ENVIRONMENTAL MONITORING NEAR INDUSTRIAL SITES METHYLCHLOROFORM



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BATTELLE Columbus Laboratories 505 King Avenue Columbus, Ohio 43201

Vincent J. DeCarlo Project Officer

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EXECUTIVE SUMMARY

The levels of methylchloroform in various environmental media were determined at four production sites, one user site, and a background site. The following sites were monitored:

Dow Chemical Co., U.S.A. . . . Freeport, Texas
PPG Industries, Inc. Lake Charles, Louisiana
Ethyl Corporation. Baton Rouge, Louisiana
Vulcan Materials Company . . . Geismor, Louisiana
Boeing Company (User Site) . . Seattle, Washington
St. Francis National Forest. . . Helena, Arkansas
(Background Site)

Approximately 2 days were devoted to monitoring ambient air levels for methylchloroform and collecting water, soil, and sediment samples at each site. The samples were returned to Battelle-Columbus Laboratories for analyses. The ambient air level of methylchloroform was determined on-site by direct injection of the ambient air into a gas chromatograph followed by detection and quantification with an electron capture detector.

For the analyses of water samples, the methylchloroform was sparged from the water and collected on a trap material using a commercial liquid sample concentrator. The trapped organic material was then backflushed onto a gas chromatograph column which was connected to an electron capture detector used to quantify the methylchloroform in the original sample. A similar technique was used for the quantification of methylchloroform in soil and sediment but the apparatus was not of commercial design.

For each site, a map is presented with sampling points indicated. The results from the analyses of the samples and detailed descriptions of the sampling locations are given and are keyed to the site map.

Considerable variation was observed in the maximum downwind levels of methylchloroform at various production plants. Concentrations of methylchloroform in ambient air ranged from less than 0.3 ppb (limit of detection) to 155 ppb.

Concentrations of methylchloroform in surface water in the vicinity of the production and user plants was even more variable ranging from fractions of a ppb to over 16 ppm. Concentrations in soil and sediment ranged from the limits of detection to 6.1 ppb.

1. INTRODUCTION

Methylchloroform (MC) is a chlorinated hydrocarbon which is produced in major quantities in the U.S. and is used in a variety of solvent cleaning operations. This compound has a relatively low boiling point; therefore, its emission into the atmosphere probably represents one of the more significant pathways to human exposure. To date, however, very little air monitoring data have been generated to assess potential exposure hazards. In particular, existing data are devoid of measurements in the environment around manufacturing and user facilities where the highest concentrations (and thus the highest exposures) might be expected.

This report describes the sampling rationale, the collection of samples, that is, the sampling protocol, and the analytical methods used to determine the environmental concentrations of methylchloroform at several sites. The results are presented using maps in conjunction with tabulated data and descriptions of the samples. A separate set of data is presented for each site monitored, and these sets are grouped together under production sites, user sites, and background site.

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2. SAMPLING RATIONALE

The objective of this sampling program was to determine levels of methylchloroform in the environment. To do this, several important factors were considered. Among these were the type of site (production, user, or background); the source of the substance (discharge practices—how the substance is released to the environment); the ecological compartments to be sampled (air, water, soil, sediment, biota); the conditions at the time of sampling (meteorological conditions, plant operation, geography, interfering elements); and statistical requirements. These factors are discussed further under specific environmental compartments in the following section on the sampling protocol.

Sites were selected based on the fact that methylchloroform is a volatile organic compound and is most likely to reach the environment where it is produced and used. Table 2.1 lists the four major producers of methylchloroform, a major user site, and a background site.

TABLE 2.1. SITES MONITORED FOR METHYLCHLOROFORM

<u>Production Sites</u>

Dow Chemical Company, U.S.A. . . Freeport, Texas
PPG Industries, Inc. Lake Charles, Louisiana
Ethyl Corporation Baton Rouge, Louisiana
Vulcan Materials Company Geismor, Louisiana

User Site

Boeing Company Seattle, Washington

Background Site

St. Francis National Forest . . . Helena, Arkansas

The air sampling effort at each facility was conducted to obtain the following information: (1) the concentration profile around the plant, (2) maximum concentration levels, (3) temporal variations in concentration, and

(4) the variation in concentration as a function of distance downwind from the plant.

