6

Air Samples

The 15 air samples were collected from five samplers which were positioned at two upwind, and three downwind locations from the general production area. The samplers were operated continuously for a period of 24 hr, with two Tenax®-GC columns in tandem.

Levels of HCB - The results of the analysis are listed in Table B-4 of Appendix B. HCB was not detected in either the upwind or the downwind samples. Sampling locations are shown in Figure 15. Based on the instrumental limit of detection for HCB, 2 pg (2×10^{-12} g), and a 50-ml solution with a 10-μl injection for gas chromatographic analysis, the amount of HCB collected in a 4,000-liter sample was less than 10 ng.

Soil and Sediment Samples

The three soil samples were collected along the southern and northern plant boundaries, and near the landfill site. The sediment samples were collected at the storm runoff outfall, settling pond inlet and outlet. The HCB level observed in the landfill sample, 0.39 $\mu g/m^3$, was higher than the level in the upwind or downwind soil samples (Table 12).

In the sediment, the highest level of HCB was detected in the storm runoff outfall sample, 0.11 μg/g. HCB (0.01 μg/g) was detected in the settling pond outlet.

Water Samples

The seven water samples were collected from plant facilities where water was used either during the production process or for cleanup following production. Two types of sampling were conducted at the settling pond, grab sampling and sampling through an Amberlite XAD-4 column.

The results of the analysis of the seven samples are listed in Table 13. HCB was not detected in the raw plant water. The highest concentration was detected in the water standing in the landfill, 2.8 μg/liter. HCB was detected in the settling pond water, 0.037 μg/liter in the inlet and 0.015 μg/liter in the outlet. However, HCB was not detected in the grab samples taken at the inlet and outlet of the settling pond because only 1 liter of water was sampled versus 125 liters sampled through the Amberlite XAD-4 column.

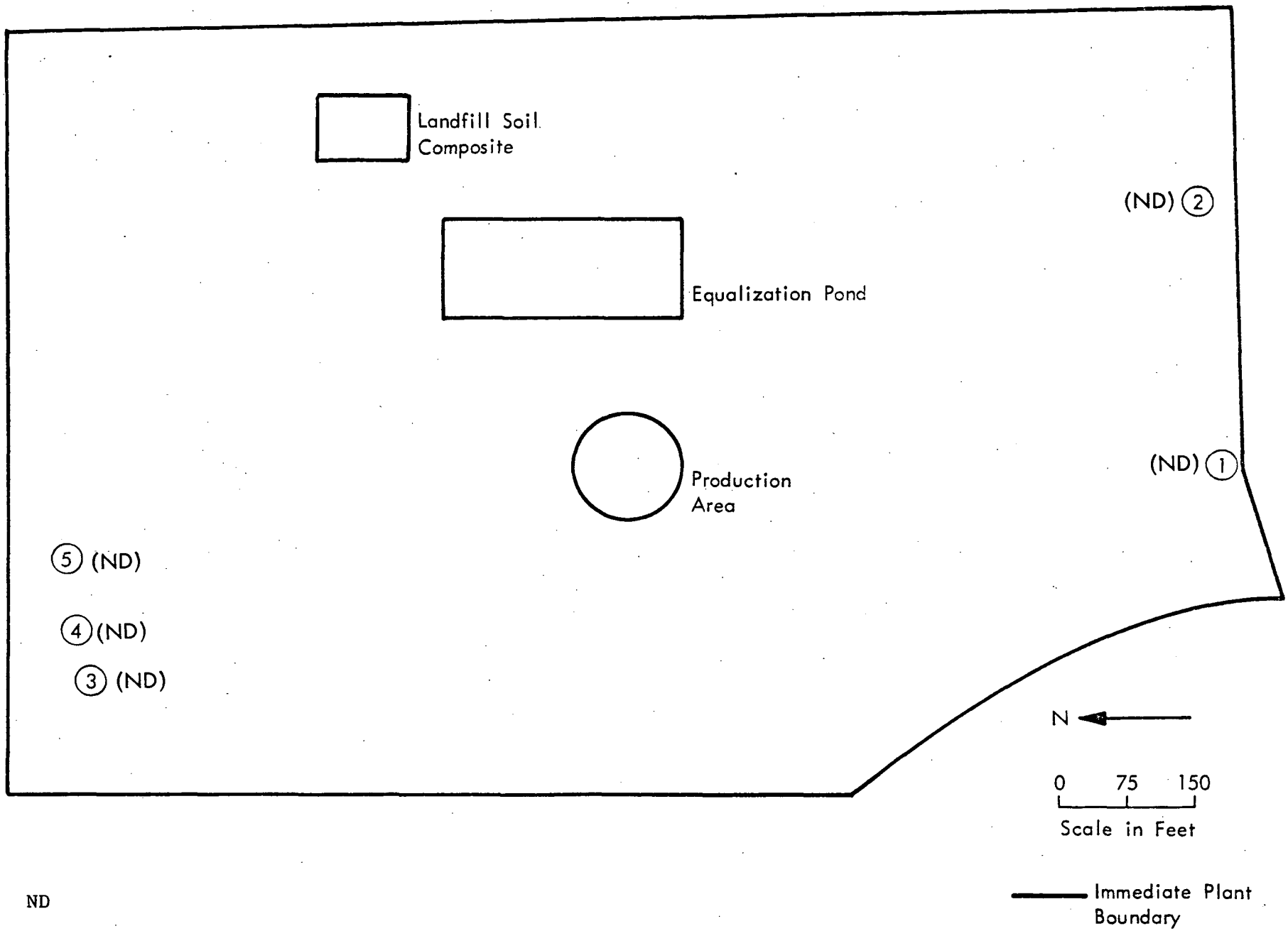

Figure 15. Sampling stations at E. I. du Pont de Nemours and Company, Corpus Christi, Texas

Table 12. HCB CONCENTRATIONS IN SOIL AND SEDIMENT FROM E. I. DU PONT DE NEMOURS AND COMPANY, CORPUS CHRISTI, TEXAS

Soil samples	Concentration (µg/g) HCB
Upwind (southern boundary)	0.16
Downwind (northern boundary)	0.015
Landfill area	0.39
Sediment	
Settling pond inlet	ND
Settling pond outlet	0.01
Storm runoff outfall	0.11

Table 13. HCB CONCENTRATIONS IN WATER FROM E. I. DU PONT DE NEMOURS AND COMPANY, CORPUS CHRISTI, TEXAS

	Samples	Concentration (µg/ℓ) HCB
W-1	Raw plant water before use	ND
W-2	Settling pond inlet (amberlite)	0.037
W-3	Settling pond inlet (grab)	ND
W-4	Settling pond outlet (amberlite)	0.015
W-5	Settling pond outlet (grab)	ND
W-6	Storm runoff outfall (grab)	ND
W-7	Water standing in landfill	2.8

Plant Summary

HCB was not detected in any of the air samples. The concentration of HCB was highest in the soil collected around the landfill area. Of the water samples, the highest concentration of HCB was detected in the landfill standing water. The landfill area is a source of elevated soil and groundwater HCB levels. In general, the levels of HCB in this plant were very low. The plant began operations as recently as 1973 and appears to be successful in minimizing HCB emissions.

DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

Field sampling at Diamond Shamrock Corporation's trichloroethylene plant at Deer Park, Texas, was conducted on August 20, 1975. A total of 24 air, including 8 filters and 16 Tenax®-GC columns, 3 soil, and 2 water samples was collected.

Air Samples

The 24 air samples were collected from eight sampling stations, five of which were positioned in a circle around the production area; two locations were along the north boundary and one at the south boundary of the plant area. Changing wind directions (TAble A-13) during sampling prevented the samplers from being positioned in upwind and downwind locations. The sampling was conducted over three 8-hr sampling periods covering 3 days. Rain interrupted the second sampling period and prevented sampling during 1200-1700 hr on any of the 3 days. The samplers were operated at 0.4 liters/min, resulting 600 liters or less of air sampled. Two Tenax®-GC columns were used in tandem.

Sources and Levels of HCB Emission - A simplified plant map with the sampling locations is shown in Figure 16. HCB was not detected in any of the samples.

Soil Samples

Three soil samples were collected from (a) along the northern plant boundary, (b) along the southern plant boundary, and (c) the production area.

The results of the analysis of the three soil samples are shown in Table 14. The highest concentration of HCB was detected in the production area, i.e., 24 μg/g. A higher level of HCB was detected in the soil sample collected near air sampling Station 1 (0.68 μg/g) than in the samples collected near Stations 7 and 8 (0.08 μg/g).

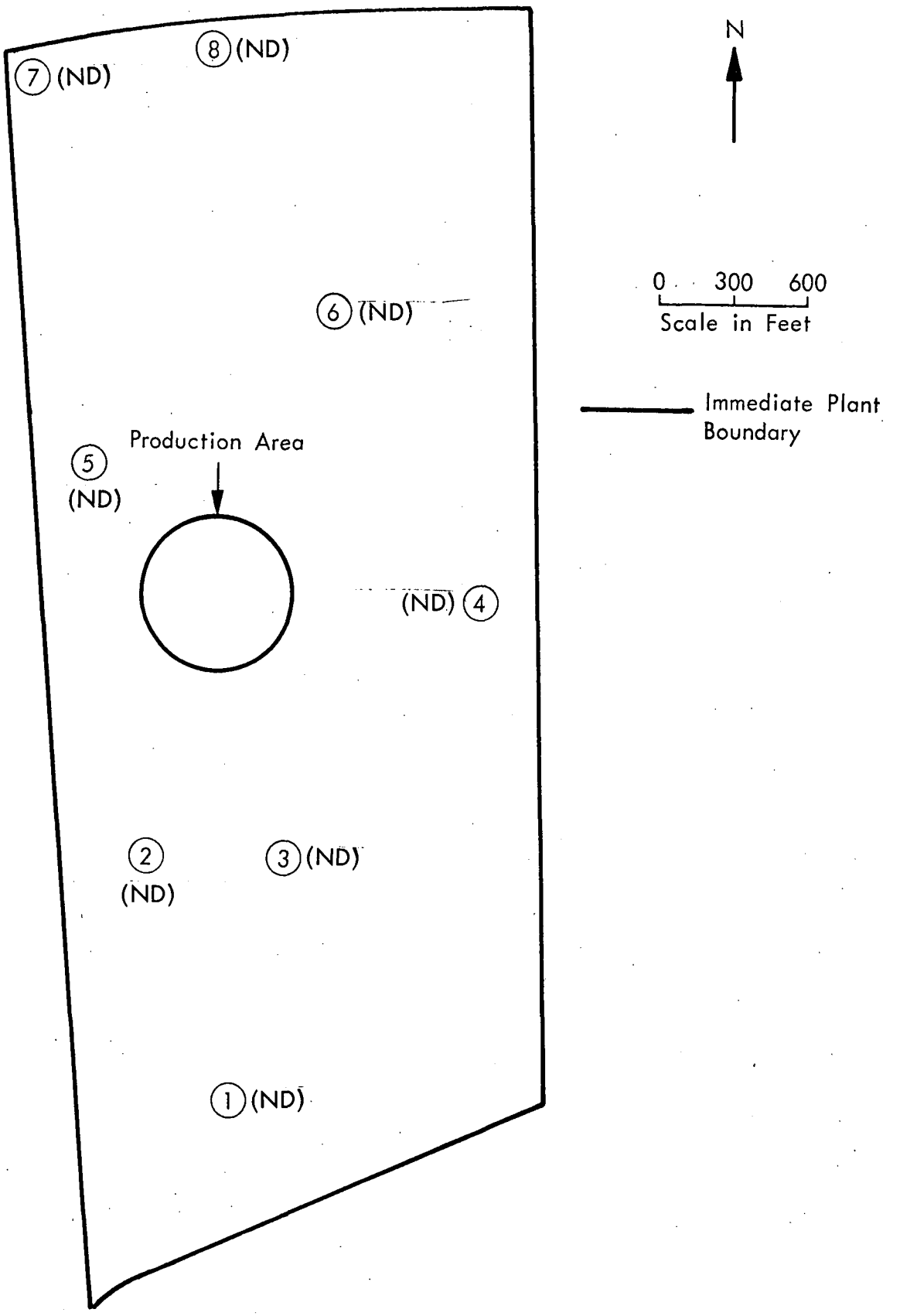

Figure 16. Stations at Diamond Shamrock Corporation, Deer Park, Texas

Table 14. HCB CONCENTRATIONS IN SOIL FROM DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

Soil samples	Concentration (µg/g) HCB
Upwind (southern boundary)	0.68
Downwind (northern boundary)	0.08
Production area	24.0

Water Samples

The two water samples collected for the analysis of HCB were the raw plant water and plant effluent. The results shown in Table 15 indicate that HCB was not detected in the raw water. However, 0.1 μg/liter of HCB was detected in the plant effluent. This effluent is discharged into nearby Pattrick Bayou.

Plant Summary

HCB was not detected in the air samples from any of the sampling stations. HCB was detected in soil samples collected along the northern and southern plant boundaries at parts per billion levels. The concentration of HCB in production area soil was 24 μg/g. The inlet plant water did not contain detectable quantities of HCB while the process plant effluent showed an HCB level of 0.1 μg/liter.

CIBA-GEIGY CORPORATION, ST. GABRIEL, LOUISIANA

Field sampling at Ciba-Geigy Corporation's triazine herbicide plant in St. Gabriel, Louisiana, was conducted on August 13, 1975. A total of 16 air, 4 soil, and 2 water samples was collected.

Air Samples

The 16 air samples were collected from eight samplers which surrounded the entire production area. Stations 2, 4, 6, and 8 were positioned about 400 ft, and Stations 1, 3, 5, and 7 were approximately 1,000 ft from the production area. One Tenax®-GC column was used in the sampling train. The sampling was conducted 2 hr of every 8-hr period, over a 24-hr period.

Sources and Levels of HCB Emission - The results of the analyses are listed in Table B-16 of Appendix B. The average concentrations of HCB are shown for each sampling station in Figure 17. HCB concentrations were from nondetectable to 0.02 $\mu g/m^3$. The wind directions were quite erratic during sampling, and upwind-downwind patterns were not observed. HCB in the plant air was in the form of vapor rather than particulate.

Soil Samples

Four soil samples were collected from the general areas of Air Sampling Stations 1, 3, 5, and 7, respectively. The results of the analysis are listed in Table 16. HCB was found in only two samples--around Air Sampling Stations 3 and 5, at 0.003 and 0.011 μg/g, respectively. The analysis of the soil samples collected near Station 7 was negative.

Table 15. HCB CONCENTRATIONS IN WATER FROM DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

Samples		Concentration (µg/ℓ) HCB
W-1	Raw unused plant water	ND
W-2	Plant effluent	0.1

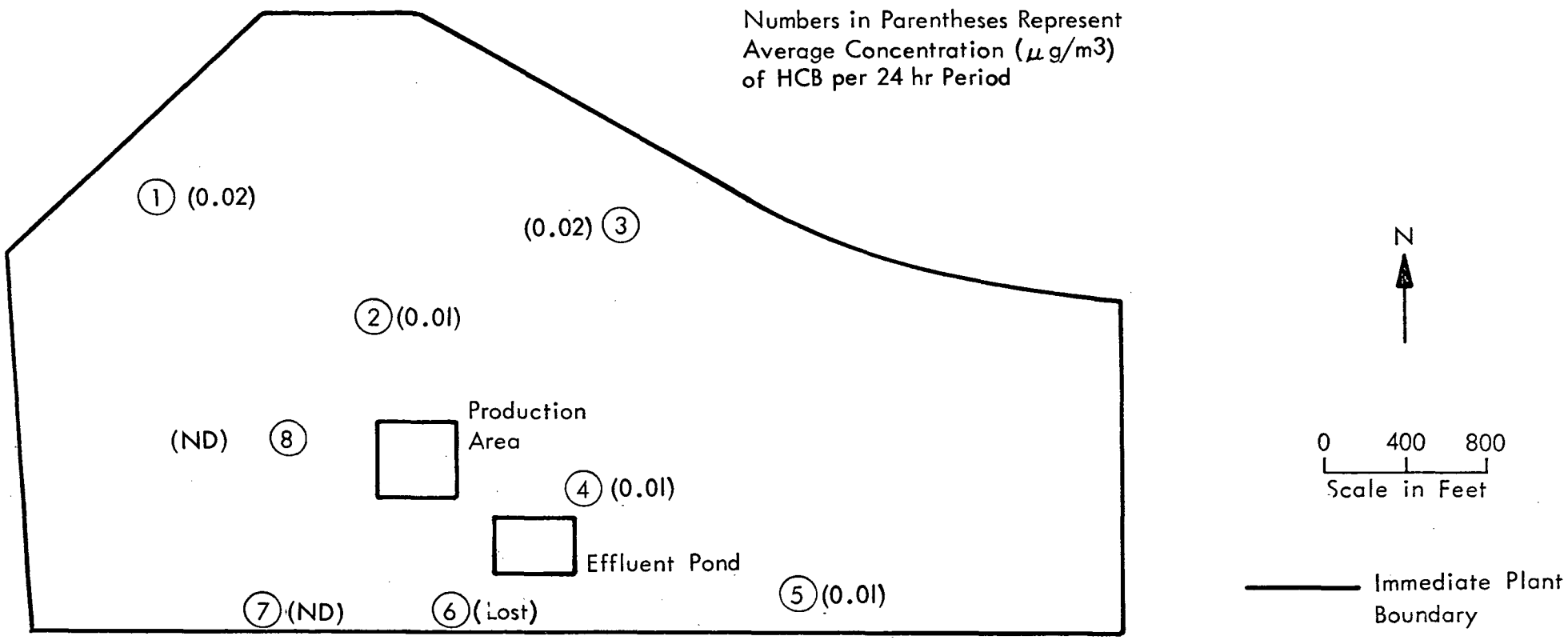

Figure 17. Average concentrations of HCB in air at eight sampling stations at Ciba-Geigy Corporation, St. Gabriel, Louisiana

Table 16. HCB CONCENTRATIONS IN SOIL FROM CIBA-GEIGY CORPORATION, ST. GABRIEL, LOUISIANA

	Soil samples	Concentration (µg/g) HCB
S-1	Northwest of plant at Air Sampling Site No. 1	ND
S-2	Northeast of plant at Air Sampling Site No. 3	0.003
S-3	Southeast of plant at Air Sampling Site No. 5	0.011
S-4	Southwest of plant at Air Sampling Site No. 7	ND

Water Samples

Two grab samples were collected, raw plant water and process effluent. HCB was not detected in either sample.

Plant Summary

The concentrations of HCB in the plant air were very low--from 0.01 to 0.02 $\mu g/m^3$. In the soil, HCB was detected in the low parts per billion range. Since HCB was not detected in the process effluent, the water discharged into the Mississippi River contains less than 10 ng/liter of HCB.

OLIN CORPORATION, MCINTOSH, ALABAMA

Field sampling at Olin Corporation's pentachloronitrobenzene (PCNB) plant at McIntosh, Alabama, was conducted on August 18, 1975. A total of 24 air, including 8 filters and 16 Tenax®-GC columns, 12 soil and sediment, and 10 water samples was collected. In addition to the PCNB plant, this facility included a chlorine production plant which used graphite electrodes in the production process.

Air Samples

The 24 air samples were collected from two samplers positioned at the southern boundary, three at the mid-plant area, three north of the PCNB production plant, and three at the northern boundary. Each sampling train consisted of a millipore filter and two Tenax®-GC columns. Essentially no breakthrough of HCB through the first column was observed. The samplers were operated 3 hr of each 8-hr period for three 8-hr periods. The wind direction during the sampling varied. Therefore, upwind-downwind stations could not be designated.

Sources and Levels of HCB Emission - The results of the air sample analysis are listed in Table B-7 of Appendix B. The sampling locations and average 24-hr HCB concentrations are shown in Figure 18; HCB concentrations ranged from 0.04 to 2.2 $\mu g/m^3$. From the map of the plant area shown in Figure 18, three possible sources of HCB are apparent: the PCNB production area, the "hex" storage area, and the chlorine production area. The highest concentrations of HCB were observed in the samples collected along the southern boundary. These samples were south-southwest of the "hex" storage area, which is in the southeast corner of the plant. The wind was recorded from almost all directions during the sampling, but the highest wind speed was observed when the direction was from the north-northeast and the north. Neglecting direction and notwithstanding Station 1, the closer the sampler was to the "hex" storage area, the greater the HCB concentration. These results indicate that the "hex" storage area is the primary source of HCB. Because of the varied wind

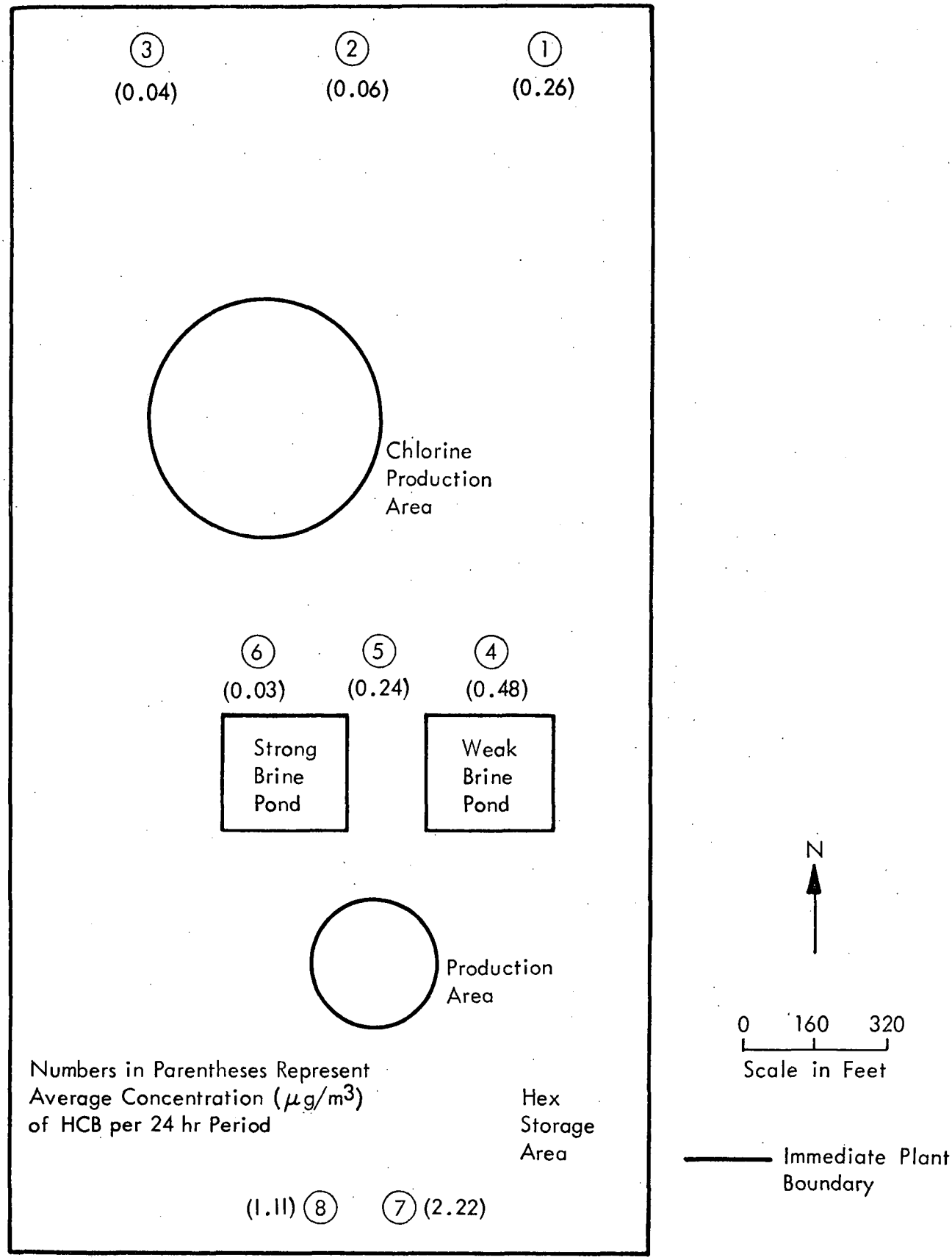

Figure 18. Average concentrations of HCB in air at eight sampling stations at Olin Corporation, McIntosh, Alabama

conditions, it is difficult to quantitatively determine the contribution of the PCNB production plant to HCB emissions but it appears to be much less than the "hex" storage area. The HCB emission from the chlorine production plant is negligible since the air concentrations at stations closest to the chlorine production area were from 0.03 to 0.06 $\mu g/m^3$.

Physical Form of HCB

HCB was not detected in any of the filters indicating that it was present as a vapor rather than in particulate form. The absence of particulate HCB is somewhat surprising since the heavy wastes from PCNB production are stored in solid blocks in the storage area. The blocks are covered with plastic sheets which appear to be effective in eliminating particulate HCB in the air but not HCB vapor. Sporadic rain and low wind speed during the sampling also may have reduced the particulate HCB.

Soil and Sediment Samples

The 12 soil and sediment samples were collected from plant boundaries, transportation routes, landfill and storage areas.

The results of the analysis of these samples are shown in Table 17. The concentration levels were from a low of 0.98 $\mu g/g$ (soil from the brine pond area) to a high of 13% (soil from the "hex" storage area). The blocks of "hex" wastes stored in this area contain up to 80 to 90% HCB. The soil sampled along the transportation route of the "hex" blocks showed an HCB concentration of 4,100 $\mu g/g$. HCB concentrations over 100 ppm were detected in samples collected along the road to the old landfill, and from within the old landfill. It was learned that prior to the practice of casting the "hex" wastes into blocks, the old landfill was used for the "hex" disposal.

Soil samples from the east road, as well as the "hex" storage area, old landfill and the current landfill area all show much higher levels of HCB than those detected on the west road. The relative concentrations of the air samples followed the same pattern.

Water Samples

Ten water samples were collected: two from ditches within the plant area, three from the nearby creek, one from the settling pond, two from the solar pond, and two from the two brine ponds (strong and weak).

Table 17. HCB CONCENTRATIONS IN SOIL AND SEDIMENT
OLIN CORPORATION, MCINTOSH, ALABAMA

Samples		Concentration (µg/g) HCB
Soil		
S-1	Northern boundary road	3,200
S-2	Old landfill (northeastern boundary)	480
S-3	Brine pond area	0.98
S-4	Center road (running north/south)	72
S-5	High-lift route (organic plant to storage area)	4,100
S-6	Southeast landfill	53
S-7	"Hex" storage area	13%
S-8[a/]	Old "Hex" dump area	Not analyzed
S-9	East road	400
S-10	West road	1.1
S-11	South road	350
Sediments		
Strong brine pond sediment		12

a/ This sample is mostly tar which is used to cover the general old "Hex" area; the extracted solution was so dirty that analysis was not possible even after cleanup.

The results are shown in Table 18. The highest level of HCB, 159 μg/liter, was detected in the spent brine pond. Concentrations ranged from nondetectable to less than 10 μg/liter in the other samples.

The relatively close agreement of the HCB concentrations determined in the single grab sample (5.0 μg/liter) versus the 24-hr composite sample (2.5 μg/liter) of the creek water collected 200 yd upstream of the basin indicates the instantaneous HCB concentration did not vary significantly from the 24-hr average. HCB was not detected in the basin water indicating that detectable amounts of HCB were not discharged into the Tombigbee River.

Plant Summary

The results of the analysis of air, soil, sediment, and water samples indicated that the "hex" storage area is the primary source of HCB contamination, whereas the PCNB production area, old landfill site, and weak brine pond are secondary sources. The chlorine production plant is not a source of HCBD. Soil and sediment samples showed relatively high levels of HCB--from 0.98 to 1.3 x 10^5 μg/g. All the plant road samples contained HCB: the lowest was on the west road (1.12 μg/g), the highest on the "high-lift road" (organic plant to storage area, 4.1 x 10^3 μg/g). Detectable quantities of HCB were not discharged into the Tombigbee River.

PPG INDUSTRIES, INC., LAKE CHARLES, LOUISIANA

Field sampling at PPG Industries' trichloroethylene and perchloroethylene plant at Lake Charles, Louisiana, was conducted on September 4, 1975. A total of 30 air, including 10 filter, 20 Tenax®-GC columns, 7 soil and sediment, and 7 water samples was collected.

Air Samples

The 30 air samples were collected from 10 samplers which circled the plant. The samplers were not positioned in an upwind-downwind array because varied wind direction was expected from information obtained during the presampling site visit. However, the wind was predominately from the east and east-southeast during sampling. Samplers were positioned to take advantage of existing electrical outlets, also. The sampling was conducted for an integrated 24-hr period; two Tenax®-GC columns in tandem were used.

Table 18. HCB CONCENTRATION IN WATER FROM OLIN CORPORATION, MCINTOSH, ALABAMA

Samples			HCB concentration (µg/ℓ)
W-1	Weak brine pond		160
W-2	Strong brine pond		1.4
W-3	Settling pond		ND
W-4	North/south running ditch		1.0
W-5	Southern ditch area (upper drop)		7.5
W-6[a/]	Combined creek (200 yard before basin)		5.0
W-7[a/]	Basin (at mouth of creek)		ND
W-8	24-Hr composite of plant effluent (combined creek)		2.5
W-9[a/]	Solar pond, west		3.8
W-10[a/]	Solar pond, east		ND
B-1	Sampling bottle blank		
B-2	Sampling bottle blank	Average	0.4
B-3	Sampling bottle blank		

a/ All water samples using Olin's sample bottles are reported after subtracting blank bottle value.

Sources and Levels of HCB Emission - The results are shown in Table B-8 in Appendix B. A simplified plant map with the sampling locations and the 24-hr average concentrations (Tenax®-GC plus filter) of HCB is shown in Figure 19. HCB concentrations were from nondetectable, at Stations 8 and 9, to 1.7 $\mu g/m^3$ at Station 7. Relatively higher concentrations of HCB were detected at Stations 4 through 7, which were located downwind of the incinerator-production area. The presence of low concentrations of HCB at Stations 2, 3, 9, and 10 could be due to other sources, e.g., the old landfill site and the barges which were located upwind.

The presence of HCB beyond the northern plant boundary is probably due to the incinerator since Station 6 is somewhat downwind. However, a relatively high level of HCB was detected at Station 1 which is upwind of the incinerator and plant area. The HCB level in this sample may indicate a general contamination of the area south of the plant. Sampling Station 6 was located in a sparsely populated residential area.

Physical Form of HCB - Figure 20 shows that HCB was predominantly in the form of particulate rather than vapor (majority detected on the filter).

The occurrence of HCB in particulate form could be the results of (a) particulates from the incinerator or (b) the production process employed by PPG for perchloroethylene and trichloroethylene. PPG uses a catalytic oxychlorination technique, resulting in a lower reaction temperature than that generated by the thermal chlorination of hydrocarbons.[6/]

Soil Samples

Four soil samples were collected: two at the plant boundaries, one along Mobil South Road, and one from the landfill. The results are shown in Table 19. The levels of HCB in the soil ranged from 0.015 $\mu g/g$, around Air Sampling Stations 8 through 10, to 0.10 $\mu g/g$, along Mobil South Road and around Air Sampling Stations 4, 5, and 7.

The elevated levels of HCB at Stations 4, 5, and 7 are consistent with a prevailing east and east-southeast wind resulting in particulate fallout and vapor condensation from the incinerator and production plant. The concentration detected in the Mobil South Road composite sample indicates the PPG canal is a possible source of HCB. The results of sediment and water analyses discussed below support this possibility.

Sediment Samples

The three sediment samples were taken one each from the PPG canal (near Air Sampling Station 1), the ship channel, and the main effluent from the organic plant. HCB was detected in all three sediment samples; the lowest level (0.01 $\mu g/g$) was in the ship channel, and the highest in the sediment at the organic plant effluent (0.87 $\mu g/g$). The presence of HCB in the ship channel sediment may be associated with waste loading into the barges.

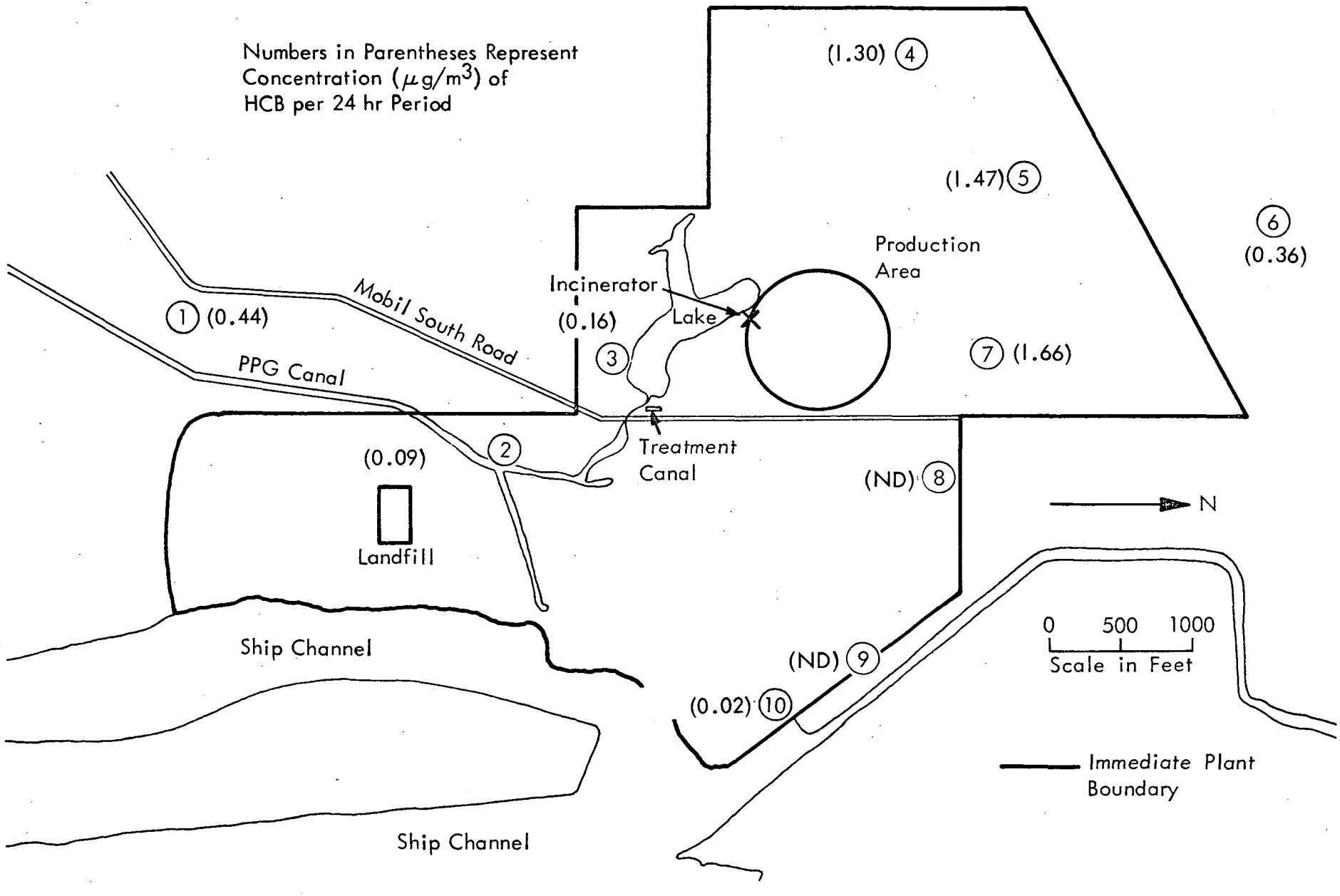

Figure 19. Average concentrations of HCB in air at 10 sampling stations at PPG Industries, Lake Charles, Louisiana

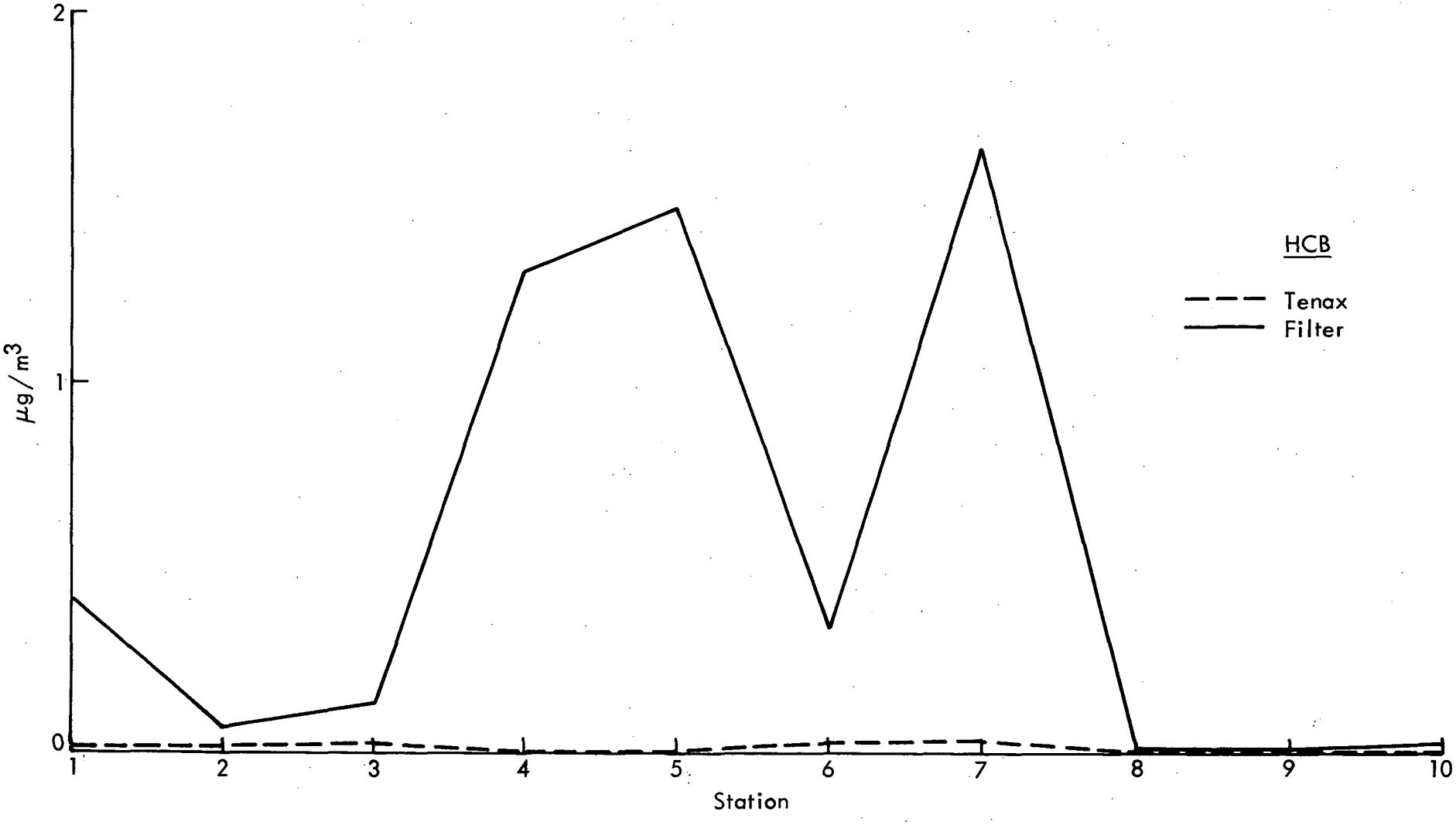

Figure 20. Average concentrations of HCB in vapor and particulate at 10 sampling stations around the plant (PPG Industries)

Table 19. HCB CONCENTRATIONS IN SOIL AND SEDIMENT FROM PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

Samples	Concentration (µg/g) HCB
Air Stations 4, 5, 7 soil composite	0.10
Air Stations 8, 9, 10 soil composite	0.015
Mobil south road	0.10
Landfill	0.025
PPG sediments	
Sediment 1 (downstream PPG canal)	4.4
Sediment 2 (main organic plant effluent)	6.9
Ship channel sediment	0.01

The presence of significant amounts of HCB (4.4 μg/g) in the downstream PPG canal sediment indicates accumulation of deposits from the organic plant effluent. This sediment was collected 1,000 ft beyond Air Sampling Station 1, i.e., at least 4,000 ft from the organic plant.

Water Samples

Seven water samples were collected: one from the lake (incinerator feed), one incinerator scrubber, two from the treatment canal, one from the landfill (standing water), one from the PPG canal, and one from the ship channel.

The results are shown in Table 20. The highest concentration of HCB (7.1 μg/liter) was detected in the treatment canal inlet sample, and the landfill surface water. The treatment canal outlet contained 4.1 μg/liter HCB, indicating that the treatment removed about 40% of the HCB. The sample collected downstream of the PPG canal near Air Sampling Station 1 contained 1.1 μg/liter HCB. This concentration in the canal water at more than 3,000 ft downstream from the effluent point is consistent with the levels detected in sediment collected 1,000 ft further downstream.

The lake water (incinerator feed water) contained 0.22 μg/liter HCB. This level of HCB could, depending on the amount of feed water versus "liquid bottoms" that passes through the incinerator, contribute significantly to the HCB emission from the incinerator.

The scrubber water from the incinerator contained a lower level of HCB (0.09 μg/liter) than the feed water and the treatment canal inlet.

Plant Summary

The primary source of HCB in air is the incinerator and organic plant. The HCB present in the incinerator feed water (0.22 ng/liter) may be a factor in HCB concentrations in air. The HCB was present predominantly as particulate in air. This plant was unique in the relative distribution of HCB in air versus soil, sediment and water. The air concentrations were relatively high at this plant when considering the relatively low concentrations in the other types of samples. The distribution of HCB may be accounted for by the plant production and waste disposal methods. Solid wastes are not formed in the low temperature catalytic oxychlorination production process. "Liquid bottoms," which contain HCB, are incinerated; process water, which contains lighter chlorinated hydrocarbons, e.g., HCBD, is treated and flows into the PPG channel and eventually the Calcasieu River. Water sampled in the PPG canal at a point approximately 3,000 ft from the organic plant effluent contained 1 ppm HCB. The HCB concentration in a sparsely populated residential area downwind of the incinerator was 0.36 $\mu g/m^3$.

Table 20. HCB CONCENTRATIONS IN WATER FROM PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

Sample	Concentration (µg/ℓ) HCB
Incinerator feed water (lake water)	0.22
Scrubber water	0.09
Inlet (treatment canal)	7.1
Outlet (treatment canal)	4.12
Surface water (landfill)	7.1
Downstream PPG canal (Mobil Bridge No. 1)	1.1
Ship channel (next to Air Station No. 10)	ND

Note: ND = none detected.

SECTION VI

SEWAGE TREATMENT FACILITIES

Chlorination as a means of disinfecting treated sewage has been known to result in the "in situ" synthesis of numerous halogenated hydrocarbons. It was not known whether HCB is produced in significant concentrations by this process. For this reason, samples were collected from two sewage treatment plants utilizing chlorination and analyzed for HCB.

SAMPLING

Samples were collected from two sewage treatment facilities in the Kansas City area. Both facilities utilize chlorination for disinfecting the treated sewage. One-gallon samples were collected from the sewage treatment stream immediately before and after the chlorination step. The samples were returned to MRI and stored at 4°C until analyzed.

EXPERIMENTAL PROCEDURES

A 1-liter sample of the sewage treatment effluent was extracted twice with 100 ml of 15% (v/v) ethylether in hexane and once with 100 ml of hexane. The extracts were combined and dried by passage through Na_2SO_4. The sample volume was reduced to 5 ml by means of a Kuderna-Danish evaporator. One milliliter was reserved and the remaining 4 ml were reduced to 1 ml using a slow stream of N_2 to evaporate the solvent. The samples were analyzed using electron capture gas chromatography. The column was 6 ft x 1/4 in. o.d. packed with 4.0% SE-30 and 6.0% OV-210 on Chromosorb W HP. The operating conditions were as follows: injector temperature, 220°C; column temperature, 200°C; detector temperature, 250°C; and carrier gas, 17 ml/min.