Measurements were made in four quadrants surrounding the plant location. The highest concentrations prevailed downwind from the plant location. Therefore, the majority of the sampling and analysis effort was then concentrated in the downwind direction to determine maximum concentrations and temporal and spatial concentration variations.

The air monitoring equipment, a field electron-capture gas chromatograph, used for the methylchloroform analyses also permitted measurement of trichloroethylene, carbon tetrachloride, and perchloroethylene. Therefore, concentrations of these chlorinated hydrocarbons in ambient air were also determined.

In order to detect concentration levels associated with process water discharge, water samples were taken in the receiving stream at the plant outfall and upstream and downstream of the outfall. Samples of aquatic animal tissue, usually fish, were also collected at locations upstream and downstream of plant outfalls. In order to measure the amount present in a normal day's discharge, which may not be accurately represented in grab samples, a 24-hour composite of the effluent was obtained from plant personnel. Water samples were also taken from the naturally occurring surface waters in the immediate area.

In order to determine possible associated levels of methylchloroform in sediments, samples were taken in close proximity to water sampling sites.

Soil, vegetation, and mammal tissue samples were also taken in the four quadrants surrounding the plant location designated for air sampling. Samples were taken as close to the exact site of the air sampling as possible. The proximity of these samples should yield data suitable for associating levels of methylchloroform in air with those found in soils.

3. SAMPLING PROTOCOL

Air

Approximately 2 days were devoted to monitoring ambient air levels of methylchloroform in the vicinity of each producer and user plant. On the first day, measurements were made at several sites (usually 6 to 12) surrounding the plant to obtain a profile of the ambient chlorinated hydrocarbon concentrations and to identify any other emission sources in the vicinity. At least two grab sample measurements were made at each site over approximately a 1-hour period.

For subsequent monitoring, the sampling and analysis van was located at downwind sites and measurements were made over a 20 to 24-hour period to determine temporal and spatial variations and maximum concentration levels. When necessary, the van was moved to attempt to remain centered in the plant plume as well as possible. During this sampling period, grab samples of the ambient air were analyzed at approximately 15 to 30-minute intervals, the sampling rate being limited by the perchloroethylene retention time. Teflonbag grab samples integrated over a 15-minute collection period were taken at upwind and crosswind sites during the period in which the van was used for downwind measurements. During the 2-day monitoring period at each location, approximately 50 ambient air measurements were performed.

At each site, two ambient air samples were collected on Tenax traps for GC/MS confirmation of the field EC/GC measurement data. The samples were collected over a 1 to 2-hour period coincident with the field measurements.

Meteorological data were collected at each of the sites during the sampling. If a U.S. Weather Bureau Station was located nearby, data were obtained from their records. If not, a MRI Model 1071 portable weather station was set up near the site to make meteorological measurements. The parameters recorded on an hourly basis were wind speed and direction, temperature, barometric pressure, relative humidity, precipitation, and general weather conditions.

During the 2 days at each plant location, water, sediment, soil, and biota samples were taken while personnel in the air sampling van monitored the air for chlorinated hydrocarbons. In addition, a 24-hour composite effluent sample was obtained from plant personnel and samples were prepared for shipment to Battelle's Columbus Laboratories for analyses. Sampling of each medium is described below. The analyses of these samples are described

in subsequent sections, except for biota samples which were not analyzed during the course of this program.

Water

Samples taken by hand were collected approximately 2 to 5 cm below the surface of the water. Care was taken to avoid bubbling as the water entered the bottle. Samples taken with the Teflon-lined vertical sampler were usually taken as close to the surface as possible. In cases where the discharge was expected to stratify in the receiving stream, different depths in the water column were sampled at one location.

All water samples in the receiving stream were taken on the same day in as short a time frame as possible. The request made to the plant personnel for the 24-hour composite effluent sample was made for the day on which the sampling was conducted.

Sample bottles and the sampler were rinsed thoroughly in the water to be collected before the samples were taken. Samples were taken in clear glass bottles sealed with septa and crimped metal caps. At the sites sampled during the initial trip, 12 samples were taken at each sampling location. During the remainder of the program the sample size was reduced to 6 per location; 3 samples were held on wet ice and 3 at ambient temperature. At all locations, 2 additional water samples were taken in 1-ounce amber bottles and frozen on dry ice. Samples of the 24-hour composite and a tap-water sample were similarly prepared.

Sediment

Whenever possible, sediment samples were taken in the same locations at the same time as the water samples. Sediments were collected either by a dredge or by hand. Upon collection, the sediment surface was placed in the bottom of the sample jar. The volume collected approximated a 2-inch soil core. Two samples were collected in glass jars at each site. Sample jars were immediately wrapped in foil and placed in wet ice, then frozen as soon as possible, usually within 8 hours.

Soil

Six 2-inch soil cores were taken in each of the four quadrants around the plant. The corer was washed and rinsed with distilled water and acetone between each location and between each soil type at one location.

To dislodge the sample from the corer, the sampler was inverted over a glass jar. (Soil surface was on the bottom of the jar.) Samples were

immediately wrapped in foil, held on wet ice, and then frozen as soon as possible.

After the initial sampling trip, the sample number was reduced to two per location.

Vegetation

Six 1-ounce-volume vegetation samples were taken at each soil and air sampling site. The vegetation sampled was directly associated with (growing out of) the soil core taken. Samples were coded with subscripts to preserve this correlation. The samples comprised live, whole plants except in cases of large plants, where parts of several were clipped to provide a more representative sample. Samples were collected in amber bottles, placed on wet ice, then frozen as soon as possible. The sample size for any future collections will be reduced to two per location.

Tissue

In the case of both fishes and mammals, specimens were held on wet ice until dissection and/or sample preparation was completed. A minimum of 10 g of muscle tissue comprised each sample. Whenever possible, the tissue was provided by three specimens. For fish, flank muscles were taken; for mammals, muscle was stripped from each of the two hind legs. For both fish and mammals, whole livers were removed. Liver and muscle tissues from the same organisms were coded to preserve possible correlations. In the case of small organisms whose dissection would not provide sufficient sample size, whole bodies were taken.

Dissected tissues were placed in amber bottles; whole bodies were placed in clear glass jars and wrapped in foil. All tissue samples were frozen.

Sample size was dependent upon the availability of the organisms. Six specimens of each species was considered maximum.

Number of Samples

A breakdown of the numbers of samples collected from eight locations monitored November, 1976, through January, 1977, is given below:

Sample Type	Producer	<u>User</u>	Background
Air determinations	354	45	23
Waterclear glass	254	42	12
Wateramber glass	58	14	4
Sediment	38	4	2
Soil	112	8	2
Vegetation	112	8	2
Tissue	79	9	12

Methods were developed for the analyses of air, water, soil, and sediment samples; and these are described in the following section. However, no satisfactory method for the analyses of vegetation and tissue samples could be developed within the time limits of this program. The vegetation and tissue samples are stored in a frozen state for possible future analyses.

4. ANALYTICAL METHODS

Determination of Methylchloroform in Ambient Air

A method for measurement of methylchloroform in air has been developed and evaluated. The method involves direct injection of the ambient air into a gas chromatograph (GC) followed by detection of the emerging compounds with an electron-capture detecter (EC).

Equipment and Procedures—A schematic diagram of the system used for on-site field measurements of methylchloroform is shown in Figure 4.1. Ambient air is continuously drawn through a stainless steel line extending about 4.2 m above the ground and passed through a 5 cc loop attached to a Carle 6-port sampling valve. During sample injection, the carrier flow is diverted through the sampling loop for 15 seconds and the 5 cc air sample is swept onto the GC column. An electronic timer is used to control the injection period and automatically start the integrator at the end of sample injection. The integrator was used primarily to record retention times. The chromatograms obtained from the stripchart recorder were used to quantify the chlorinated hydrocarbon concentrations based on peak height.

Two EC/GC systems were used in the ambient air analysis program. Measurements at Dow, Ethyl Corporation, Vulcan Materials, PPG, and St. Francis National Forest (rural background) were performed with a Varian 1200 EC/GC system. A system using the more sensitive Analog Technology Corporation, Model 140A, EC detector was used for measurements at the Boeing Company plant. The operating conditions and performance characteristics of the two systems are given in Table 4.1.

Primary calibration of the EC/GC systems is discussed in the following section. Secondary calibrations in the field were performed with a standard TCE/nitrogen gas mixture. The sampling system was checked regularly for contamination by injection of the same gas (zero oxygen nitrogen) which was used as the carrier. Very slight, uniform residual background levels equivalent to about 0.1 ppbv of methylchloroform and perchloroethylene were obtained with the more sensitive ATC system. Ambient air measurements made with the system were corrected for these background considerations. Residual backgrounds from trichloroethylene and carbon tetrachloride were not detected.