RESULTS

Analysis of both sewage samples collected ahead of the chlorination facilities showed no significant peaks at the retention time of HCB. Based upon the instrumental sensitivity toward HCB and the concentration enhancement by extraction and evaporation, the sewage samples contained less than 1 part per trillicn HCB.

The samples collected after the chlorination facility gave highly complex gas chromatograms. Numerous peaks were present at and near the retention time of HCB. Assuming that the observed peak at the retention time of HCB was due solely to HCB, the sewage samples contained a maximum of 4 to 8 ppt HCB. As this was undiluted sewage, it appears that chlorination of raw sewage produces a negligible quantity of HCB.

SECTION VII

SUMMARY AND CONCLUSIONS FOR PROGRAM TASK IA

SUMMARY

A summary of results is listed in Table 21. The high and low concentrations of HCB are listed for each type of sample, along with the products and waste-disposal methods for each site.

Figure 21 shows the highest levels of HCB in air and the levels detected in the samples taken the greatest distance downwind from the suspected source(s) at each plant. Sampling distances from each source are shown in parentheses. The concentrations shown in Figure 21 should not be compared directly because of differences in sampling distances, meteorological conditions, etc., during sampling.

In general, higher concentrations of HCB were associated with the production of perchloroethylene and trichloroethylene than with other industries. However, most of the chlorinated hydrocarbon plants produced a combination (perchloroethylene, trichloroethylene, carbon tetrachloride, etc.) of products which makes it difficult to extrapolate the results obtained at a particular plant to a single product. In the one plant that produced only carbon tetrachloride, the HCB levels were quite low. The HCB concentrations detected in samples from the pentachloronitrobenzene production plant were relatively high, i.e., $\mu g/m^3$ range in air. The levels of HCB associated with plants producing chlorine and triazine herbicides were very low.

Several different waste-disposal methods were used at the perchloro- and trichloroethylene plants that were sampled. They included off-site and on-site landfill combined with open pit or pond storage and off-site and on-site incineration. Higher levels of HCB were detected in air and soil at the plant using on-site landfill and open pit storage. The levels of HCB detected in soil and samples taken upwind of suspected point sources indicated a wide area of HCB contamination at this plant. Elevated HCB levels were detected in loading and transfer areas at plants using off-site

Table 21. DATA SUMMARY FOR PROGRAM TASK NO. 1

Company	Products	Substance	Air (μg/m³) High	Air (μg/m³) Low	Water (μg/ℓ) High	Water (μg/ℓ) Low	Soil (μg/g) High	Soil (μg/g) Low	Sediment (μg/g) High	Sediment (μg/g) Low	Waste disposal
Vulcan Materials Company Wichita, Kansas	Perchloroethylene Carbon tetrachloride Chlorine	HCB	24	0.53	300	0.009	5%	1.1	No sample		On-site landfill, and deep well
Stauffer Chemical Company Louisville, Kentucky	Perchloroethylene Carbon tetrachloride Methylen chloride Chloroform, chlorine	HCB	7.0	0.24	35	0.2	5,700	0.25	280	0.008	Off-site landfill
Dow Chemical Company Pittsburg, California	Perchloroethylene Carbon tetrachloride Chlorine	HCB	0.08	< 0.02	No sample		2.61	0.014	No sample		Incineration
E. I. du Pont de Nemours Corpus Christi, Texas	Carbon tetrachloride	HCB	ND	ND	2.8	ND	0.39	0.015	0.11	ND	On-site landfill and off-site disposal
Diamond Shamrock Deer Park, Texas	Trichloroethylene Perchloroethylene Chlorine	HCB	ND	ND	0.1	ND	24	0.08	No sample		Off-site incineration
Olin Corporation McIntosh, Alabama	Pentachloronitrobenzene Chlorine	HCB	2.2	0.03	160	ND	13%	0.98	12.4	Only one sample	Solid wastes (in blocks) stored in open field covered with plastic
Ciba-Geigy Corporation St. Gabriel, Louisiana	Atrazine Propazine Simazine	HCB	0.02	ND	ND	ND	0.01	ND	No sample		Off-site incineration
PPG Industries Lake Charles, Louisiana	Trichloroethylene Perchloroethylene Vinyl chloride Vinylidene chloride Chlorine, etc.	HCB	1.7	ND	7.1	ND	0.10	0.015	69	0.01	Incineration, landfill, and treatment canal
Linden Chlorine Linden, New Jersey	Chlorine	HCB	No sample		0.34	ND	1.7	Only one sample	7.6	0.10	Holding pond

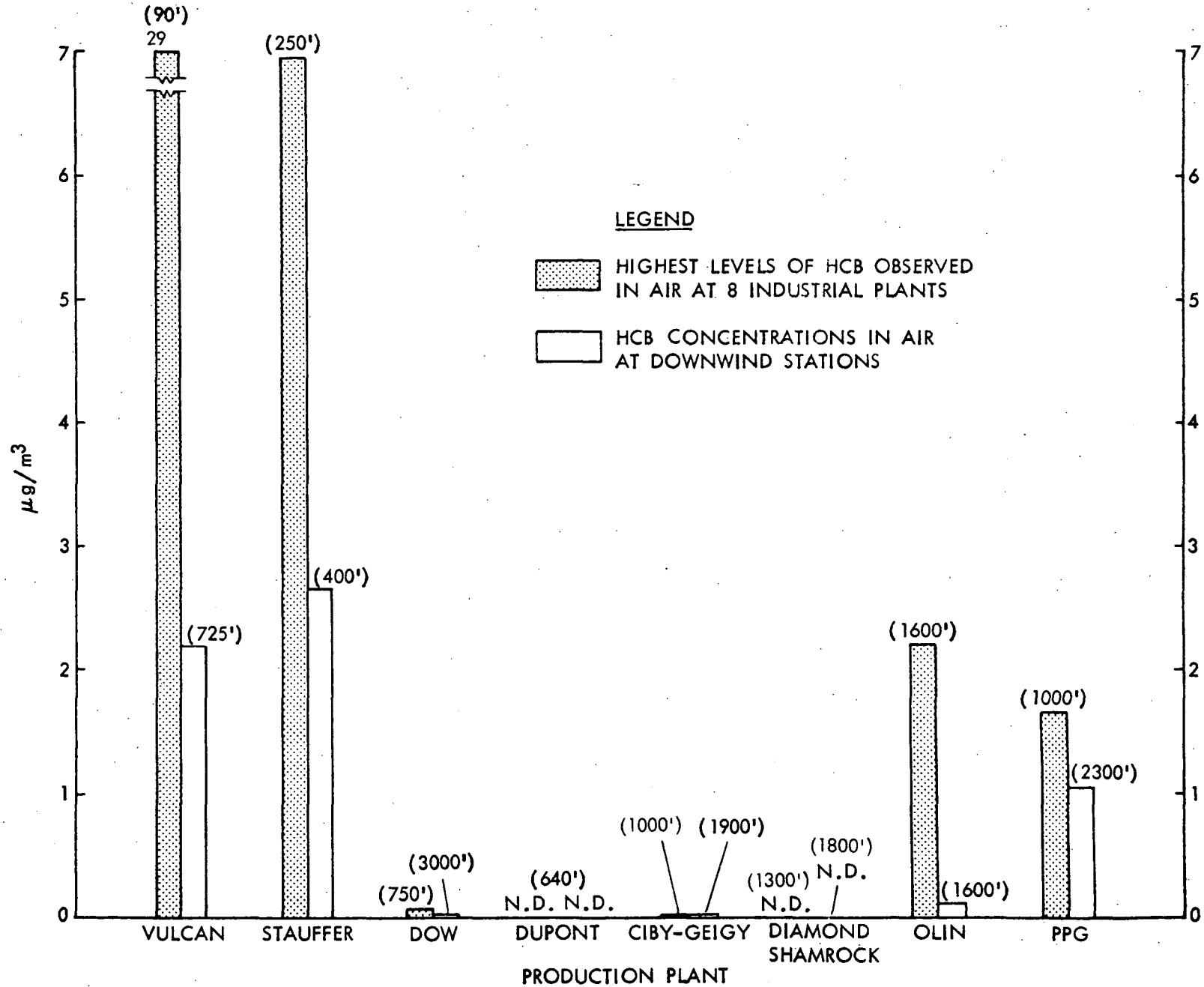

Figure 21. Summary of HCB concentrations in air

disposal methods. Lower HCB levels were detected at plants using on-site incineration, but downwind air concentrations were elevated above background at both plants; for example, an HCB concentration of 1 $\mu g/m^3$ was detected 2,300 ft downwind of the incinerator at one site. The lowest levels of HCB for perchloro- and trichloroethylene production plants were detected at the plant which used off-site incineration. Waste treatment at the PCNB production plant involved casting the solids into blocks which are stored under plastic. HCB vapor was detected in the low micrograms per cubic meter range near the storage area.

HCB was detected as vapor and particulate in three plants. In two of the three, the particulate HCB in air coincided with relatively high soil concentrations in the vicinity of the air sampling stations. At the third plant, which used a low temperature reaction process, analysis of the incinerator scrubber water showed 0.2 μg/liter HCB. Negligible levels of HCB were detected in samples from two sewage treatment facilities.

The overall relative standard deviation of the air sampling and analysis procedure, i.e., sample collection, storage, extraction, and analysis, was calculated to be less than 20%, based on determinations of HCB levels from sample pairs positioned at the same distances, but at different heights from the emission source. Good agreement was obtained from the analysis of water samples collected by "grab" sampling and by concentration of HCB on XAD-4 resin.

CONCLUSIONS

Industrial Sources of HCB

Considering the estimated production volumes of each of the six industries and the concentrations detected in this study, perchloroethylene and trichloroethylene production was easily the most significant source of HCB for the industries sampled. Although the total volume of chlorine produced was estimated to be 20 times that of perchloroethylene and trichloroethylene combined, the production of chlorine did not appear to be a significant source of HCB. Carbon tetrachloride production alone did not appear to be a significant source of HCB but this conclusion could be biased by the fact that the single-product carbon tetrachloride plant was the newest facility that was sampled (on-line in 1973). The production of PCNB resulted in the detection of moderately high levels of HCB but the total quantity of HCB released to the air was not significant because of the relatively low estimated production volume of PCNB. Estimated triazine herbicide production volumes and the associated HCB levels determined in this study were very low; therefore, the production of these compounds is not a significant source of HCB.

Effects of Waste Disposal Methods

In general, methods that involve open storage (pits, lagoons, etc.) resulted in elevated levels of HCB in air and surrounding soil. Waste holding areas were often the most significant emission source within the plant area. Contaminated soil appeared to be a secondary source of both particulate and vaporous HCB in air at two sites. Transportation of wastes resulted in at least part per million levels of HCB in roadside soil within plant areas. On-site incineration resulted in elevated air HCB levels for 750 ft and 2,300 ft, respectively, at two sites. Plastic sheets that were used to cover solids from PCNB production were effective in reducing particulate HCB but the storage area was the major source of HCB vapor at the plant. The HCB levels in water were reduced by approximately 50% at two plants that passed liquid wastes through holding ponds or treatment canals.

Physical Form of HCB

HCB was detected in particulate and vapor form. The detection of particulate HCB in air can be attributed to contaminated soil or blowing waste solids in the vicinity of air sampling stations. An exception was the incineration of wastes at the low temperature oxychlorination plant. The particulate HCB observed at this plant was due to either the production process or the incineration of liquid "bottoms." The latter conclusion is supported by the detection of a significant concentration of HCB in the incinerator scrubber water.

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6. Personal communication with Mr. C. A. Burns, Environmental Control Specialist, PPG Industries, Lake Charles, Louisiana, November 1975.

APPENDIX A

PRESAMPLING SURVEY AND FIELD SAMPLING

Presampling surveys and field sampling were conducted on the recommended industrial plants according to the schedule shown in Figure A-1. Essentially, during the presampling survey, information such as the surrounding terrain, meteorological conditions, production technology, and waste disposal technique was gathered. Following each site visit, a detailed field sampling strategy was devised and carried out approximately 2 to 4 weeks after the presampling survey date. Presented below are detailed descriptions of the presampling survey and field sampling conducted at each plant.

VULCAN MATERIAL COMPANY, WICHITA, KANSAS

PRESAMPLING SITE SURVEY

The presampling site survey at Vulcan Materials Company's Wichita, Kansas, plant was conducted on May 6, 1975. The following personnel were present:

Mr. J. I. Jordan, Jr.	Manager, Research and Development, Vulcan Materials Company
Mr. R. A. Bondurant, Jr.	Director, Environmental Control Safety, Vulcan Materials Company
Mr. Dave Harrison	Acting Technical Manager, Wichita Plant, Vulcan Materials Company
Mr. P. Constant	Midwest Research Institute
Mr. P. Kuykendall	Midwest Research Institute
Dr. J. Spigarelli	Midwest Research Institute

Vulcan Materials Company is located approximately 7 miles southwest of downtown Wichita and approximately 4 miles from any major residential area. The surrounding terrain is level with only one nearby water source, Cowskin Creek. The prevailing wind in May is generally from the south, southeast or southwest.

Perchloroethylene is produced by the reaction of hydrocarbons and chlorine. The hydrocarbons are generally of a widely variable composition and are obtained from many sources. The chlorine is produced by Vulcan and piped directly from their liquification station to the perchloroethylene reaction pot. Their chlorine production utilizes diaphragm cells and approximately 25% of their anodes are graphite, the remainder being dimensionally stabilized anodes.

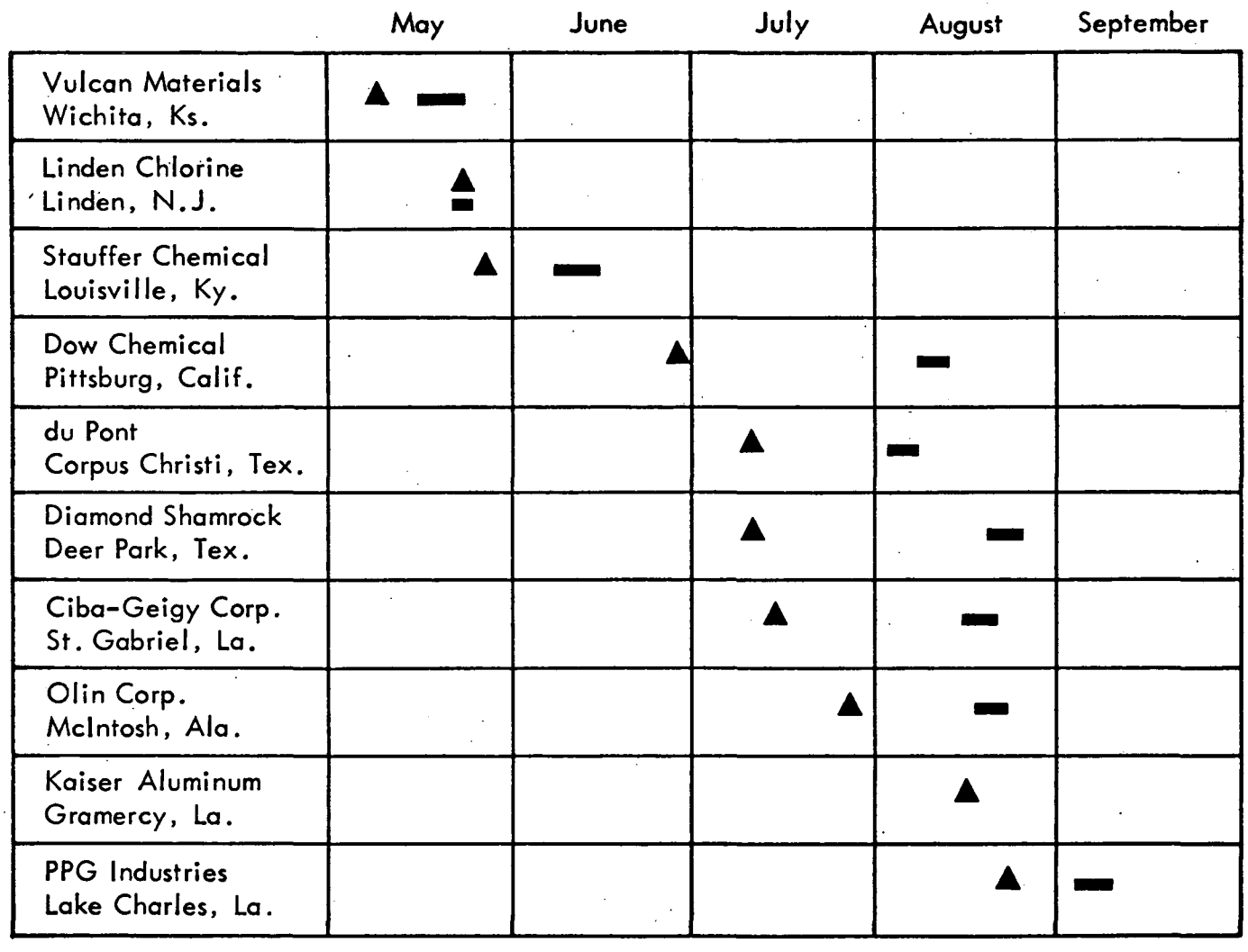


Figure A-1. Presampling survey and field sampling schedule

Vulcan uses two types of waste disposal, deep wells and landfills. The deep wells are used for the disposal of storm runoff, while the landfills are used for the disposal of "heavy ends" waste from perchloroethylene production. The residues are collected in a sealed vessel, transferred to another sealed vessel mounted on a trailer, and transported to an open pit where they are stored under water. When the residue level in the pit reaches a certain level, it is transferred by means of a backhoe to a dump truck and transported to the landfill, which is located southeast of the plant. There it is dumped, covered with polyethylene sheeting, then covered with dirt.

At the conclusion of the presampling survey, it was agreed upon that field sampling would be tentatively scheduled in the week of May 19, 1975.

FIELD SAMPLING

Field sampling at the Vulcan plant was conducted on May 20, 1975. Air, soil, and water samples were collected as planned. Detailed description of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Five general areas were chosen for air sampling: (a) perchloroethylene plant; (b) "Hex Pit;" (c) solar pond - landfill; (d) downwind of these locations; and (e) upwind of these locations. The total sampling time was divided into 4-hr periods. The upwind and downwind samplers were operated continuously during each 4-hr period, whereas all other samplers were operated only for the 1st hr of each 4-hr period. Each sampling location is shown in Figure A-2. Exact location was measured with respect to the suspected emission source and was reported along with other sampling data in Table A-1.