The gas chromatograph system was operated in Battelle's Columbus Laboratories mobile sampling laboratory. The laboratory is equipped with a 7.6 kw gas-powered generator to provide power for sampling and analysis in any location accessible via a roadway.

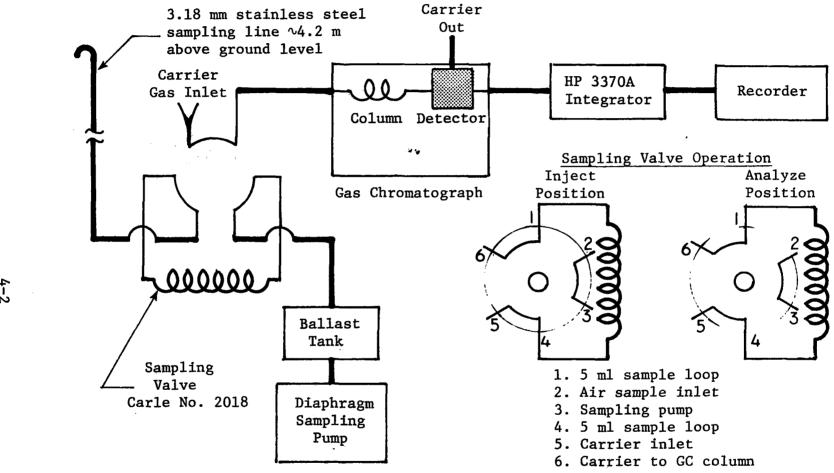


Figure 4.1. Schematic of EC/GC ambient air analysis system.

TABLE 4.1. OPERATING CONDITIONS AND PERFORMANCE CHARACTERISTICS OF THE EC/GC SYSTEMS USED FOR AMBIENT AIR MEASUREMENTS OF CHLORINATED HYDROCARBONS

	Varian 1200	ATC-140A
Column size/material	3.18 mm x 305 cm, stain- less steel	3.18 mm x 305 cm, stain- less steel
Column material	20% SP-2100/0.1% Carbo- wax-1500 on 100-120 mesh Supelcoport	20% SP-2100/0.1% Carbo- wax-1500 on 100-120 mesh Supelcoport
Column temperature	50 C, isothermal	55 C, isothermal
Carrier gas	Matheson nitrogen, oxygen free	Matheson nitrogen, oxygen free
Carrier flow	∿35 cc/min	37.5 cc/min
Detector	EC, tritiated titanium	EC, tritiated scandium
Detector temperature	150 C	240 C
Baseline adjustment	NA	275
Read-out	Honeywell 193 recorder, 1.27 cm/min	Honeywell 193 recorder, 1.27 cm/min
Air sample volume	5 cc	5 cc
Injection time	15 sec	15 sec
Typical Retention Times, sec Chloroform Methylchloroform Carbon tetrachloride Trichloroethylene Perchloroethylene	326 424 498 633 1632	240 308 365 462 1124
Relative Detector Respons at 50 ppb, TCE = 1.0 Methylchloroform Carbon tetrachloride Trichloroethylene Perchloroethylene	3.9 28.5 1.0 4.8	1.9 8.6 1.0 2.0
Estimated Minimum Detection Levels, ppbv Methylchloroform Carbon tetrachloride Trichloroethylene Perchloroethylene	0.3 0.05 1.0 0.3	0.02 <0.01 0.03 0.02

In addition to direct injection of ambient air, grab samples were also collected in Terlon bags for GC analysis. A stainless steel diaphragm pump powered by a portable gas-powered generator was used for sample collection. Analysis was performed with the Varian or ATC EC/GC systems by attaching the Teflon bag to the inlet of the sampling loop on the Carle valve.

Gas Chromatograph Calibration—The Varian 1200 EC/GC system was calibrated for measurement of methylchloroform, trichloroethylene, carbon tetrachloride, and perchloroethylene over the concentration range of 1 to 1000 ppbv. The ATC 140A system was calibrated for the four compounds over the concentration range of about 0.1 to 100 ppbv. The calibrations were performed by concurrently injecting the four compounds into the Battelle smog chamber to produce initial concentrations of either 100 or 1000 ppbv. Successive dilutions of the chamber air were made to produce a series lower, known concentrations to complete the calibration curve. The dilution factor for each dilution step was determined independently by following the decrease in concentration of methane injected into the chamber with the chlorinated hydrocarbons. A Beckman Model 109 hydrocarbon analyzer was used for the methane measurements.

In calibrating the Varian GC system, a Matheson 1200 ppbv TCE standard was compared with the chamber concentration of TCE to verify that an initial concentration of 1000 ppbv was obtained.

The calibration curves showing detector response for the Varian and ATC systems are shown in Figures 4.2 and 4.3, respectively. Each point on the calibration curves is the average of two determinations. Agreement between all duplicate determinations was within 5 percent. Both systems exhibit excellent linearity over the concentration range encountered in the field monitoring program.

Field calibrations were performed to verify detector response and retention times using a Matheson gas mixture of 1200 ppbv TCE in nitrogen. Calibrations were performed before, after, and at 6 to 8-hour intervals during the sampling program at each plant.

Determination of Methylchloroform in Water

The analytical method selected for development is based on sparging the methylchloroform from the water with an inert gas. These compounds are collected on a trap material and then desorbed onto a gas chromatography column for analysis.

If an inert gas is bubbled through water containing organic compounds which exhibit a low solubility in water, the compounds will be quantitatively partitioned into the gas phase. The enriched gas phase is then passed through a trap that retains the organics but allows the purge gas and most of the water to pass through. A large concentration factor of the volatile

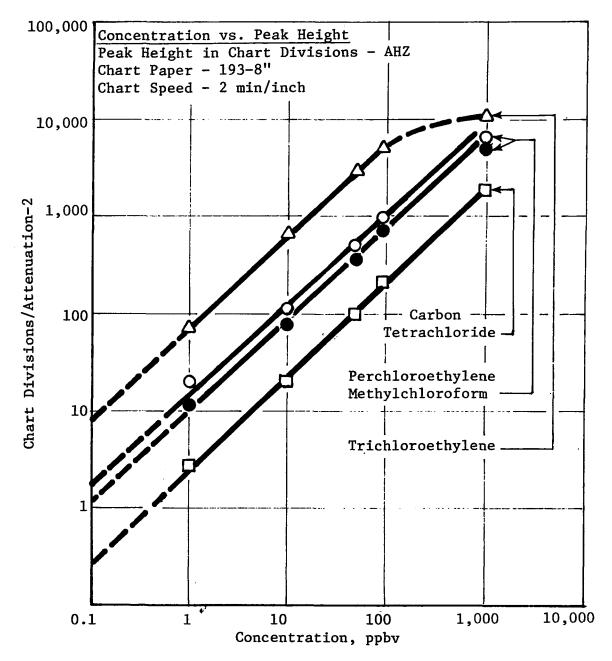


Figure 4.2. Calibration curves for chlorinated hydrocarbons--Varian 1200/EC Detector (Reference: Matheson 1.2 ppm TCE - 36.4 divisions - Attenuation-100).

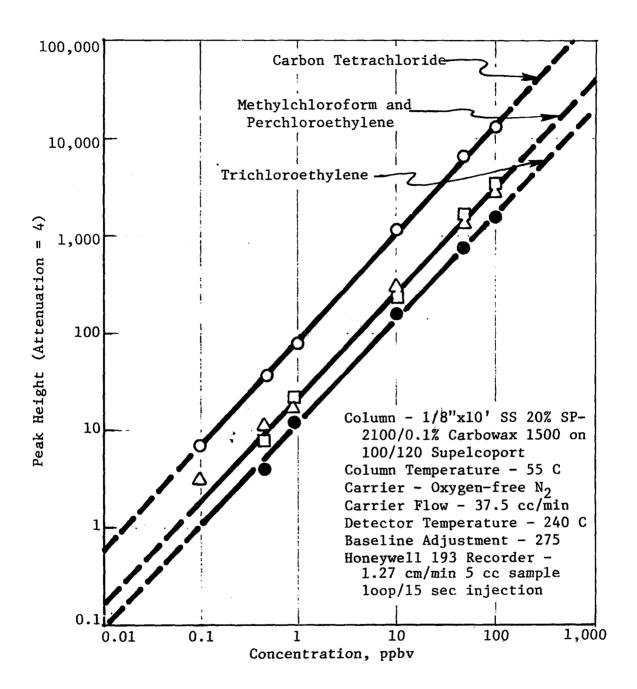


Figure 4.3. Calibration curves for chlorinated hydrocarbons--ATC 140A/EC detector.

organics out of the aqueous phase is accomplished. The trapped organics are then back-flushed onto a gas chromatography column or into a mass spectrometer for analysis.