Soil Sampling

Soil sampling covered eight general areas:

S-1 Around the perchloroethylene plant

S-2 Route from perchloroethylene plant to "Hex Pit"

S-3 Around the "Hex Pit"

S-4 Route from "Hex Pit" to landfill

S-5 Between old and current landfill sites

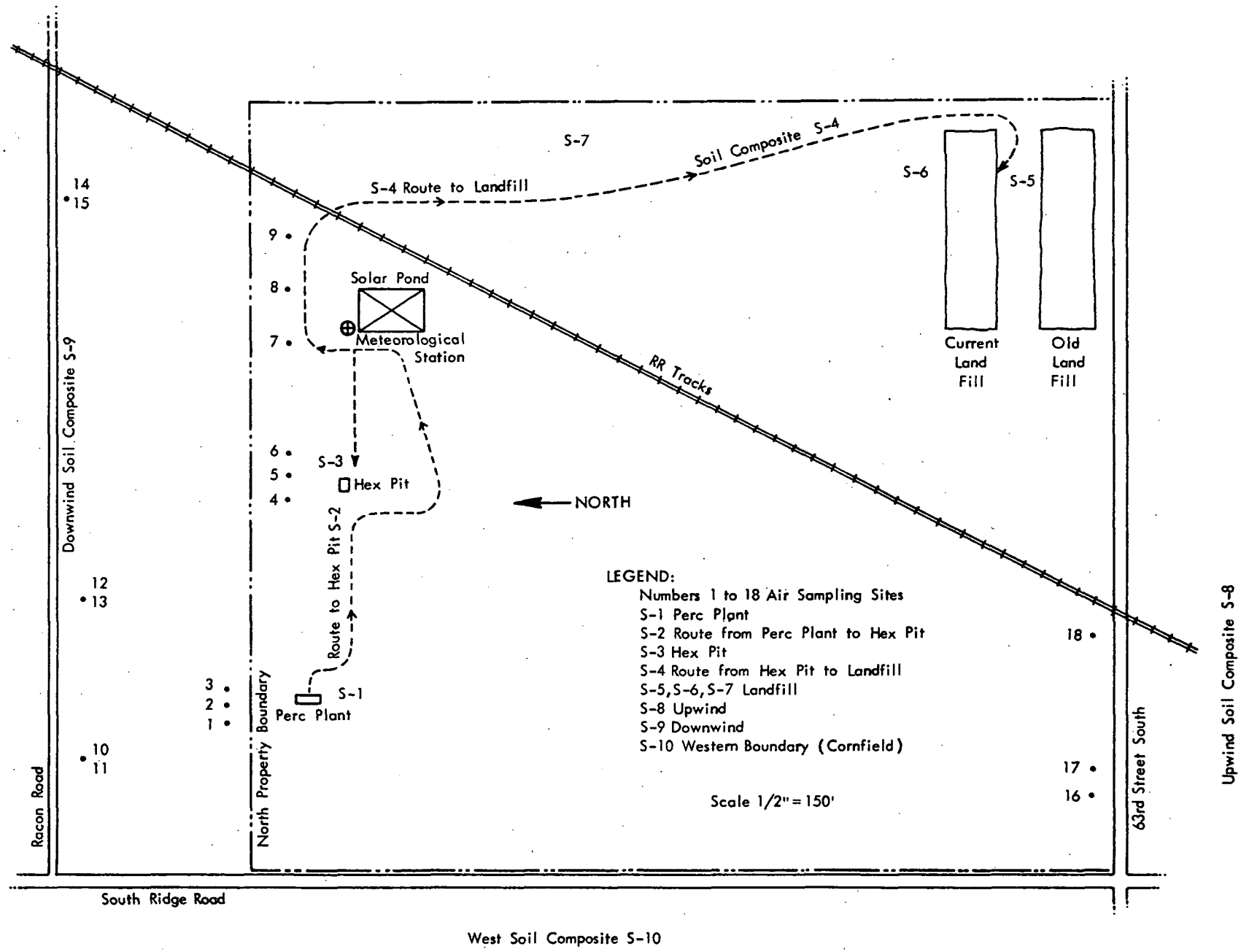


Figure A-2. Sampling locations at Vulcan Materials Company - Wichita plant

Table A-1. AIR SAMPLING DATA AT VULCAN MATERIALS COMPANY, WICHITA, KANSAS

General area	Sample no.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Total sample vol. (ℓ)	Sampler height (ft)
"Perc Plant"	1	250 ft north of "Perc Plant"- 250 ft west of Sample No. 2	1st hr of 4 hr	19.5	0.5	178	11
	2	250 ft north of "Perc Plant"	1st hr of 4 hr	19.5	0.5	149	11
	3	250 ft north of "Perc Plant"- 50 ft east of Sample No. 2	1st hr of 4 hr	19.5	0.5	207	11
"Hex Pit"	4	150 ft north of "Hex Pit" 75 ft west of Sample No. 2	1st hr of 4 hr	19.5	0.5	156	4
	5	150 ft north of "Hex Pit"	1st hr of 4 hr	19.5	0.5	232	4
	6	150 ft north of "Hex pit"- 75 ft east of Sample No. 2	1st hr of 4 hr	19.5	0.5	195	4
Solar pond-landfill	7	1,500 ft north of landfill- 225 ft northwest of solar pond	1st hr of 4 hr	19.5	3.5	813	4
	8	1,500 ft north of landfill- 225 ft north of solar pond	1st hr of 4 hr	19.5	3.5	1,123	4
	9	1,500 ft north of landfill- 440 ft northeast of solar pond	1st hr of 4 hr	19.5	3.5	1,198	4
Downwind	10 and 11	525 ft north of plant boundary-	4 hr	19.5	3.5	3,646	10-4
		340 ft east of Ridge Road	4 hr	19.5	3.5	3,862	11-11
	12 and 13	525 ft north of plant boundary-	4 hr	19.5	3.5	3,930	12-4
		850 ft east of Ridge Road	4 hr	19.5	3.5	4,172	13-11
	14 and 15	525 ft north of plant boundary-	4 hr	19.5	3.5	4,291	14-4
		2,100 ft north of Ridge Road	4 hr	19.5	3.5	4,272	15-11
	16	On southern plant boundary- 225 ft east of Ridge Road	4 hr	19.5	3.5	3,744	4
	17	On southern plant boundary- 300 ft east of Ridge Road	4 hr	19.5	3.5	3,176	4
	18	On southern plant boundary- 700 ft east of Ridge Road	4 hr	19.5	3.5	3,353	4

S-6 North of current landfill site

S-7 Along the eastern plant boundary

S-8 Along the southern plant boundary, passed 63rd Street

S-9 Beyond the northern plant boundary, along Racon Road

S-10 Along the western plant boundary, along the cornfield

All the samples were composites except the landfill samples. In addition, a sample of the "Hex Pit" solids was also collected.

Water Sampling

Water samples were taken from four general areas:

1. Upstream from waste inflow - Cowskin Creek
2. Downstream from waste inflow - Cowskin creek
3. Solar pond water
4. "Hex Pit" water

The location of the solar pond and "hex" pit is shown in Figure A-2; the sampling locations in Cowskin Creek are shown in Figure A-3.

Samples from Cowskin Creek were collected on Amberlite XAD-4 resin via a battery-operated pump. Grab samples were taken from the solar pond and "Hex Pit."

Plant Activities and Weather Conditions

Plant activities were observed during the sampling period and are tabulated in Table A-2.

The weather conditions during the sampling period are summarized in Table A-3. Fortunately, the wind was from the south or southeast during the entire sampling period except for the last 4-hr interval. The change in wind direction coincided with a thunderstorm which forced a termination of sampling during the final 4-hr period.

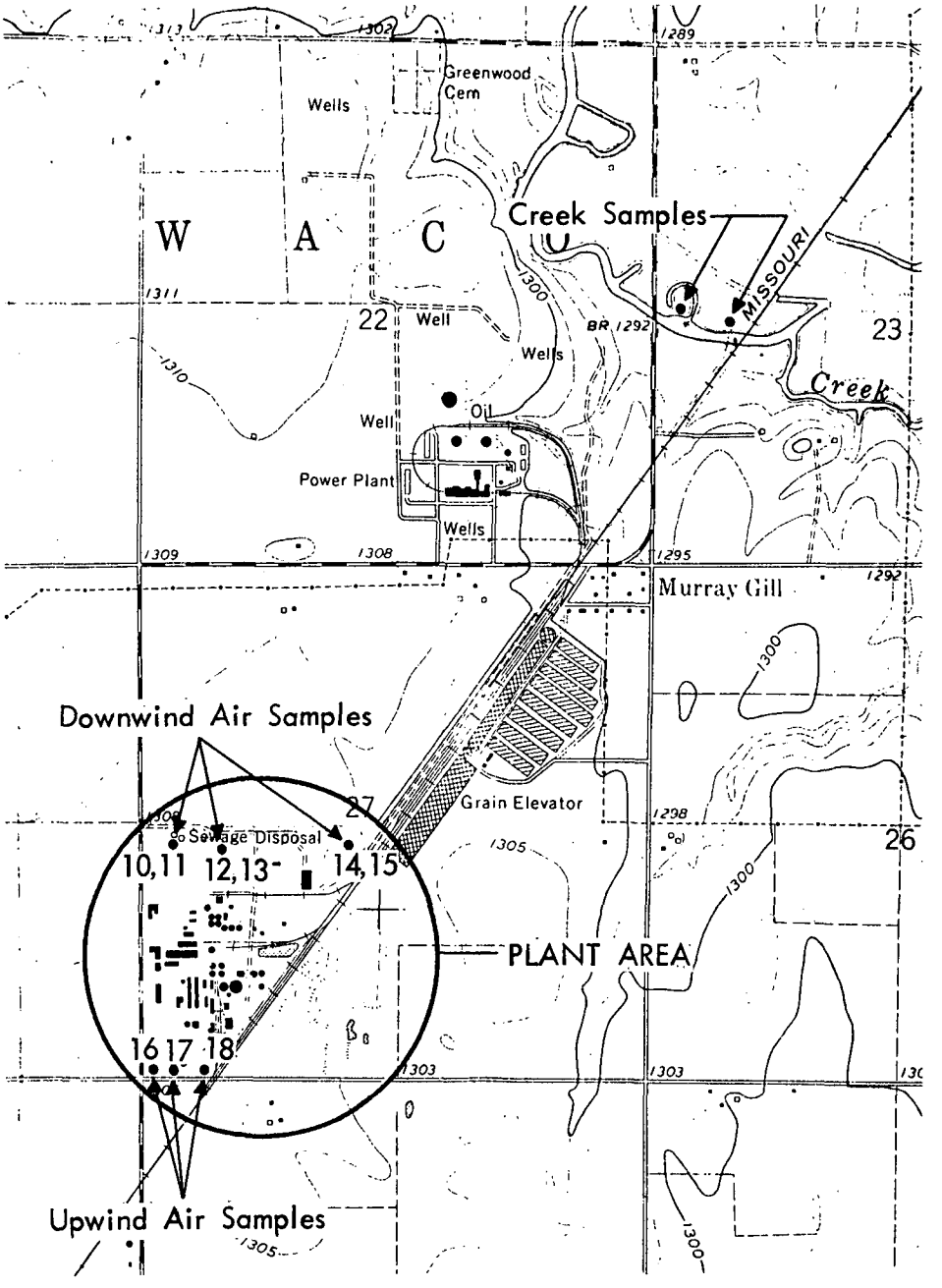


Figure A-3. Vulcan Materials Company - Wichita plant, and surrounding areas (2.6 in. = 1 mile)

Table A-2. PLANT ACTIVITIES DURING SAMPLING AT VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Time	"Perc plant"	"Hex pit"	Solar pond landfill	Downwind	Upwind
May 21					
1900	Normal	Fuller than usual	Normal	Normal	Normal
2000	Normal	Fuller than usual	Normal	Normal	Normal
2100	Normal	Fuller than usual	Normal	Normal	Normal
2200	Normal	Fuller than usual	Normal	Normal	Normal
2300	Normal	Fuller than usual	Normal	Normal	Normal
2400	Dumped "Hex"	Fuller than usual	Normal	Dumped "Hex"	Normal
May 22					
0100	Normal	Dumped "Hex"	Normal	Dumped "Hex"	Normal
0200	Normal	Normal	Normal	Normal	Normal
0300	Normal	Normal	Normal	Normal	Normal
0400	Normal	Normal	Normal	Normal	Normal
0500	Normal	Normal	Normal	Normal	Normal
0600	Normal	Normal	Normal	Normal	Normal
0700	Normal	Normal	Normal	Normal	Normal
0800	Normal	Normal	Normal	Normal	Normal
0900	Normal	Normal	Normal	Normal	Normal
1000	Normal	Normal	Normal	Normal	Normal
1100	Normal	Normal	Normal	Normal	Normal
1200	Normal	Normal	Normal	Normal	Normal
1300	Dumped "Hex"	Normal	Normal	Dumped "Hex"	Normal
1400	Normal	Dumped "Hex"	Normal	Dumped "Hex"	Normal
1500	Rain	Rain	Rain	Rain	Rain

Table A-3. WEATHER CONDITIONS DURING SAMPLING AT VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Time	Temperature (°C)	Barometric pressure (mm Hg)	Precipitation	Wind	
				Speed	Direction
May 21					
1900	27	722	None	15	South southeast
2000	27	722	None	12	South southeast
2100	24	723	None	11	Southeast
2200	23	724	None	12	Southeast
2300	23	724	None	13	South southeast
2400	23	724	None	12	South
May 22					
0100	23	724	None	11	South southeast
0200	23	724	None	12	Southeast
0300	23	724	None	13	South southeast
0400	23	724	None	10	South southeast
0500	21	723	None	9	South
0600	21	723	None	9	South southeast
0700	23	723	None	9	South southeast
0800	25	725	None	9	South southeast
0900	25	725	None	11	South southeast
1000	26	725	None	10	South
1100	26	725	None	12	South
1200	27	725	None	15	South
1300	29	725	None	15	South
1400	29	725	None	12	South
1500	29	725	Rain	10	Northwest

LINDEN CHLORINE COMPANY, LINDEN, NEW JERSEY

Presampling site survey at Linden Chlorine Company was conducted on May 29, 1975. The following personnel were present:

Mr. Ronald Burkett	Linden Chlorine Plant
Mr. Bill Heineman	Linden Chlorine Plant
Mr. Edward J. Finfer	Environmental Protection Agency, Region II
Mr. Martin L. Sanvito	DEP, BAPC
Mr. William J. O'Sullivan	DEP, Springfield office
Mr. William A. McGough	Central Jersey Regional Air Pollution Control Agency
Mr. Richard Hills	Central Jersey Regional Air Pollution Control Agency
Dr. George Scheil	Midwest Research Institute
Dr. J. Spigarelli	Midwest Research Institute

Linden Chlorine Company is located in the middle of a heavy industrial area. The closest residential areas are greater than 1 mile away, and nearby water source is the Arther Kill River.

During the visit, it was learned that graphite electrodes are no longer used in production of chlorine, instead platinum-coated titanium electrodes (dimensionally stabilized anodes) have been used since the end of March 1975. Because the potential for producing HCB and HCBD is much less for this type of electrode, it was decided that air sampling was not necessary. However, the holding pond that contains brine sludge and eroded graphite from previously used electrodes may contain significant quantities of chlorinated organics. In addition to carbon from eroded electrodes, the pond also contains spent carbon filters that were used to remove organics from process and surface water which flows through an open ditch to the Arthur Kill River. By far the largest portion of this water is made up of wastes from the nearby Gaf dye plant. The Gaf flume appeared to have a high organic content before the wastes from the chlorine plant enter the stream. Because of possible past HCB and HCBD contamination in the holding pond, samples were taken from the pond and wastewater stream. The samples are described below:

Sample type	Sample location
Water	Holding pond, inlet
Water	Holding pond, outlet
Water	Gaf weir, upstream of Cl_2 plant
Water	Waste stream, downstream of Cl_2 plant
Water	Tap water (control)
Solids	From holding pond, settled and suspended
Solids	Dredged solids adjacent to holding pond
Sludge	Waste stream, downstream of Cl_2 plant

The following samples were sent to Midwest Research Institute, on a later date by the Linden Chlorine Plant: process water, circulating brine, and uncontaminated soil.

STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

PRESAMPLING SITE SURVEY

The presampling site survey at Stauffer Chemical Company's Louisville, Kentucky, plant was conducted on May 30, 1975. The following personnel were present:

Mr. Arthur Wood	Manufacturing Manager, Stauffer Chemical Company
Mr. Harry Kutz	Plant Manager, Louisville plant
Mr. Kenneth G. Hebel	EPA/OSHA Testing Coordinator, Eastern Research Center, Dobbs Ferry, New York
Mr. Arthur E. Dungan	Assistant Plant Manager, Louisville plant
Mr. John R. Blunk	Process Superintendent, Louisville plant
Dr. George Scheil	Midwest Research Institute
Dr. J. Spigarelli	Midwest Research Institute

Stauffer Chemical Company is located on the east bank of the Ohio River approximately 6 miles southwest of downtown Louisville, Residential areas surround the plant, the closest being about 1 mile northeast of the plant. The wind direction, according to the weather bureau records (10-year average) at the Louisville airport indicated that during the month of June there is a 50% probability of a south wind (from one of the four southern quadrants) and a much lower probability from any other direction.

The "hex" solids from the perchloroethylene production plant are gravity fed into drums (batch-wise). After several drums (unsealed, possibly covered with a pleastic sheet) accumulate, they are removed from the plant area. Company officials said that the drums are usually removed once a day at 8:00 a.m. The plant operated 24 hr a day, 7 days a week.

The drums are transported to an area just west of the surplus storage building where they are loaded onto trucks and taken to an approved landfill site approximately 15 miles from the plant. Cooling water and surface runoff from the plant area are fed to a sump where the pH is adjusted to 6 to 9, the liquid is pumped to a concrete settling pond, and gravity fed through a pipe into the Ohio River.

Based on the plant operation described above, three possible sources of HCB and HCBD contamination were considered, namely: (a) the production area, especially at the location of the open barrels, (b) the settling pond and (c) the "hex" loading area and the transportation route to the landfill.

FIELD SAMPLING

Field sampling at the Stauffer plant was conducted on June 12, 1975. Air, water, soil, and sediment samples in and around the plant were collected. Detailed descriptions of the sampling and plant activities during sampling are discussed below.

Air Sampling

Eight sampling stations encircling the immediate plant area, and one downwind station were set up. The total sampling period was divided into six 4-hr periods and samplers were operated 2 hr of each 4-hr period. Each sampling location is shown in Figure A-4. Exact locations with respect to the perchloroethylene plant area, are listed along with other sampling data in Table A-4.

Soil Sampling

Soil sampling was conducted in five general areas:

S-1 Upwind (along the southern plant boundary)

S-2 Plant road (along the main road)

S-3 Drum loading area

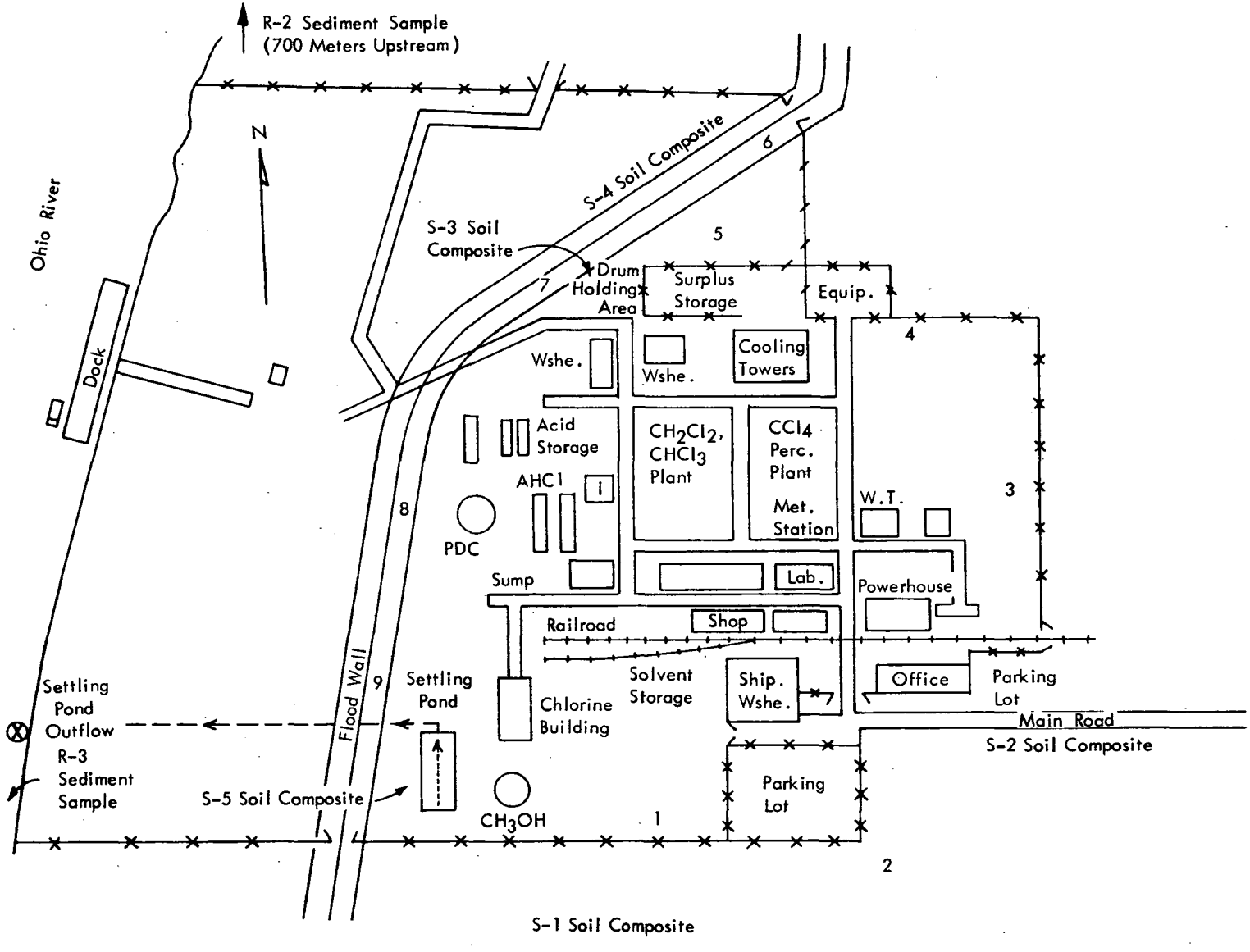


Figure A-4. Sampling locations at Stauffer Chemical Company - Louisville, Kentucky

Table A-4. AIR SAMPLING DATA AT STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

General area	Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Total Sample vol. (ℓ)	Sampler height (ft)
Upwind	1	450 ft south southwest of "Perc Plant" area	1st 2 hr of 4 hr	12.3	3.5	2,588	4
Upwind	2	500 ft south southeast of "Perc Plant" area	1st 2 hr of 4 hr	11.8	3.5	2,626	4
East of "Perc Plant"	3	340 ft east of "Perc Plant" area	1st 2 hr of 4 hr	10.6	3.5	2,221	4
Northeast of "Perc Plant"	4	250 ft northeast of "Perc Plant" area	1st 2 hr of 4 hr	12.5	3.5	2,768	4
Downwind	5	300 ft north of "Perc Plant" area	1st 2 hr of 4 hr	12.9	3.5	2,787	4
Downwind	6	400 ft north of "Perc Plant" area	1st 2 hr of 4 hr	13.1	3.5	2,662	4
Northwest of "Perc Plant"	7	330 ft northwest of "Perc Plant" area	1st 2 hr of 4 hr	13.2	3.5	2,850	4
West of "Perc Plant" north of settling pond	8	420 ft west of "Perc Plant" area	1st 2 hr of 4 hr	13.3	3.5	2,872	4
Southwest of "Perc Plant," northwest of settling pond	9	540 ft southwest of "Perc Plant" area	1st 2 hr of 4 hr	13.5	3.5	2.926	4

S-4 Downwind (along the northern plant boundary)

S-5 Settling pond area

All samples were composites.

Water Sampling

Water sampling was limited to the plant well water and the settling pond.

W-1 Plant well water

W-2 Settling pond inlet (24 hr composite)

W-3 Settling pond inlet (grab sample)

W-4 Settling pond outlet (24 hr composite)

W-5 Settling pond outlet (grab sample)

W-6 Settling pond outlet (through Amberlite XAD-4)

Sediment Sampling

Sediment sampling was conducted at three locations:

R-1 Settling pond sediment

R-2 Ohio River, 700 m upstream of Stauffer outflow

R-3 Ohio River, 250 m downstream of Stauffer outflow

Plant Activities and Weather Conditions

Plant activities were observed during the sampling period and are shown in Table A-5.

The weather conditions during the sampling period are summarized in Table A-6.