Instrumentation—A Tekmar Liquid Sample Concentrator, Model LSC-1, was purchased and integrated with a Packard 1700 Series gas chromatography instrument, as shown in Figure 4.4, which shows the purge gas entering the flow meter, the purge-gas-rate control valve, and passing through the water sample sparging tube. The partitioned organics enter the gas phase and are deposited on the collection trap after passing through one path of the 6-way gas control valve. During the water purging cycle, the gas-chromatography carrier gas enters the 6-way control valve at the desorb gas "in" location and passes out of the valve onto the gas chromatography column.

The desorb mode is shown in the lower portion of Figure 4.4, with the 6-way control valve switched so the gas chromatography carrier gas backflushes the organics onto the gas chromatography column at the same time the trap is heated. The water sample purge gas valve is off during this period and another water sample can be sparged during the analysis of the first sample.

Initial evaluation of this system was not satisfactory because of broadening of the chromatographic peaks. The LSC-1 was modified to replace the resistance heater wrapped around the trap column with direct resistance heating of the stainless steel trap column. A step-down transformer, coupled with a Variac set at 50, heated the trap to 150 C in 8 seconds compared with the 3 minutes required for the original heater. Other modifications included replacing some Teflon lines with stainless steel and heating the transfer line from the LSC-1 to the gas-chromatography instrument.

Column Selection for the Gas Chromatograph—Several column materials were evaluated for the separation of chlorinated organic compounds. The material selected was 20 percent SP-2100 with 0.1 percent Carbowax-1500 on 100 to 120 mesh Supelcoport. Figure 4.5 shows a chromatogram from the vendor literature for 11 chlorinated solvents. The most likely interference with methylchloroform (1,1,1-trichloroethane), peak 5, is 1,2-dichloroethane, peak 4.

The SP-2100 is methyl silicone. The support material is diatomaceous earth which has been acid washed and silane treated. The addition of the 0.1 percent Carbowax is essential for high-quality chromatograms. A standard mixture containing methylchloroform, chloroform, carbon tetrachloride, trichloroethylene, and 1,2-dichloroethane showed the latter compound to be extremely insensitive to the electron-capture detector.

Quantitative Analysis Using the Flame Ionization Detector—The initial quantitative evaluation of this system was done in the 50 to 500—ppb range using a flame ionization detector. A 3-component standard containing methylchloroform, trichloroethylene, and perchloroethylene was prepared in water that had been demineralized and double distilled. A 500 ppb volume per volume in water at ambient temperature and a 50 ppb standard were prepared.

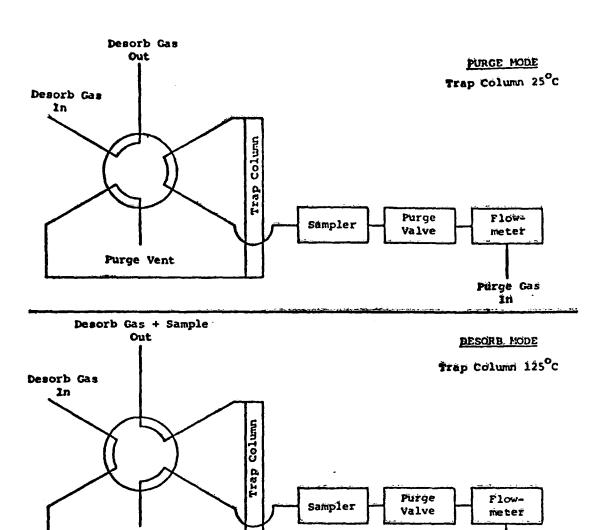


Figure 4.4. Schematic of a liquid sample concentrator.

Purge Gas

Purge Vent

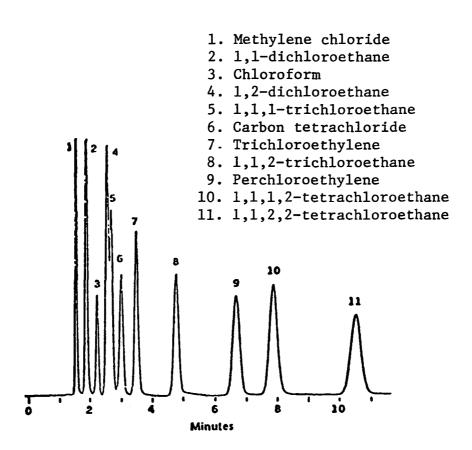


Figure 4.5. Chromatogram of chlorinated solvents.

Instrumental conditions used for analysis of this standard were as follows:

Liquid Sample Concentrator

Sample size - 5 cc standard water solution Purge gas rate - nitrogen at 40 cc/min for 10 min Degas temperature - 150 C reached in 10 sec Trap material - Tenax plus silica gel

Gas Chromatography Conditions

Column - 10 ft x 1/8-in stainless steel packed
with 20 percent SP 2100/0.1% Carbowax
1500 on 100 to 120-mesh Supelcoport

Temperatures - Column - 55 C
Detector - 150 C
Injection - 140 C

Gas flows - Nitrogen carrier - 30 ml/min
Hydrogen - 30 ml/min
Air - 300 ml/min

Electrometer - 1 x 10⁻¹⁰ amp, 500 volts.

The recording system was a HP 3380A integrator which prints retention time directly above each peak, and then records the total counts for each peak, based on the total area under the peak. The area percentage for each peak recorded in the right-hand column is not significant in this work because it is based on percentage of total area and we do not know the sensitivity factor or the identification of other peaks appearing in the chromatograms.

A reproduction of the actual chromatograms is shown in Figures 4.6 and 4.7. Both chromatograms show some impurities which are caused by the dilution water and/or impurities in the compounds added to the water. The methylchloroform has a retention time of 3.44 to 3.36 minutes and the trichloroethylene 4.99 and 5.01 minutes for Figures 4.6 and 4.7, respectively. The integrator counts representing the area under these peaks were as follows:

Compound	500 ppb	50 ppb
Methylchloroform	624,861	62,092
Trichloroethylene	1,121,598	122,636

The linearity from 50 to 500 ppb using the flame ionization detector is excellent for these two compounds. The degas temperature for the 50 ppb chromatogram did not reach 150 C and this probably explains why the perchloroethylene was not linear. The x values directly above the retention-time values are the attenuation factors used to keep the peaks on scale. Since both compounds were attenuated times 8 and a 5 ml sample was used, it would appear that a sensitivity of about 500 ppt could be reached with a 20-ml sample using the FID detector, provided purer water is used for the standards. The 50 ppb methylchloroform and trichloroethylene represents

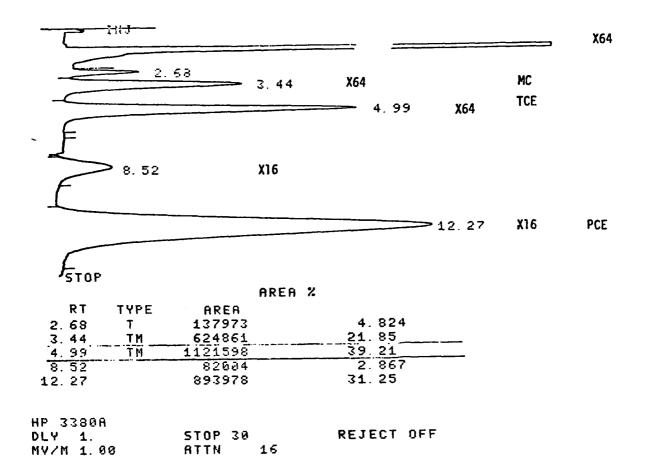


Figure 4.6. Chromatogram showing 500 ppb each of methylchloroform (3.44 minutes), trichloroethylene (4.99 minutes), and perchloroethylene (12.27 minutes) using FID detector.