Table A-5. PLANT ACTIVITIES DURING SAMPLING AT STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Time	"Perc plant"[a]	Drum loading area	Settling pond
12 June			
1000	Normal	Normal	Normal flow
1100	Normal	Normal	Normal flow
1200	Normal	Normal	Normal flow
1300	Normal	Normal	Normal flow
1400	Normal	"Hex" drums removed	Normal flow
1500	Normal	"Hex" drums removed	Normal flow
1600	Normal	Normal	Normal flow
1700	Normal	Normal	Normal flow
1800	Normal	Normal	Normal flow
1900	Normal	Normal	Normal flow
2000	Normal	Normal	Normal flow
2100	Normal	Normal	Normal flow
2200	Normal	Normal	Normal flow
2300	Normal	Normal	Normal flow
2400	Normal	Normal	Normal flow
13 June			
0100	Normal	Normal	Normal flow
0200	Normal	Normal	Normal flow
0300	Normal	Normal	Normal flow
0400	Normal	Normal	Normal flow
0500	Normal	Normal	Normal flow
0600	Normal	Normal	Normal flow
0700	Normal	Normal	Normal flow
0800	Normal	Normal	Normal flow
0900	Normal	Normal	Normal flow
1000	Normal	Normal	Normal flow

a/ Normal operation utilizing HCBD recovery.

Table A-6. WEATHER CONDITIONS DURING SAMPLING AT STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Time	Temperature (°C)	Barometric pressure (mm Hg)	Precipitation	Wind	
				Speed	Direction
12 June					
1000	26	757	None	4	S
1100	28	757	None	4	S
1200	29	757	None	4	S
1300	29	757	None	6	S
1400	29	757	None	8	S
1500	29	757	None	7	S
1600	29	758	None	6	S
1700	27	758	None	7	S
1800	25	758	None	6	S
1900	24	758	None	6	S
2000	25	759	None	5	S
2100	23	759	None	2	S
2200	21	759	None	5	S
2300	20	759	10 min rain	5	E
2400	20	759	None	4	S
13 June					
0100	19	760	None	4	S
0200	18	760	None	2	S
0300	17	760	None	2	S
0400	16	760	None	0	S
0500	16	760	None	0	S
0600	17	760	None	0	S
0700	17	760	None	0	S
0800	19	760	None	0	S
0900	22	760	None	0	S
1000	25	760	None	0	S

DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA

PRESAMPLING SITE SURVEY

Presampling site survey at Dow Chemical Company's Pittsubrg, California, plant was conducted on June 30, 1975. The following personnel were present:

Mr. Ed Elkins	Manager, Environmental and operational Services, Dow Chemical Company
Mr. David Baur	Dow Chemical Company
Mr. Mike Thomas	Dow Chemical Company
Mr. Paul Constant	Midwest Resesrch Institute
Mr. Jim Spigarelli	Midwest Research Institute

Dow Chemical Company is located approximately 2 miles northeast of Pittsburg, California, and 4 miles west of Antioch, California, and is situated on the southern bank of the New York slough of the San Joaquin River. The immediate vicinity is flat, but mountains lie approximately 5 miles south of the plant. Approximately 70% of the time during July the wind is from west or west-northwest. Residential areas are in Pittsburg and Antioch.

Chemicals produced at this Dow facility include chlorine, carbon tetrachloride, and perchloroethylene. According to Mr. Elkins, all wastes from the chlorinated hydrocarbon production flow to a thermal oxidizer, are converted to hydrochloric acid and are recycled. In most instances, surface runoff from the plant areas flow into a solar pond. However, inspection on the plant and the solar pond area were not allowed. Only the plant perimeter and beyond were surveyed.

Air sampling locations were planned based on the thermal oxidizer parameters such as stack gas temperature, gas flow rate, and stack height and diameter.

FIELD SAMPLING

Field sampling at the Dow plant was conducted on August 7, 1975, after several postponements. Air, soil, and water samples were collected. Detailed descriptions of the sampling, plant activities, and weather conditions are discussed below.

Air Sampling

Eight sampling stations were located so that two upwind, three near downwind, and three far downwind samples were obtained. Air was sampled over a 24-hr period with the exception of Stations 2 (upwind) and 7 (far downwind). Two air sampling tubes were operated in series at each station. Each sampling location is shown in Figure A-5. The exact location was measured with respect to the production plant area and is listed along with other sampling data in Table A-7.

Soil Sampling

Soil sampling was conducted in three general areas:

S-1 Upwind (western plant boundary)

S-2 Southern property boundary

S-3 Downwind (eastern plant boundary)

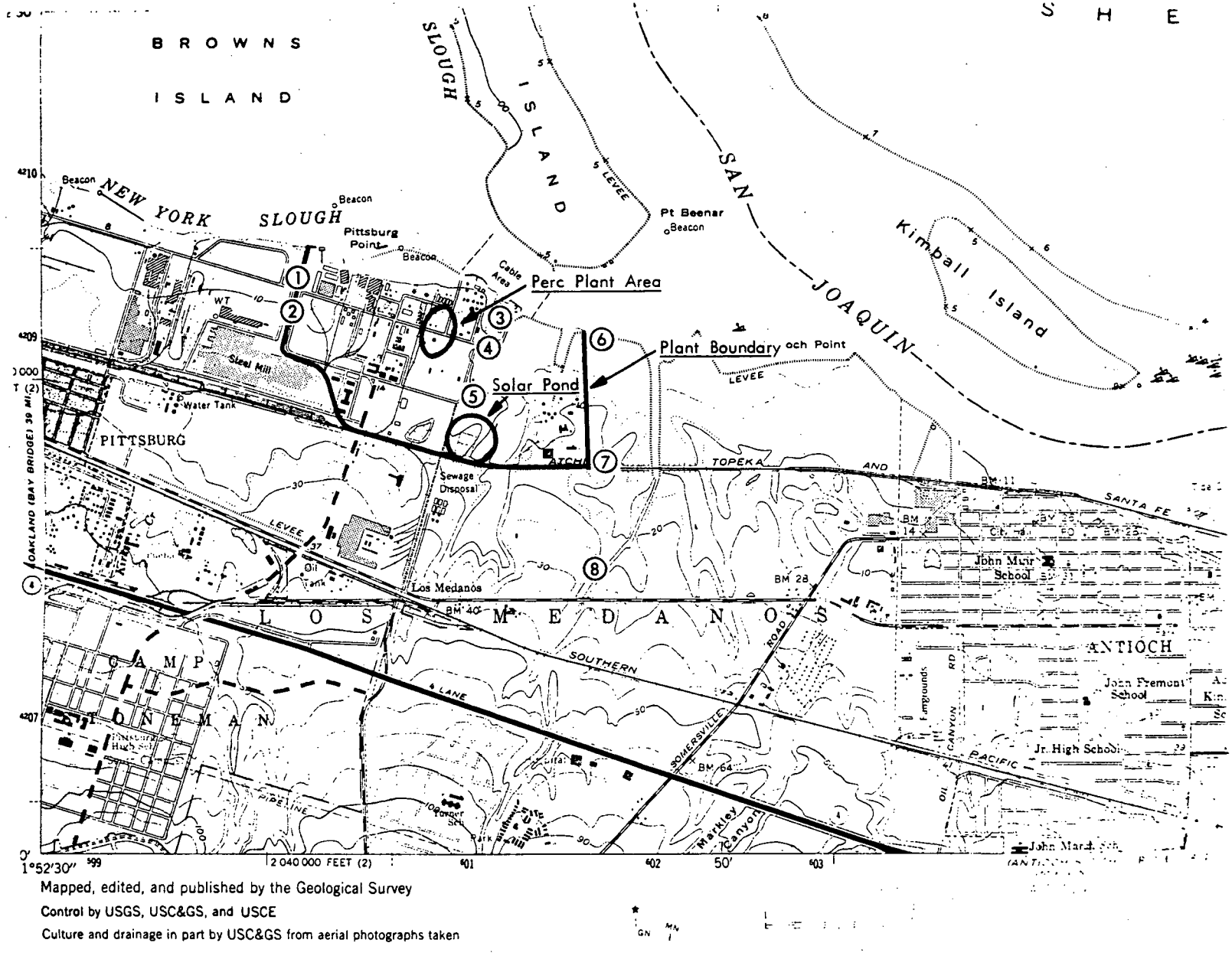
All samples were composites.

Water Sampling

One grab water sample of the New York Slough was obtained at the northeast corner of Dow's property. Water sampling at the solar pond was not permitted by the Dow officials.

Plant Activities and Weather Conditions

Plant activities and weather conditions during sampling are shown in Table A-8.



Mapped, edited, and published by the Geological Survey

Control by USGS, USC&GS, and USCE

Culture and drainage in part by USC&GS from aerial photographs taken

Figure A-5. Sample locations at Dow Chemical Company, Pittsburg, California

Table A-7. AIR SAMPLING DATA AT DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA

General area	Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
Upwind	1	2,630 ft west-northwest of "Perc Plant"	24 hr continuous	20.7	3.5	4,336	4
Upwind	2	2,780 ft west of "Perc Plant"	24 hr continuous	2.0[a/]	3.5	427	4
Near downwind	3	900 ft east-northeast of "Perc Plant"	24 hr continuous	19.8	3.5	4,166	4
Near downwind	4	830 ft southeast of "Perc Plant"	24 hr continuous	18.4	3.5	3,870	4
Near downwind	5	1,280 ft south-southeast of "Perc Plant"	24 hr continuous	17.7	3.5	3,713	4
Far downwind	6	2,550 ft east of "Perc Plant"	24 hr continuous	20.5	3.5	4,314	4
Far downwind	7	3,600 ft southeast of "Perc Plant"	24 hr continuous	4.6[a/]	3.5	962	4
Far downwind	8	5,100 ft south-southeast of "Perc Plant"	24 hr continuous	18.9	3.5	3.963	4

a/ Generator failure.

Table A-8. WEATHER CONDITIONS AND PLANT ACTIVITIES DURING SAMPLING AT DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA[a/]

Time	Temperature (°F)	Barometric pressure (mm Hg)	Wind		Plant activities
			Speed	Direction	
August 7					
1700	97	760	8	West	Normal
1800	96		12	West	Normal
1900	92		10	West	Normal
2000	87		10	West	Normal
2100	84	760	8	West	Normal
2200	82		8	West	Normal
2300	79		6	West	Normal
2400	80		2	West	Normal
August 8					
0100	74	760	0	-	Normal
0200	74		6	West	Normal
0300	74		8	West	Normal
0400	73		6	West	Normal
0500	71	760	4	West	Normal
0600	69		0	-	Normal
0700	67		0	-	Normal
0800	73		0	-	Normal
0900	83	760	0	-	Normal
1000	87		2	West	Incinerator feed rate reduced
1100	91		6	West	Normal
1200	96		2	Northwest	Normal
1300	98	760	4	Northwest	Normal
1400	101		4	West	Normal
1500	101		10	West	Possible event
1600	103		12	West	Normal
1700	102	759	12	West	Normal
1800	99		13	West	Normal

a/ No precipitation during sampling period.

E. I. du PONT de NEMOURS AND COMPANY, INC., CORPUS CHRISTI, TEXAS

PRESAMPLING SITE SURVEY

The presampling site survey at du Pont's Corpus Christi, Texas, plant was conducted on July 11, 1975. The following personnel were present:

Mr. Charles Evans	Plant Manager, du Pont
Mr. Dave Brooks	Assistant Plant Manager, du Pont
Mr. Phil Kuykendall	Midwest Research Institute

This du Pont plant is located on Corpus Christi Bay approximately 3 miles northeast of downtown Corpus Christi. The immediate area surrounding the plant contains several industries, but the small town of Ingleside is about 1 mile east of the plant. Wind direction in the summer is generally from the south, south southeast, and south southwest.

Carbon tetrachloride is produced by chlorination of methane or ethylene at elevated temperatures. All by-products are continuously recycled to insure total chlorination. Chlorine is obtained from an outside source. Solid wastes from the process are minimal and are not frequently removed from the reaction vessel.

Solid wastes are generally drummed and shipped to an outside firm for disposal although some wastes are dumped into du Pont's two landfills. One landfill is dedicated to the disposal of a mixture of cement, lime, and catalyst from Freon production. The other landfill normally receives such wastes as contaminated containers, spills, or "heavy ends" waste from the carbon tetrachloride production.

Wastewater is handled separately as process waste and storm runoff. Process wastes are channeled via open concrete ditches, to an equalization pond where the residence time is 3 to 6 days. Underground pipes discharge the wastewater into Lacita Channel at a flow rate of approximately 2,000 gal/min.

Storm runoff is discharged, via open dirt ditches, directly into Lacita Channel

FIELD SAMPLING

Field sampling at the du Pont plant was conducted on August 3, 1975. Air, soil, water, and sediment samples were collected. Detailed descriptions of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Due to equipment failure,only five air sampling stations were operated. Two were upwind and three were downwind. All samples were operated for 24 hr utilizing one filter and two Tenax tubes in tandem. Sampling locations are shown in Figure A-6. Exact distance of each station to the production area was determined and reported along with other sampling data in Table A-9.

Soil Sampling

Soil sampling was conducted in three general areas:

S-1 Upwind (at Stations 1 and 2 on southern boundary)

S-2 Downwind (at Stations 3, 4, and 5 on northern boundary)

S-3 Landfill area (20 ft from edge of miscellaneous landfill)

All soil samples were composites.

Water Sampling

Water sampling was conducted at seven locations:

W-1 Raw plant water before use

W-2 Settling pond inlet (amberlite)

W-3 Settling pond inlet (grab)

W-4 Settling pond outlet (amberlite)

W-5 Settling pond outlet (grab)

W-6 Storm runoff outfall (grab)

W-7 Water standing in landfill

Sediment Sampling

Sediment sampling was conducted at four locations:

R-1 Settling pond inlet

R-2 Settling pond outlet

R-3 Storm runoff outfall

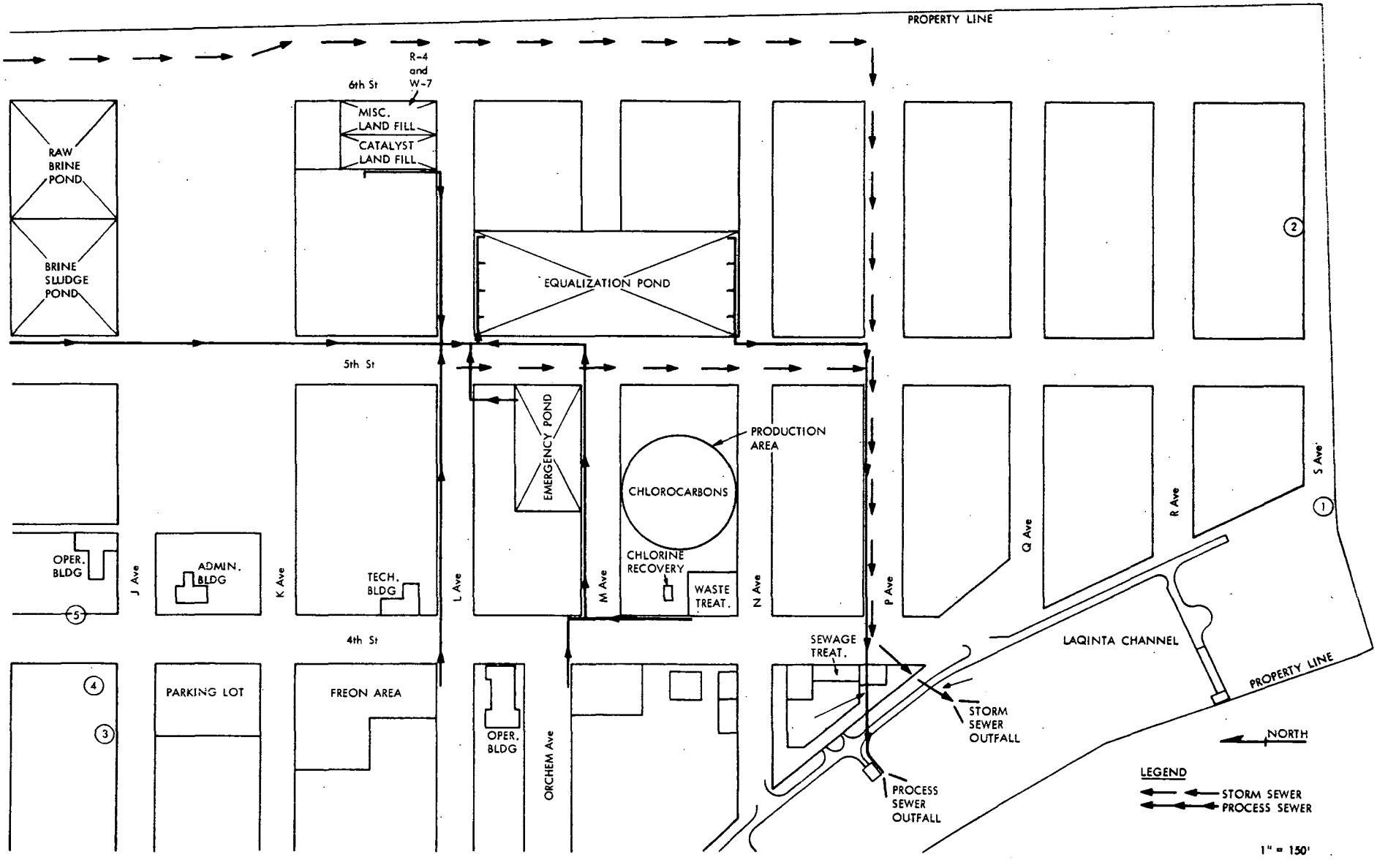


Figure A-6. Sample locations at E. I. du Pont de Nemours and Company, Inc., Corpus Christi, Texas

Table A-9. AIR SAMPLING DATA AT E. I. DU PONT DE NEMOURS AND COMPANY, INC., CORPUS CHRISTI, TEXAS

General area	Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
Upwind	1	675 ft south of production area	24 hr continuous	22.8	3.5	4,371	4
Upwind	2	700 ft south southeast of production area	24 hr continuous	19.5	3.5	3,621	4
Downwind	3	640 ft north northwest of production area	24 hr continuous	21.2	3.5	4,070	4
Downwind	4	64- ft north northwest of production area	24 hr continuous	21.2	3.5	4,007	4
Downwind	5	640 ft north northwest of production area	24 hr continuous	21.3	3.5	3,965	4

Plant Activities

Plant activities during sampling are shown in Tables A-10 and A-11, respectively.

Table A-10. PLANT ACTIVITIES DURING SAMPLING AT E. I. DU PONT DE NEMOURS AND COMPANY, INC., CORPUS CHRISTI, TEXAS

Time	Chlorocarbon unit	Landfill	Settling pond flow (gal/min)
August 3			
0700	Normal production	No activity	2,300
0800	Normal production	No activity	2,300
0900	Normal production	No activity	2,300
1000	Down	No activity	2,300
1100	Down	No activity	2,300
1200	Normal production	No activity	2,300
1300	Normal production	No activity	2,300
1400	Normal production	No activity	2,300
1500	Normal production	No activity	2,300
1600	Normal production	No activity	2,300
1700	Normal production	No activity	2,300
1800	Normal production	No activity	2,300
1900	Normal production	No activity	2,300
2000	Normal production	No activity	2,300
2100	Normal production	No activity	2,300
2200	Normal production	No activity	2,300
2300	Normal production	No activity	2,300
2400	Normal production	No activity	2,300
0100	Normal production	No activity	2,300
0200	Normal production	No activity	2,300
0300	Normal production	No activity	2,300
0400	Normal production	No activity	2,300
0500	Normal production	No activity	2,300
0600	Normal production	No activity	2,300
0700	Normal production	No activity	2,300
0800	Normal production	No activity	2,300
0900	Normal production	No activity	2,300
1000	Normal production	No activity	2,300

Table A-11. WEATHER CONDITIONS DURING SAMPLING AT E. I. DU PONT DE NEMOURS AND COMPANY, INC., CORPUS CHRISTI, TEXAS

Time	Temperature (°C)	Barometric pressure (mm Hg)	Precipitation	Wind	
				Speed	Direction
August 3					
0700	28	762	none	19	South
0800	28	762	none	17	South
0900	28	762	none	15	South
1000	29	762	none	11	South
1100	29	761	none	11	South southeast
1200	29	761	none	11	South southeast
1300	29	761	none	10	South southeast
1400	30	761	none	10	South southeast
1500	29	761	none	12	South southeast
1600	29	760	rain	22	North northwest
1700	27	760	rain	9	East
1800	28	760	none	10	East southeast
1900	28	760	none	10	Southeast
2000	28	760	none	9	Southeast
2100	28	760	none	9	Southeast
2200	27	762	none	7	Southeast
2300	27	762	none	10	Southeast
2400	27	762	none	10	South southeast
August 4					
0100	27	762	none	10	South southwest
0200	26	762	none	18	South
0300	28	761	none	16	South
0400	27	761	none	17	South
0500	28	761	none	17	South
0600	29	761	none	15	South
0700	29	761	none	15	South southwest
0800	29	760	none	18	South southwest
0900	30	760	none	15	South southwest
1000	30	760	none	13	South southwest
1100	30	760	rain	12	Southwest

DIAMOND SHAMROCK CORPORATION, DEERK PARK, TEXAS

PRESAMPLING SITE SURVEY

The presampling site survey at Diamond Shamrock's, Deer Park, Texas, plant was conducted on July 10, 1975. The following personnel were present:

Mr. Lavern R. Heble	Environment Control Manager, Gulf Coast Area, Diamond Shamrock Corporation
Mr. William C. Hutton	Senior Environmental Control Engineer, Diamond Shamrock Corporation
Mr. Bob Baxter	Perchloroethylene Unit Manager, Diamond Shamrock Corporation
Ms. Sandra Quinlivan	TRW, Rodondo Beach, California
Mr. Phil Kuykendall	Midwest Research Institute

This Diamond Shamrock plant is located in the heart of a huge industrial area along the Honston ship channel. The nearest residential area is Deer Park, located approximately 5 miles south of the plant. Wind direction in the summer months is mostly from the south.

Perchloroethylene and trichloroethylene are produced in this plant by the reaction of chlorine and hydrocarbons. The chlorine used is produced at a nearby Diamond Shamrock plant and is piped to the production area. "Hex" solids resulting from the process are stored in large tanks awaiting disposal.

The major possible sources of HCB and HCBD are (a) the production area and (b) the "Hex" solids storage area.

Diamond Shamrock uses two types of waste disposal; contracted solid waste disposal and channeling of wastewater into Patrick Bayou. The solid wastes are transferred from the holding tank to a tank truck which transports the waste to an outside firm for incineration or landfill disposal. Solid waste removal does not occur daily. A solvent flush of the lines into the tank truck follows each dumping of "Hex" solids. Wastewater is steam-stripped in the production area then piped to a waste treatment plant prior to discharge into Patrick Bayou.

FIELD SAMPLING

Field sampling at the Diamond Shamrock plant was conducted on August 20, 1975 after several postponements due to plant down time. Air, soil, and water samples were collected. Detailed description of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Eight air sampling stations were used--three upwind and five downwind. To avoid possible breakthrough of the Tenax®-GC, smaller critical orifices were used. All samples were operated for three 8-hr periods, resulting in a 24-hr sampling time. Sampling locations are shown in Figure A-7. The exact distance of each station to the production area was determined and reported along with other sampling data in Table A-12.

Soil Sampling

Soil sampling was conducted at three areas:

S-1 Upwind (along southern boundary)

S-2 Downwind (along northern boundary)

S-3 Production area

All soil samples were composites.

Water Sampling

Water sampling was conducted at two points:

W-1 Incoming channel water

W-2 Process water outfall

All samples were grab samples.

No sediment sampling was conducted because of no existing sampling site.

Plant Activities and Weather Conditions

The weather conditions during sampling are shown in Table A-13. Plant activities were normal during the entire sampling period.

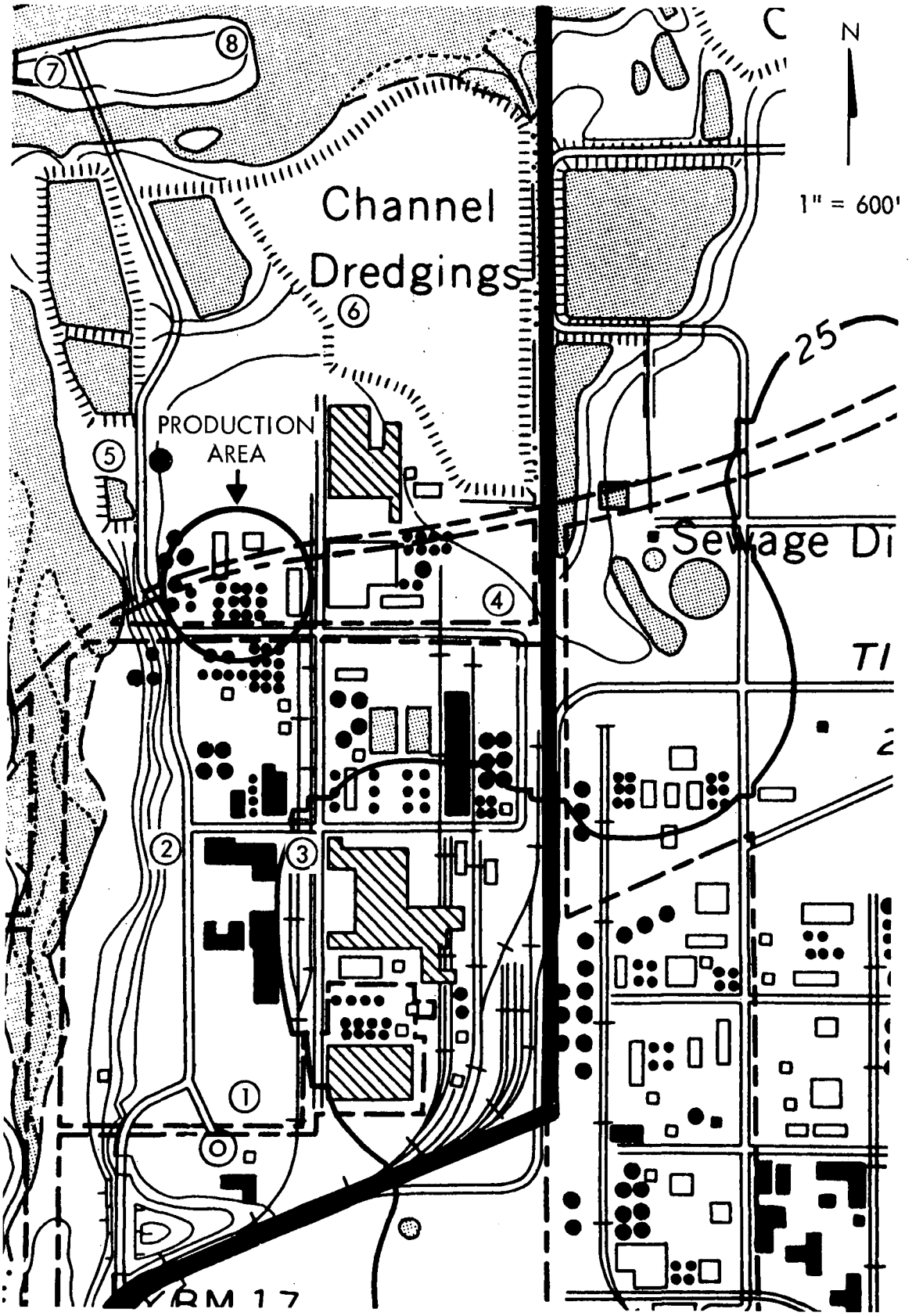


Figure A-7. Sampling locations at Diamond Shamrock Corporation, Deer Park, Texas

Table A-12. AIR SAMPLING DATA AT DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

General area	Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
Far upwind	1	1,300 ft south of "Perc Plant" area	24 hr continuous	24.2	0.4	580	4
Near upwind	2	510 ft southwest of "Perc Plant" area	24 hr continuous	24.9	0.4	598	4
Near upwind	3	420 ft southeast of "Perc Plant" area	24 hr continuous	20.2	0.4	485	4
Near downwind	4	1,200 ft northeast of "Perc Plant" area	24 hr continuous	22.5	0.4	540	4
Near downwind	5	1,300 ft northwest of "Perc Plant" area	24 hr continuous	22.6	0.4	542	4
Near downwind	6	1,860 ft north northeast of "Perc Plant" area	24 hr continuous	25.3	0.4	608	4
Far downwind	7	2,850 ft north northwest of "Perc Plant" area	24 hr continuous	23.3	0.4	559	4
Far downwind	8	2,900 ft north of "Perc Plant" area	24 hr continuous	23.1	0.4	555	4

Table A-13. WEATHER CONDITIONS DURING SAMPLING AT DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

Time	Temperature (°C)	Barometric pressure	Precipitation	Wind Speed	Wind Direction
August 20					
1600	34	766	None	4	Southeast
1700	34	766		5	South southeast
1800	33	766		4	South
1900	33	766		3	South southeast
2000	31	767		2	South southeast
2100	31	767		1	South
2200	29	767		1	South
2300	29	767		1	South
2400	27	767		1	South
August 21					
0100	26	766		1	South
0200	26	766		1	South
0300	28	766		1	North northwest
0400	28	766		1	North
0500	30	765		2	North northwest
0600	30	765		6	Southeast
0700	30	765		5	South
0800	30	765	Rain	3	North northwest
2100	28	765	Rain	1	East northeast
2200	28	765	None	1	East
2300	27	765		1	North northeast
2400	25	765		1	North
August 22					
0100	25	765		1	North northeast
0200	28	765		1	North
0300	29	766		1	West northwest
0400	29	766		1	East
0500	31	766		1	East
0600	31	766		1	East
0700	31	766		1	North northeast
0800	30	766		1	East northeast
0900	31	766		2	North northeast

CIBA-GEIGY CORPORATION, ST. GABRIEL, LOUISIANA

PRESAMPLING SITE SURVEY

The presampling site survey at Ciba-Geigy Corporation, St. Gabriel, Louisiana, plant was conducted on July 15, 1975. The following personnel were present:

Mr. William F. Snyder	Staff Engineer, Environmental Engineering, Ciba-Geigy
Mr. Phil Kuykendall	Midwest Research Institute

Ciba-Geigy Corporation is located in a large industrial area which is approximately 20 miles south of Baton Rouge. The plant is on the east bank of the Mississippi River in a predominantly swampy area with minimal residences. Wind direction during the summer months are generally from the south and west.

Triazine herbicides are produced by the amination of cyanuric chloride. The production processes result in the accumulation of "still bottoms" with an approximate concentration of 2,000 µg/g.

Solid wastes are drummed and shipped to an outside firm for incineration. A vent scrubber is used for vapor emissions. Wastewater is discharged via open ditches to a holding pond, then discharged into the Mississippi River at an average flow rate of 2,000 gal/min. An emergency wastewater outfall into Bayou Braud is occasionally used.

FIELD SAMPLING

Field sampling at the Ciba-Geigy plant was conducted on August 13, 1975. Air, soil, and water samples were collected. Detailed description of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Eight air sampling stations were positioned around the plant because of the erratic wind direction. Only one Tenax®-GC tube was used. To avoid possible breakthrough of the Tenax®-GC, sampling was operated 2 hr out of every 8-hr period. The sampling locations are shown in Figure A-8. Exact distance of each station to the production areas was determined and reported along with other sampling data in Table A-14.

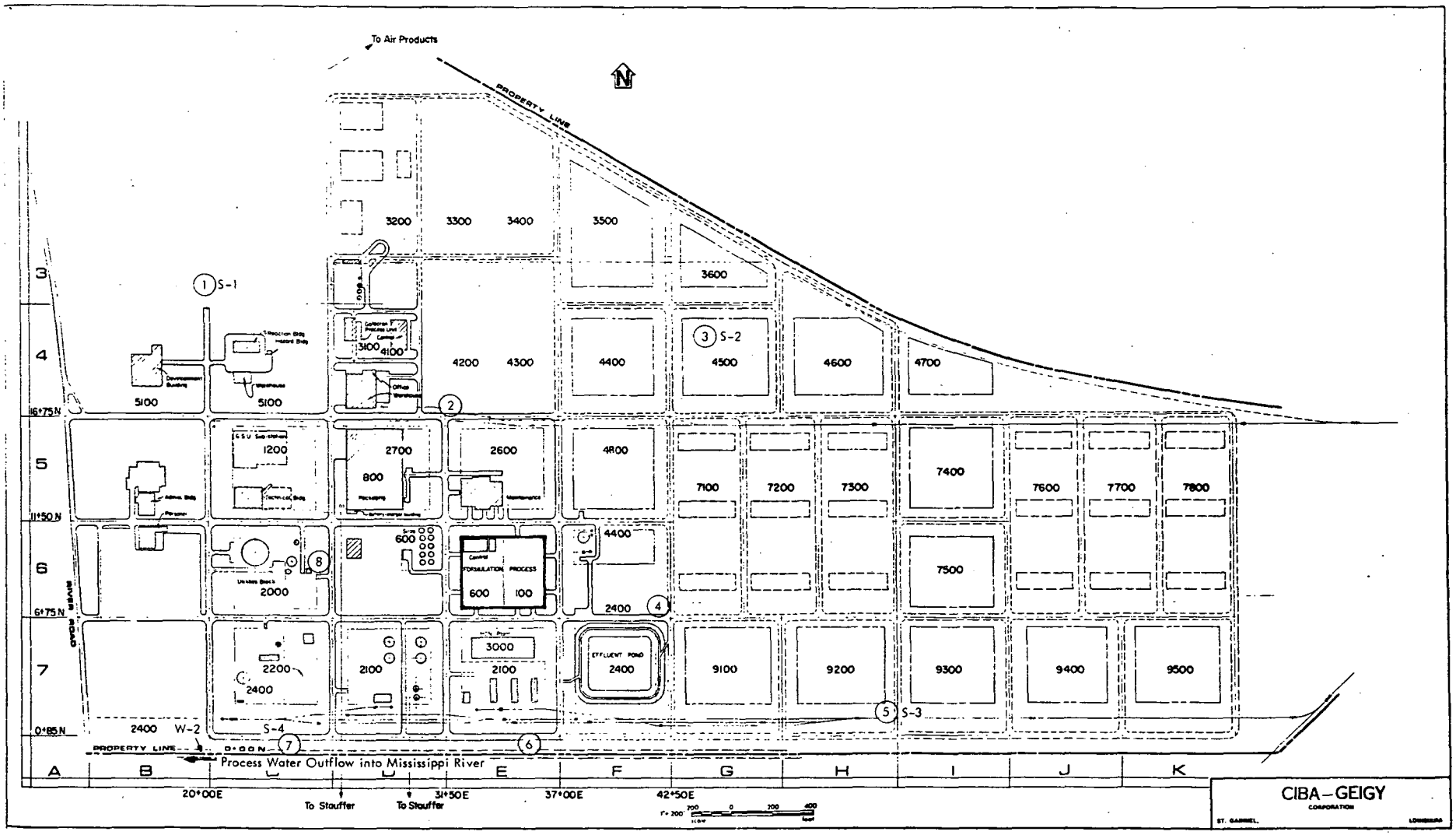


Figure A-8. Sample locations at Ciba-Geigy Corporation, St. Gabriel, Louisiana

Table A-14. AIR SAMPLING AT CIBA-GEIGY CORPORATION, ST. GABRIEL, LOUISIANA

Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
1	2,200 ft northwest of production area	1st 2 hr of 8 hr	7.6	3.5	1,772	4
2	920 ft north of production area	1st 2 hr of 8 hr	8.8	3.5	2,164	4
3	1,600 ft northeast of production area	1st 2 hr of 8 hr	6.8	3.5	1,630	4
4	800 ft east southeast of production area	1st 2 hr of 8 hr	6.3	3.5	1,442	4
5	2,200 ft southeast of production area	1st 2 hr of 8 hr	6.4	3.5	1,561	4
6	950 ft south of production area	1st 2 hr of 8 hr	Lost	3.5	Lost	4
7	1,600 ft southwest of production area	1st 2 hr of 8 hr	6.1	3.5	1,277	4
8	1,000 ft west of production area	1st 2 hr of 8 hr	5.6	3.5	1,298	4

Soil Sampling

Soil sampling was conducted at four locations:

S-1 Northwest of plant at Air Sampling Site No. 1

S-2 Northeast of plant at Air Sampling Site No. 3

S-3 Southeast of plant at Air Sampling Site No. 5

S-4 Southwest of plant at Air Sampling Site No. 7

All soil samples were composites.

Water Sampling

Water sampling was conducted at two locations:

W-1 Raw plant water before use

W-2 Process outflow

All water samples were grab samples.

Sediment Sampling

No sediment samples were taken. The process outflow ditch has a gravel bottom. The effluent pond was not available for sampling, per Ciba-Geigy's request.

Plant Activities and Weather Conditions

The weather conditions during sampling are shown in Table A-15. Plant activities were normal.

Table A-15. WEATHER CONDITIONS DURING SAMPLING AT CIBA-GEIGY, ST. GABRIEL, LOUISIANA

Time	Temperature (°C)	Precipitation	Wind Speed	Wind Direction
August 13				
1500	39	none	3	North northwest
1600	37		7	Northwest
1700	37		5	Northwest
1800	36		5	North northwest
1900	36		4	Southwest
2000	36		7	South
2100	34		5	South
2200	32		5	South
2300	32		3	Southwest
2400	29		5	Southwest
August 14				
0100	27		5	West southwest
0200	27		6	Southwest
0300	27		8	Souwthwest
0400	29		7	West
0500	29		7	West
0600	30		5	West
0700	32		4	West northwest
0800	32		6	Northwest
0900	33		5	Northwest
1000	35		6	North northwest
1100	35		4	North northwest
1200	36		4	North northwest
1300	36		2	North northwest

OLIN CORPORATION, MCINTOSH, ALABAMA

PRESAMPLING SITE SURVEY

The presampling site survey at Olin Corporation's McIntosh, Alabama, plant was conducted on July 23, 1975. The following personnel were present:

Mr. J. Oertling	Works Manager, Olin
Mr. F. Champion	Production Manager, Organic Section, Olin
Mr. C. Hovater	Q. C. Manager, Olin
Mr. R. Reams	Technical Manager, Olin
Mr. N. Barone	Specialist - Environmental Affairs, Olin
Mr. D. Sauter	Midwest Research Institute

Olin Corporation is located approximately 30 miles north of Mobile, Alabama. The area in the immediate vicinity of the plant is flat and marshy. Residential areas around the plant are minimal. Winds are generally from the south during the summer.

The production of pentachloronitrobenzene is by chlorination and nitration of isomeric chlorobenzenes. The production process results in the accumulation of "still bottoms" which are cast into 27-ft^3 blocks containing 80 to 90% HCB. The rated capacity of the plant for PCNB is approximately 7 million pounds per year. Approximately 2.8 to 3.0 million pounds per year of HCB is generated in this process.

Chlorine is also produced by mercury cells using carbon electrodes at a rate of 130,000 tons/year.

Solid wastes (HCB blocks) are stored in an open field in the southeast corner of the plant. The HCB block pile is covered with plastic. This pile represents HCB wastes from the last 2-1/2 years of PCNB production. Ultimately, Olin hopes to reclaim the HCB.

Wastewater is discharged into two open ditches with an average combined flow of 5 million gallons per day. The south ditch, which is adjacent to the PCNB plant and receives runoff from the waste disposal settling pond, contributes mostly to this flow. Both ditches combine outside the plant boundary, and flow into a basin, and ultimately into the Tombigbee River.

At least two landfills were observed. The first landfill is located directly outside the northeast corner of the plant boundary. This landfill was used for HCB disposal before 1971. The second landfill is the southeastern section of the plant is primarily a garbage dump. No landfills are currently in use for chemical disposal.

At the conclusion of the presampling survey, it was agreed upon that field sampling would be tentatively scheduled in the week of August 18, 1975.

FIELD SAMPLING

Field sampling at the Olin plant was conducted on August 18, 1975. Air, soil, water, and sediment samples were collected. Detailed description of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Eight sampling stations were positioned to give two upwind, three midplant, and three downwind sites. Two Tenax®-GC sampling tubes were operated in tandem at each site for three, 3-hr periods. Sampling locations are shown in Figure A-9. The exact distance of each station to the production area was determined and reported along with other sampling data in Table A-16.

Soil Sampling

Composite grab samples were taken outside and inside the plant boundary:

S-1 Downwind (north boundary)

S-2 Old landfill (northeast boundary)

S-3 Brine pond area

S-4 Center road (running north/south)

S-5 High lift route (organic plant to storage area)

S-6 Southeast landfill

S-7 "Hex" storage area

S-8 Old "Hex" dump area

S-9 East road

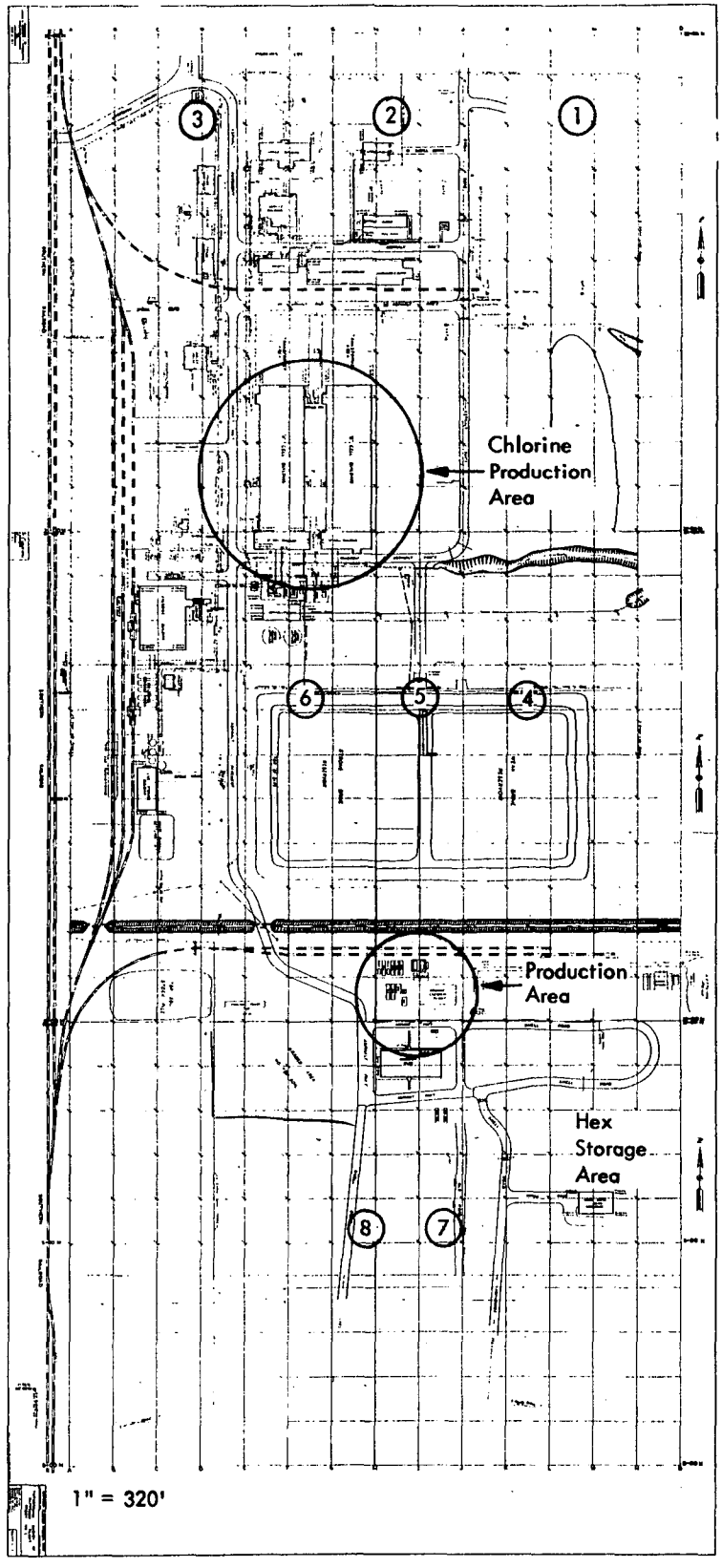


Figure A-9. Sampling locations at Olin Corporation, McIntosh, Alabama

Table A-16. AIR SAMPLING DATA AT OLIN CORPORATION, MCINTOSH, ALABAMA

General area	Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
Far downwind	1	2,100 ft north northeast of production area	1st 3 hr of 12 hr	10.0	3.5	2,103	4
Far downwind	2	2,100 ft north of production area	1st 3 hr of 12 hr	5.2	3.5	1,100	4
Far downwind	3	2,200 ft north northwest of production area	1st 3 hr of 12 hr	5.7	3.5	1,204	4
Near downwind	4	720 ft north northeast of production area	1st 3 hr of 12 hr	6.9	3.5	1,445	20[a/]
Near downwind	5	700 ft north of production area	1st 3 hr of 12 hr	7.0	3.5	1,473	20[a/]
Near downwind	6	800 ft north northwest of production area	1st 3 hr of 12 hr	7.0	3.5	1,472	20[a/]
Upwind	7	640 ft south of production area	1st 3 hr of 12 hr	7.8	3.5	1,630	4
Upwind	8	640 ft south of production area	1st 3 hr of 12 hr	7.3	3.5	1,525	4

a/ Samplers were positioned on Brine Pond dike.

S-10 West road

S-11 Upwind south road

Water Sampling

The following grab samples were obtained:

W-1 Weak brine pond

W-2 Strong brine pond

W-3 Settling pond

W-4 North/south running ditch

W-5 Southern ditch area (upper drop)

W-6 Combined creek (200 yard before basin)

W-7 Basin (at mouth of creek)

W-8 24-Hr composite of plant effluent (combined creek)

W-9 Solar pond, west

W-10 Solar pond, east

Sediment Sampling

One sediment sample was collected at the strong brine pond.

Plant Activities and Weather Conditions

The weather conditions during sampling are shown in Table A-17. Plant activities were normal.

Table A-17. WEATHER CONDITIONS DURING SAMPLING AT OLIN CORPORATION, MCINTOSH, ALABAMA

Time	Temperature (°C)	Barometric pressure (mm Hg)	Wind		Precipitation
			Speed	Direction	
August 18					
1500[a]	37	738	2-4	South	None
1600[a]	37	738	2-4	Southeast	None
1700[a]	36	738	4-10	North northeast	None
1800			6-13	North	Rain
1900			4-6	Southeast	None
2000			2-6	East	None
2100			2-4	Northwest	None
2200			2-4	West	None
2300			-	No wind	None
2400[a]	26	756	2-4	South	None
August 19					
0100[a]	23	741	2-4	Southwest	None
0200[a]	23	740	-	No wind	None
0300[a]	23	740	-	No wind	None
0400			-	No wind	None
0500			-	No wind	None
0600			-	No wind	None
0700			2-4	North northeast	None
0800[a]	25	740	-	No wind	None
0900[a]	27	740	-	No wind	None
1000[a]	29	739	-	No wind	None
1100[a]	32	739	-	No wind	None
1200[a]	35	738	2-6	North	None

a/ Indicates air sampling.

KAISER ALUMINUM AND CHEMICAL CORPORATION, GRAMERCY, LOUISIANA

The presampling site survey at Kaiser Aluminum was conducted on August 14, 1975. The following personnel were present:

Dr. Robert M. Hansen	Research and Development, Kaiser
Mr. Phil Fourmet	Environmental Manager, Kaiser
Mr. Bob Curtis	Environmental Control Specialist, Kaiser
Mr. Phil Kuykendall	Midwest Research Institute

Upon discussion with the plant officials, it was learned that chlorine production utilizing graphite anodes had been terminated in 1973, and was replaced by dimensionally stabilized anodes. For waste disposal, prior to 1973, solid wastes were dispoded by landfill. These residues have since been covered by aluminum production wastes. Water effluent is channeled into the Mississippi river following on-line pH adjustment.

As a result of the change in production technology and the solid wastes disposed since 1973, it was decided that sampling at this plant would yield no usable data. Furthermore, samples collected from the Linden Chlorine Company, at Linden, New Jersey, and the other chlorohydrocarbon plants, which also produce chlorine, would serve the purpose of monitoring the chlorine production plant for the HCB and HCBD emission.

PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

PRESAMPLING SITE SURVEY

The presampling site survey at PPG Industries' Lake Charles, Louisiana, plant was conducted on August 22, 1975. The following personnel were present:

Mr. T. G. Taylor	Technical Plant Manager, PPG
Mr. Thomas C. Jeffery	Chief Process Engineer, PPG
Dr. Earl Gorton	Senior Research Supervisor, Organics, PPG
Mr. C. A. Burns	Environmental Control Specialist, PPG
Mr. Mark Wood	Environmental Analysis Coordinator, PPG
Dr. Raymond Li	Midwest Research Institute

PPG Industries is located more than 2 miles west of downtown Lake Charles, Louisiana. The surrounding terrain is flat and marshy. The PPG canal runs through the plant and into the Calcasieu River which flows to Lake Charles. The closest residential area is about 1 mile northwest of the plant. The wind, in the summer months, is very variable but most likely from south southeast and least likely from the west.

Chemicals produced in this plant include trichloroethylene, perchloroethylene, ethylene dichloride, ethyl chloride, vinylidene chloride, methyl chloroform, vinyl chloride, chlorine, hydrochloric acid, caustic soda, and silica pigments. The current production capacities of trichloro- and perchloroethylene are 350 tons/day. However, the production of 725 tons/day could be achieved and was produced in the past. The plant operates 24 hr a day, 7 days a week.

Trichloro- and perchloroethylene are produced by a catalytic oxychlorination process rather than the thermal process of chlorine and hydrocarbons, thus resulting at a lower reaction temperature. The chlorine is produced in the plant (DSA has been used since 1969). The production wastes are piped into the incinerator and burnt at a residence time from 1/4 to 1/3 sec at 2500°F. The water effluent is channeled into the PPG canal which flows into the Calcasieu River. The PPG canal also received runoffs from the organochlorine production as well as effluents from the power plant.

Prior to the operation of the incinerator, landfill was used for waste disposal. The old landfill site was covered with water. It is still being used for wastes than cannot be burnt in the incinerator or when incinerator breakdown occurs.

At the conclusion of the presampling survey, it was agreed upon that field sampling would be tentatively scheduled in the week of September 2, 1975.

FIELD SAMPLING

Field sampling at the PPG plant was conducted on September 4, 1975. Air, soil, water, and sediment samples were collected. Detailed description of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Ten sampling stations were positioned to encircle the plant's incinerator and organochlorine production area. Two Tenax-GC® sampling tubes were operated in tandem with 24-hr continuous sampling time. Sampling

locations are shown in Figure A-10. The exact distance of each station to the incinerator/production area is listed along with other sampling data in Table A-18.

Soil Sampling

Composite grab samples were taken outside and inside the plant area.

S-1 Landfill composite

S-2 Composite at Air Stations 7, 5, and 4

S-3 Composite at Air Stations 8, 9, and 10

S-4 Composite near Air Station 1 on Columbia Southern Road

Water Sampling

Grab samples were obtained at the following locations:

W-1 Incinerator feed water, lake water

W-2 Scrubber water

W-3 Inlet treatment canal organic effluent before scimmer

W-4 Outlet treatment canal organic effluent after scimmer.

W-5 Surface water, landfill

W-6 Downstream PPG canal, at Mobile Bridge No. 1, 1 gal. taken

W-7 Ship channel, adjacent to Air Station No. 10

Sediment Sampling

Sediment samples were collected at three general areas.

R-1 Downstream PPG canal near Air Sampling Station No. 1

R-2 Main organic plant effluent, near Air Sampling Station No. 2

R-3 PPG ship channel, near Air Sampling Station No. 10

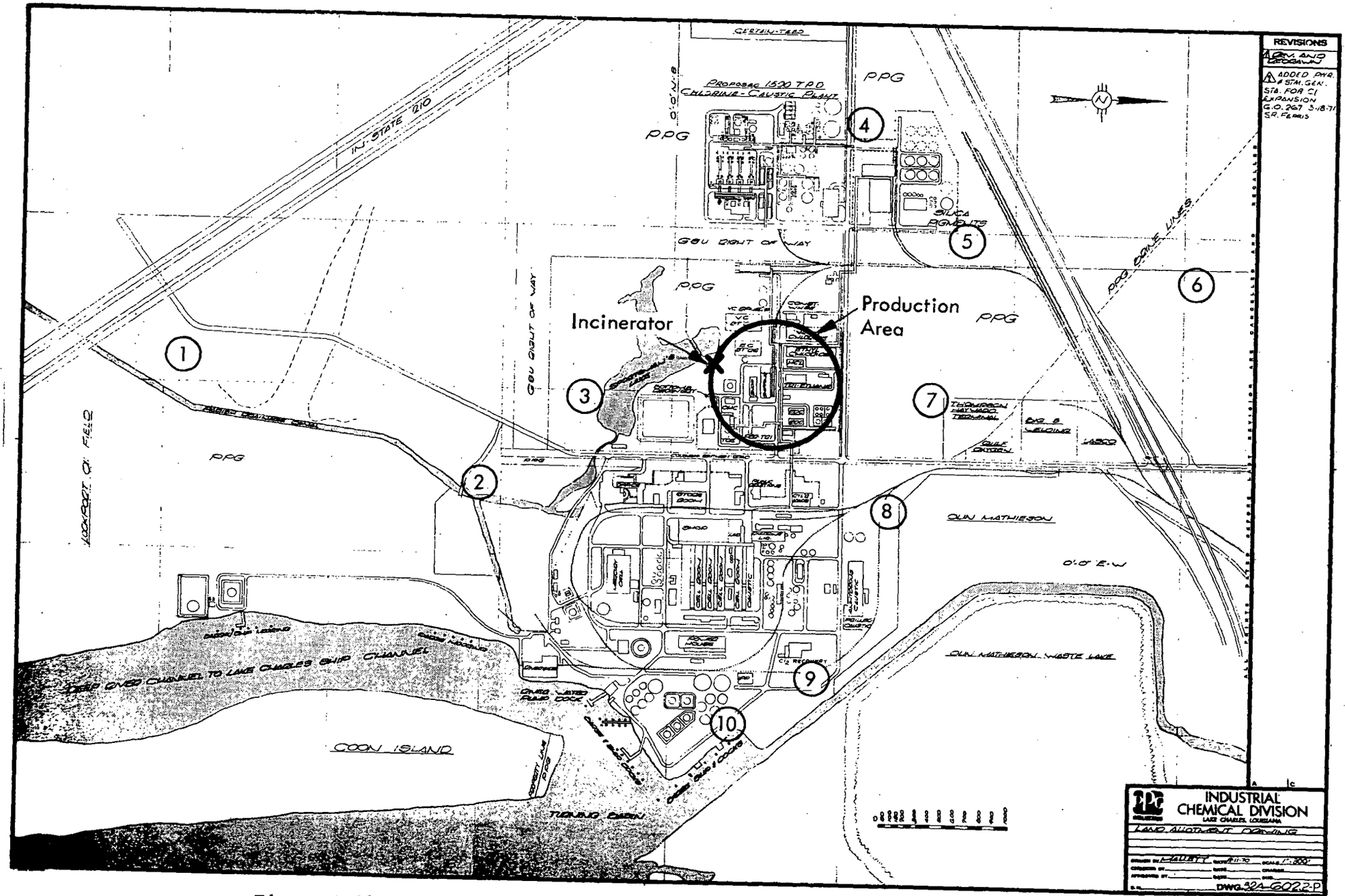


Figure A-10. Sampling locations at PPG Industries, Lake Charles, Louisiana

Table A-18. AIR SAMPLING DATA AT PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

Sample No.[a]	Location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
1	4,700 ft south of production area	24 hr continuous	21.6	0.9	1,180	4
2	2,500 ft south southeast of production area	24 hr continuous	21.4	0.9	1,170	4
3	1,550 ft south of production area	24 hr continuous	21.3	0.9	1,210	4
4	2,300 ft west northwest of production area	24 hr continuous	21.0	0.9	1,170	4
5	2,000 ft northwest of production area	24 hr continuous	19.0[b]	0.9	950[b]	4
6	3,500 ft north northwest of production area	24 hr continuous	22.0	0.9	1,250	4
7	1,250 ft north of production area	24 hr continuous	20.7	0.9	1,180	4
8	1,250 ft northeast of production area	24 hr continuous	21.8	0.9	1,190	4
9	2,250 ft east of production area	24 hr continuous	21.9	0.9	1,250	4
10	2,700 ft east southeast of production area	24 hr continuous	21.9	0.9	1,130	4

a/ Stations were positioned surrounding the production area.
b/ Approximate value due to pump failure, indicates minimum volume.

Plant Activities and Weather Conditions

The weather conditions during sampling are shown in Table A-19. Plant activities were normal.

Table A-19. WEATHER CONDITIONS DURING SAMPLING AT PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

Time	Temperature (°F)	Barometeric pressure (mm Hg)	Wind	
			Speed (mph)	Direction
Spetember 4				
1100	85	763	7	East
1200	86	762	7	East
1300	88	762	7	East
1400	85	762	7	East
1500	85	762	7	East
1600	85	762	7	East southeast
1700	82	762	8	East southeast
1800	80	762	7	East
1900	78	763	5	East
2000	77	763	4	East
2100	76	763	3	East
2200	75	763	2	West
2300	76	762	-	-
2400	75	762	-	-
September 5				
0100	75	762	3	East
0200	75	762	6	East
0300	75	762	5	East
0400	75	762	3	East
0500	75	762	2	East
0600	75	763	3	East
0700	74	763	2	East
0800	73	763	3	East
0900	72	763	3	East[a/]

a/ Rain.

APPENDIX B

ANALYTICAL DATA

Table B-1. HCB CONCENTRATIONS IN AIR SAMPLES FROM VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Sampling station	Sampling time	Volume sampled (liter)	Type of sample	HCB Total ng	HCB $\mu g/m^3$ [a]
1	1935-2035	26	Filter	5	0.6
			Tenax	10	
	0120-0227	42	Filter	< 2	0.2
			Tenax	7	
	0430-0523	33	Filter	< 2	0.2
			Tenax	6	
	0841-0941	37	Filter	< 2	0.3
			Tenax	10	
	1320-1424	40	Filter	4	0.4
			Tenax	10	
2	1935-2035	17	Filter	167	11.3
			Tenax	25	
	0120-0227	36	Filter	< 2	0.6
			Tenax	18	
	0430-0523	29	Filter	< 2	1.6
			Tenax	45	
	0841-0941	32	Filter	< 2	0.3
			Tenax	7	
	1320-1424	35	Filter	< 2	1.0
			Tenax	32	
3	1935-	Lost	Filter		
			Tenax		
	0120-0227	57	Filter	< 2	0.1
			Tenax	5	
	0430-0523	45	Filter	< 2	0.2
			Tenax	6	
	0841-0941	51	Filter	< 2	0.2
			Tenax	8	
	1320-1424	54	Filter	65	1.7
			Tenax	25	
4	1945-2045	27	Filter	275	11.5
			Tenax	35	
	0110-0220	32	Filter	2.5	0.6
			Tenax	18	
	0450-0555	30	Filter	< 2	0.8
			Tenax	20	
	0902-1010	31	Filter	270	9.7
			Tenax	30	
	1307-1425	36	Filter	144	5.0
			Tenax	36	

Table B-1. (continued)

Sampling station	Sampling time	Volume sampled (liter)	Type of sample	HCB Total ng	HCB µg/m³
5	1945-2045	41	Filter	100	3.4
			Tenax	40	
	0110-0220	48	Filter	38	1.1
			Tenax	15	
	0450-0555	44	Filter	2.2	0.4
			Tenax	12	
	0902-1010	46	Filter	50	1.4
			Tenax	16	
	1307-1425	53	Filter	75	1.5
			Tenax	3.3	
6	1945-2045	34	Filter	< 2	1.1
			Tenax	38	
	0110-0220	40	Filter	< 2	0.5
			Tenax	18	
	0450-0555	37	Filter	< 2	0.4
			Tenax	14	
	0902-1010	39	Filter	900	23.6
			Tenax	21	
	1307-1425	45	Filter	119	3.0
			Tenax	20	
7	1950-2050	148	Filter	< 2	1.7
			Tenax	250	
	0051-0158	166	Filter	< 2	0.8
			Tenax	125	
	0456-0555	146	Filter	< 2	1.0
			Tenax	142	
	0904-1010	163	Filter	< 2	0.9
			Tenax	150	
	1308-1425	190	Filter	< 2	1.5
			Tenax	275	
8	1950-2050	212	Filter	< 2	0.6
			Tenax	116	
	0103-0202	209	Filter	< 2	0.2
			Tenax	46	
	0458-0600	220	Filter	< 2	0.3
			Tenax	66	
	0908-1010	227	Filter	< 2	0.3
			Tenax	66	
	1308-1420	255	Filter	< 2	0.4
			Tenax	88	

Table B-1. (continued)

Sampling station	Sampling time	Volume sampled (liter)	Type of sample	HCB Total ng	HCB µg/m³
9	1950-2050	227	Filter	7	0.8
			Tenax	167	
	0103-0202	223	Filter	< 2	0.3
			Tenax	65	
	0458-0600	234	Filter	< 2	0.5
			Tenax	114	
	0908-1010	242	Filter	< 2	0.4
			Tenax	87	
	1308-1420	272	Filter	7	0.5
			Tenax	135	
10	1910-2310	809	Filter	< 2	0.3
			Tenax	250	
	0005-0330	691	Filter	< 2	0.2
			Tenax	150	
	0340-0728	768	Filter	< 2	0.2
			Tenax	134	
	0737-1117	741	Filter	< 2	0.1
			Tenax	91	
	1124-1433	637	Filter	< 2	0.2
			Tenax	97	
11	1910-2310	856	Filter	< 2	0.3
			Tenax	278	
	0005-0330	732	Filter	< 2	0.2
			Tenax	109	
	0340-0728	814	Filter	< 2	0.1
			Tenax	75	
	0737-1117	785	Filter	< 2	0.1
			Tenax	78	
	1124-1433	675	Filter	Sample lost	0.2
			Tenax	106	
12	1919-2315	863	Filter	14	1.0
			Tenax	850	
	2350-0315	738	Filter	< 2	0.4
			Tenax	300	
	0325-0712	817	Filter	815	1.2
			Tenax	190	
	0721-1101	792	Filter	875	1.4
			Tenax	210	
	1113-1433	720	Filter	18	0.4
			Tenax	260	

Table B-1. (continued)

Sampling station	Sampling time	Volume sampled (liter)	Type of sample	HCB Total ng	HCB µg/m³
13	1915-2315	917	Filter	5	1.0
			Tenax	950	
	2350-0315	784	Filter	91	0.5
			Tenax	320	
	0325-0712	867	Filter	950	1.3
			Tenax	200	
	0721-1101	840	Filter	900	1.3
			Tenax	180	
	1113-1433	764	Filter	980	1.7
			Tenax	280	
14	1920-2259	806	Filter	< 2	0.1
			Tenax	40	
	2335-0258	816	Filter	< 2	0.1
			Tenax	41	
	0310-0640	844	Filter	< 2	0.1
			Tenax	60	
	0650-1046	949	Filter	< 2	0.1
			Tenax	85	
	1057-1435	876	Filter	< 2	0.3
			Tenax	225	
15	1920-2259	837	Filter	< 2	0.03
			Tenax	25	
	2335-0258	805	Filter	< 2	0.1
			Tenax	60	
	0310-0640	832	Filter	< 2	0.1
			Tenax	97	
	0650-1046	935	Filter	< 2	0.1
			Tenax	81	
	1057-1435	863	Filter	5	0.2
			Tenax	200	
16	2005-0010	862	Filter	4	0.4
			Tenax	330	
	0025-0345	702	Filter	< 2	0.2
			Tenax	138	
	0355-0744	804	Filter	< 2	0.2
			Tenax	160	
	0752-1154	849	Filter	21	0.3
			Tenax	230	
	1200-1430	527	Filter	< 2	0.8
			Tenax	440	

Table B-1. (concluded)

Sampling station	Sampling time	Volume sampled (liter)	Type of sample	HCB Total ng	HCB µg/m³
17	2005-0010	730	Filter	ND[b]	0.6
			Tenax	420	
	0025-0345	596	Filter	< 2	0.3
			Tenax	200	
	0355-0744	682	Filter	950	1.6
			Tenax	130	
	0752-1154	721	Filter	21	0.8
			Tenax	350	
	1200-1430	447	Filter	925	2.1
			Tenax	330	
18	2010-	548	Filter	9	0.7
			Tenax	395	
	0022-0355	711	Filter	< 2	0.3
			Tenax	230	
	0400-0755	785	Filter	935	1.6
			Tenax	280	
	0759-1121	858	Filter	3	0.6
			Tenax	490	
	1215-1430	451	Filter	10	0.7
			Tenax	320	
Tenax® GC blank					ND
Millipore filter blank					ND

a/ Concentration based on the sum of ng found on the filter and Tenax.
b/ ND - None detected.

Table B-2. HCB CONCENTRATIONS IN AIR SAMPLES FROM STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Sampling station	Sampling time	Type of sample	Volume sampled (liter)	Concentration ($\mu g/m^3$) HCB
1	1000-1400	Tenax®-GC	415	0.04
	1400-1800	Tenax®-GC	414	0.07
	1800-2200	Tenax®-GC	540	0.12
	2200-0200	Tenax®-GC	390	0.05
	0200-0600	Tenax®-GC	380	0.05
	0600-1000	Tenax®-GC	449	0.08
		Filters		< 0.01
2	1000-1400	Tenax®-GC	455	0.08
	1400-1800	Tenax®-GC	408	0.04
	1800-2200	Tenax®-GC	514	0.04
	2200-0200	Tenax®-GC	463	0.03
	0200-0600	Tenax®-GC	336	0.07
	0600-1000	Tenax®-GC	450	0.05
		Filters		< 0.01
3	1000-1400	Tenax®-GC	384	1.1
	1400-1800	Tenax®-GC	438	1.4
	1800-2200	Tenax®-GC	490	0.35
	2200-0200	Tenax®-GC	448	0.35
	0200-0600	Tenax®-GC	Lost	-
	0600-1000	Tenax®-GC	461	0.19
		Filters		0.07
4	1000-1400	Tenax®-GC	432	2.56
	1400-1800	Tenax®-GC	456	2.06
	1800-2200	Tenax®-GC	454	0.62
	2200-0200	Tenax®-GC	519	0.42
	0200-0600	Tenax®-GC	437	0.16
	0600-1000	Tenax®-GC	470	0.13
		Filters		1.0
5	1000-1400	Tenax®-GC	420	0.85
	1400-1800	Tenax®-GC	470	1.03
	1800-2200	Tenax®-GC	540	0.71
	2200-0200	Tenax®-GC	496	0.36
	0200-0600	Tenax®-GC	426	0.20
	0600-1000	Tenax®-GC	435	0.34
		Filters		0.09

Table B-2. (concluded)

Sampling station	Sampling time	Type of sample	Volume sampled (liter)	Concentration ($\mu g/m^3$) HCB
6	1000-1400	Tenax®-GC	408	0.69
	1400-1800	Tenax®-GC	455	0.71
	1800-2200	Tenax®-GC	464	0.45
	2200-0200	Tenax®-GC	442	0.31
	0200-0600	Tenax®-GC	425	0.11
	0600-1000	Tenax®-GC	468	0.19
		Filters		0.20
7	1000-1400	Tenax®-GC	450	0.57
	1400-1800	Tenax®-GC	472	0.53
	1800-2200	Tenax®-GC	563	0.24
	2200-0200	Tenax®-GC	469	0.23
	0200-0600	Tenax®-GC	426	0.12
	0600-1000	Tenax®-GC	470	0.28
		Filters		< 0.01
8	1000-1400	Tenax®-GC	450	0.32
	1400-1800	Tenax®-GC	488	0.20
	1800-2200	Tenax®-GC	554	0.07
	2200-0200	Tenax®-GC	476	0.16
	0200-0600	Tenax®-GC	436	0.05
	0600-1000	Tenax®-GC	468	0.06
		Filters		0.01
9	1000-1400	Tenax®-GC	455	0.05
	1400-1800	Tenax®-GC	492	0.06
	1800-2200	Tenax®-GC	562	0.02
	2200-0200	Tenax®-GC	483	0.05
	0200-0600	Tenax®-GC	466	0.02
	0600-1000	Tenax®-GC	468	0.03
		Filters		< 0.01

Table B-3. HCB CONCENTRATIONS IN AIR SAMPLES FROM DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA

Sampling station	Type of sample	Volume sampled (liter)	Concentration ($\mu g/m^3$) HCB
1	Tenax®GC (front)	4,336	0.02
	Tenax®GC (back)		ND
	Filter		ND
2	Tenax®GC (front)	427	< 0.02
	Tenax®GC (back)		ND
	Filter		ND
3	Tenax®GC (front)	4,166	0.05
	Tenax®GC (back)		ND
	Filter		ND
4	Tenax®GC (front)	3,870	0.08
	Tenax®GC (back)		ND
	Filter		ND
5	Tenax®GC (front)	3,713	0.02
	Tenax®GC (back)		ND
	Filter		ND
6	Tenax®GC (front)	4,314	< 0.02
	Tenax®GC (back)		ND
			ND
7	Tenax®GC (front)	962	< 0.02
	Tenax®GC (back)		ND
	Filter		ND
8	Tenax®GC (front)	3,963	< 0.02
	Tenax®GC (back)		ND
	Filter		ND

Note: ND = none detected.

Table B-4. HCB CONCENTRATIONS IN AIR SAMPLES FROM E. I. DU PONT DE NEMOURS AND COMPANY, CORPUS CHRISTI, TEXAS

Sampling station	Type of sample	Volume sampled (ℓ)	Concentration (μg/m^3) HCB
1	Tenax®-GC, front	4,371	ND
	Tenax®-GC, back	4,371	ND
	Filter	4,371	ND
2	Tenax®-GC, front	3,621	ND
	Tenax®-GC, back	3,621	ND
	Filter	3,621	ND
3	Tenax®-GC, front	4,070	ND
	Tenax®-GC, back	4,070	ND
	Filter	4,070	ND
4	Tenax®-GC, front	4,007	ND
	Tenax®-GC, back	4,007	ND
	Filter	4,007	ND
5	Tenax®-GC, front	3,965	ND
	Tenax®-GC, back	3,965	ND
	Filter	3,965	ND

Table B-5. HCB CONCENTRATIONS IN AIR SAMPLES FROM DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

Sampling station	Type of sample	Volume sampled (ℓ)	Concentration (μg/m³) HCB
1	Tenax®-GC, front	580	ND
	Tenax®-GC, back	580	ND
	Filter	580	ND
2	Tenax®-GC, front	598	ND
	Tenax®-GC, back	598	ND
	Filter	598	ND
3	Tenax®-GC, front	485	ND
	Tenax®-GC, back	485	ND
	Filter	485	ND
4	Tenax®-GC, front	540	ND
	Tenax®-GC, back	540	ND
	Filter	540	ND
5	Tenax®-GC, front	542	ND
	Tenax®-GC, back	542	ND
	Filter	542	ND
6	Tenax®-GC, front	608	ND
	Tenax®-GC, back	608	ND
	Filter	608	ND
7	Tenax®-GC, front	559	ND
	Tenax®-GC, back	559	ND
	Filter	559	ND
8	Tenax®-GC, front	555	ND
	Tenax®-GC, back	555	ND
	Filter	555	ND

Table B-6. HCB CONCENTRATIONS IN AIR SAMPLES FROM CIBA-GEIGY CORPORATION, ST. GABRIEL, LOUISIANA

Sampling station	Type of sample	Volume sampled (ℓ)	Concentration ($\mu g/m^3$) HCB
1	Tenax®-GC	1,772	0.02
	Filter	1,772	ND
2	Tenax®-GC	2,164	0.01
	Filter	2,164	ND
3	Tenax®-GC	1,630	0.02
	Filter	1,630	ND
4	Tenax®-GC	1,442	0.01
	Filter	1,442	ND
5	Tenax®-GC	1,561	0.01
	Filter	1,561	ND
6	Lost	Lost	Lost
7	Tenax®-GC	1,277	ND
	Filter	1,277	ND
8	Tenax®-GC	1,298	ND
	Filter	1,298	ND

Table B-7. HCB CONCENTRATIONS IN AIR SAMPLES FROM OLIN CORPORATION, MCINTOSH, ALABAMA

Sampling station	Type of sample	Volume sampled (ℓ)	Concentration (μg/m³) HCB
1	Tenax®-GC, front	2,103	0.26
	Tenax®-GC, back	2,103	ND
	Filter	2,103	ND
2	Tenax®-GC, front	1,100	0.06
	Tenax®-GC, back	1,100	ND
	Filter	1,100	ND
3	Tenax®-GC, front	1,204	0.04
	Tenax®-GC, back	1,204	ND
	Filter	1,204	ND
4	Tenax®-GC, front	1,445	0.48
	Tenax®-GC, back	1,445	0.08
	Filter	1,445	ND
5	Tenax®-GC, front	1,473	0.24
	Tenax®-GC, back	1,473	ND
	Filter	1,473	ND
6	Tenax®-GC, front	1,472	0.03
	Tenax®-GC, back	1,472	ND
	Filter	1,472	ND
7	Tenax®-GC, front	1,630	2.22
	Tenax®-GC, back	1,630	ND
	Filter	1,630	ND
8	Tenax®-GC, front	1,525	1.11
	Tenax®-GC, back	1,525	ND
	Filter	1,525	ND

Table B-8. HCB CONCENTRATIONS IN AIR SAMPLES FROM PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

Sampling station	Type of sample	Volume sampled (ℓ)	Concentration (μg/m³) HCB
1	Tenax®-GC, front	1,180	0.02
	Tenax®-GC, back	1,180	ND
	Filter	1,180	0.42
2	Tenax®-GC, front	1,170	0.02
	Tenax®-GC, back	1,170	ND
	Filter	1,170	0.07
3	Tenax®-GC, front	1,210	0.03
	Tenax®-GC, back	1,210	ND
	Filter	1,210	0.13
4	Tenax®-GC, front	1,170	ND
	Tenax®-GC, back	1,170	ND
	Filter	1,170	1.30
5	Tenax®-GC, front	950	ND
	Tenax®-GC, back	950	ND
	Filter	950	1.47
6	Tenax®-GC, front	1,250	0.03
	Tenax®-GC, back	1,250	ND
	Filter	1,250	0.33
7	Tenax®-GC, front	1,180	0.03
	Tenax®-GC, back	1,180	ND
	Filter	1,180	1.63
8	Tenax®-GC, front	1,190	ND
	Tenax®-GC, back	1,190	ND
	Filter	1,190	ND
9	Tenax®-GC, front	1,250	ND
	Tenax®-GC, back	1,250	ND
	Filter	1,250	ND
10	Tenax®-GC, front	1,130	ND
	Tenax®-GC, back	1,130	ND
	Filter	1,130	0.02

APPENDIX C

METHOD DEVELOPMENT FOR SAMPLING AND ANALYSIS

LITERATURE SEARCH

A search of the literature to 1967 revealed that there was no specific method for sampling HCB in water. The method used most often for sampling pollutants in water was the "grab technique." Detailed procedures of the grab technique can be found in many of the standard method texts.[1-3] HCB in water samples is concentrated by extraction with appropriate organic solvents. In some cases, HCB is concentrated by passing the water sample through a column filled with an appropriate trapping medium. Gesser et al.[4] used a glass column with two polyurethane plugs, and found that HCB, along with a number of polychlorinated biphenyls, could be absorbed on the column. These compounds were then extracted by treating with acetone and hexane. This kind of concentration technique, with other trapping media such as activated charcoal, and polystyrene copolymer, Amberlite XAD-2 and XAD-4, has been successfully applied in trapping other chlorinated pesticides.[5-10]

Sampling of HCB in air is generally carried out by trapping the compound either in an appropriate organic solvent or in an appropriate organic resin. Columns of wood-charcoal cigarette-filter in series as well as silica gel have been used to trap HCB in air.[11-14] The HCB is recovered by appropriate solvent extraction. Organic resins such as Chromosorb A and Chromosorb 101 have been used to trap HCB and other chlorinated pesticides.[15,16] A nylon-chiffon cloth (0.25 or 0.5 m^2) impregnated with ethanediol and held vertically in a wooden frame and exposed to the atmosphere for 5 days has been reported to trap organochlorine as well as organophosphorus compounds.[17] Another organic resin, Tenax®-GC, has been reported to be an efficient trapping medium for aromatic hydrocarbon and organochlorine.[18]

WATER SAMPLING AND RECOVERY STUDIES

As a result of the literature search, it was decided that for water sampling, the "grab" method would be employed as the primary method. If the concentration of HCB was suspected to be low, an Amberlite XAD-4 column would be used to concentrate the two substances. Both sampling techniques were evaluated prior to actual field sampling.

Hexane Extraction

Table C-1 shows the results of recovery studies for n-hexane extraction of HCB from water samples fortified with 1 to 30 µg/liter. Each of the water samples (1 liter) was extracted with three 10-ml aliquots n-hexane, made up to the mark of a 100-ml volumetric flask, and analyzed for HCB by gas chromatography. The average recovery was over 100% for HCB. The slightly positive error observed in the HCB recovery studies was probably due to the fact that the fortified HCB samples and the standard HCB solution used for calibration were prepared from two different stock solutions.

Table C-1. RECOVERY STUDIES OF HCB BY n-HEXANE EXTRACTION

Sample	Amount in 1 liter of water (μg)	Amount found in n-hexane (μg)	% Recovery
1	1	1.1	110
2	2	2.2	110
3	3	3.4	113
4	5	5	100
5	10	11	110
6	20	22	110
7	30	32	106
Blank	None	None	

Elution from Amberlite XAD-4

Water samples fortified with 1 to 30 μg/liter of HCB were passed through Amberlite XAD-4 columns containing 7 g of the trapping material. Recovery of the two substances was accomplished by eluting, first with a small amount of acetone, followed by a larger volume of n-hexane. Table C-2 shows that the average recovery of HCB was greater than 70% (first five runs).* Recoveries of HCB with either a closed or open system showed no significant difference, indicating volatilization of HCB is not a problem over short time periods.

Table C-2. RECOVERY OF HCB FROM WATER BY CONCENTRATION ON XAD-4

Run (μg/ℓ)	Flow rate (ml/min)	Recovery from n-hexane elution (%)	Recovery from Soxhlet extraction	Recovery from water extraction (%)
1 (30)	2	65	0	13
2 (30)	2	63	0	17
3 (1)	2	85	0	0
4 (5)	2	73	0	0
5 (30)	8	80	0	3
6 (30)a/	10	77	0	14

a/ Closed elution system.

* When actual environmental samples were analyzed, the amount of Amberlite XAD-4 used in sampling (250 g) was so high that recovery by elution was very time-consuming. Elution was replaced by overnight Soxhlet extraction.

Evidence of volatilization loss of HCB was demonstrated by the results of the following experiment. Five 250 ml water samples fortified with 5 μg/liter of HCB were placed in five 250 ml separatory funnels. Three of the funnels were left uncapped overnight while the remaining two funnels were capped. Each water sample was then extracted and analyzed for HCB. The results, shown in Table C-3, indicate that measurable amounts of HCB were lost due to volatilization from the open system.

Table C-3. LOSS OF HCB DUE TO VAPORIZATION

Run	Separatory funnel	HCB recovery (%)
1	Capped	94
2	Capped	94
3	Uncapped	58
4	Uncapped	55
5	Uncapped	51

AIR SAMPLING AND RECOVERY STUDIES

Chromosorb 101 and Tenax®-GC were tested for their trapping efficiency utilizing a device shown in Figure C-1. This device was initially designed to check the recovery of HCB from water by vaporization at reduced pressure. The results also indicate efficiency for collecting HCB from water-saturated air.

One liter of water, fortified with 1 to 30 μg/liter of HCB, was placed in a one-neck 24/40 flask with a thermometer pit so that the water temperature was monitored. The vapor was drawn through a Tenax®-GC or Chromosorb 101 (approximately 1 g) column with a vacuum pump or water aspirator. The vapor flow rate through the trapping column was monitored with a calibrated flowmeter and was maintained at 3 liters/min. After passing a known volume of vapor through the column, the trapping material was first extracted with n-hexane in an ultrasonic bath, then by overnight Soxhlet extraction. The remaining water was also extracted with n-hexane. All the extracts were analyzed for HCB by electron capture gas chromatography.

Table C-4 shows the results of a set of seven experiments. Runs 1 and 2 were designed to compare the efficiency of Chromosorb 101 and Tenax®-GC, while Runs 3 through 7 were repeated experiments to evaluate the efficiency of Tenax®-GC at various HCB concentrations. In general, under these experimental conditions, the trapping and recovery of HCB with Tenax®-GC is more effective than with Chromosorb 101.

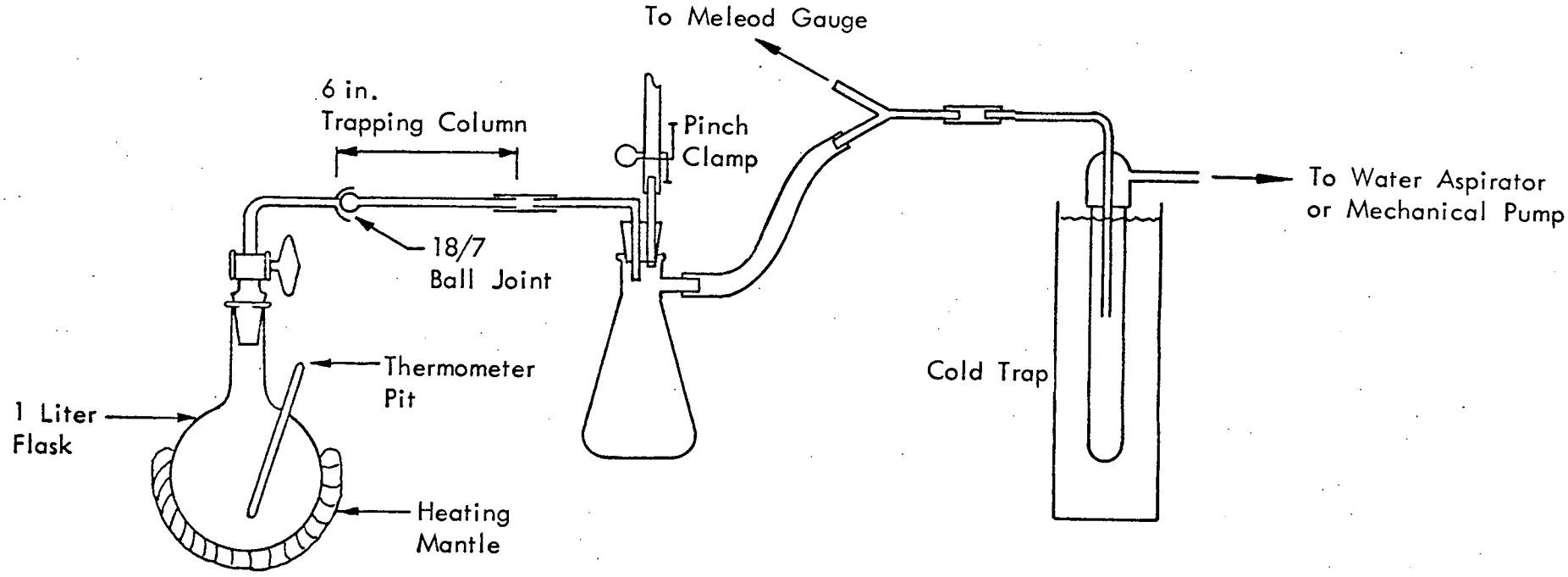


Figure C-1. Apparatus for recovery of HCB from water by vaporization at reduced pressure

Table C-4. RECOVERY OF HCB FROM WATER-SATURATED AIR

Run	Column	Total HCB in sample (μg)	% Recovery from ultrasonic extraction	% Recovery from Soxhlet extraction	% Recovery from extraction of water
1	Chromosorb 101	30	None detected	None detected	46
2	Tenax®-GC	30	100	Trace	6
3	Tenax®-GC	1	94	Trace	5
4	Tenax®-GC	1	85	Trace	2
5	Tenax®-GC	5	102	Trace	4
6	Tenax®-GC	30	90	2	3
7	Tenax®-GC	30	86	2	9

SEDIMENT SAMPLING AND RECOVERY STUDIES

Sediment was taken from two Kansas City area creeks. The samples were collected from the top 1 to 2 in. of sediment. The samples were fortified with HCB and recoveries were determined using standard procedures (for sediments) described in the Manual of Analytical Methods prepared by the Pesticides and Toxic Substances Effects Laboratory of the National Environmental Research Center, USEPA. A Florisil™ column was used for sample cleanup and 6% ethyl ether in petroleum ether was used for the elution of HCB.

Two different procedures were used to prepare fortified sediment samples. In the first method, HCB was added to known amounts of sediment prior to evaporation of moisture from the sediment; in the second method, HCB was added after the moisture in the sediment had evaporated almost to dryness. Results of the recovery studies of these sediment samples (Runs 1 through 4) are shown in Table C-5.

Because of the low recovery of HCB in Runs 1 through 4, direct Soxhlet extraction (1:1 acetone/hexane) of the fortified sediment samples was tested. Runs 6 and 7 in Table C-5 show the results of Soxhlet extraction. The recovery of HCB improved significantly. Therefore, direct Soxhlet extraction of the sediment was chosen as the standard method. However, if interferences from other impurities were present, Florisil™ cleanup would be used. The amount of HCB determined in the sample analysis was reported on dry weight basis.

Table C-5. RECOVERY OF HCB FROM SEDIMENTS

Run	Sample weight (g)	Amount HCB added (μg)	% Recovery (HCB)
1[a/]	50	5	59
2[a/]	50	5	64
3[b/]	50	5	73
4[b/]	50	5	67
5	50	Control	ND
6[c/]	50	5	95
7[c/]	50	5	98

a/ HCB added before moisture in sample was almost evaporated to dryness.
b/ HCB added after moisture in sample was almost evaporated to dryness.
c/ Direct Soxhlet extract of sample.

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16. ABSTRACT

Nine industrial plants were sampled to determine hexachlorobenzene (HCB) levels in air, water, soil and sediment. The plants represent six major industries: perchloroethylene, trichloroethylene, carbon tetrachloride, chlorine, triazine herbicides, and pentachloronitrobenzene. In general, higher levels of HCB were associated with the production of lower chlorinated hydrocarbons than with the production of other chemicals. HCB levels in soil and air at the pentachloronitrobenzene plant were relatively high. The levels of HCB associated with plants producting chlorine and triazine herbicides were very low. Several different waste-disposal methods were used at the perchloro- and trichloroethylene plants that were sampled. The highest level of HCB was detected in air and soil at the plant using on-site landfill and open pit storage. High HCB levels were detected in loading and transfer areas at plants using off-site disposal methods.

The highest level of HCB found in the air on plant property was 24 $\mu g/m^3$. The HCB level in an open waste treatment pond was 306 µg/liter. The level of HCB in soil within the plant area was over 1,000 µg/g at three plants. The maximum concentration
(continued)

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16. Abstract

of HCB in air sampled off plant property was 0.36 μg/m^3. A level of 3 μg/m^3 was detected at the boundary of another plant. Soil taken from a cornfield adjacent to one plant contained 1.1 μg/g, and over 3,000 μg/g were detected along a boundary road of another. HCB levels in water sampled beyond the plant property exceeded 1 μg/liter at two plants.

Samples were collected from two sewage treatment plants; negligible quantities of HCB were detected.