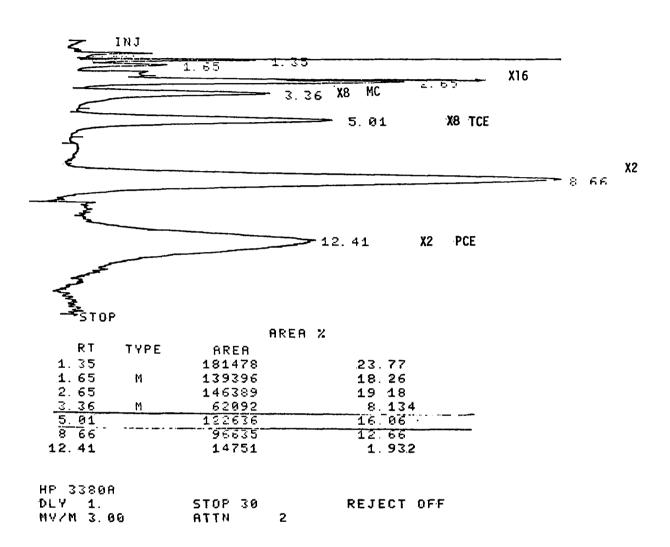


Figure 4.7. Chromatogram showing 50 ppb each of methylchloroform (3.36 minutes), trichloroethylene (5.01) minutes), and perchloroethylene (12.41 minutes) using FID detector.

 50×10^{-9} ml/ml of water or 68 and 73 ng/ml of water, respectively. Figure 4.8 shows the FID calibration curve.

Quantitative Analysis Using the Electron Capture Detector—The instrumental settings for the liquid-sample concentrator and the gas chromatography were the same as described above except for the detector power source and the air and hydrogen required for the operation of the flame. The electron-capture-detector electrometer settings were 1 x 10^{-10} A and 25 V.

Preparation of standards required special treatment of the dilution water. All distilled and demineralized water supplies checked contained methylchloroform and trichloroethylene, including a demineralized and double-distilled supply. Untreated well water was lowest in these compounds but had a high iron content; therefore, the well water was not used for dilutions. The integrator counts representing total impurities in the various waters checked are shown in Table 4.2. The Ohio State University demineralized and double-distilled water was sparged with nitrogen while being boiled for 3 hours, which reduced the methylchloroform and trichloroethylene to undetectable amounts; therefore, this water was used to prepare standards.

TABLE 4.2. VOLATILE IMPURITIES IN WATERS

Source of Water	Impurity Counts
City Products Corp., storeroom supply Tap water, City of Columbus, Ohio Biology Department, glass still Deionized water, analytical section Ohio State University, double distilled Ohio State University, sparged with nitrogen Olentangy River, 5th Ave. and King Ave. north of dam Ohio State University, boiled and sparged with N2 Well water, untreated, Fairfield County	10,160,000 8,910,000 5,792,000 810,000 317,000 217,000 211,000 15,825 3,203a

All values were determined by concentrating 5 ml of water except this well water; 20 ml was concentrated with a total impurity count of 12,812; therefore, one-fourth this amount was reported above.

Standards containing methylchloroform and trichloroethylene at a concentration of 500, 100, and 50 ppt were prepared in the special dilution water using the electron-capture detector. The calibration curves for these

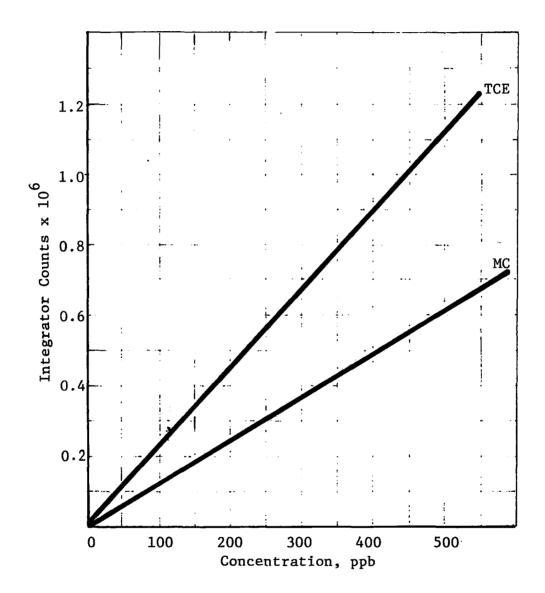


Figure 4.8. Calibration curves for the determination of trichloroethylene and methylchloroform using the FID detector.

runs are shown in Figure 4.9. The sensitivity for methylchloroform is much greater than that for trichloroethylene, which is reversed with the flame ionization detector. The 500 and 100 ppt standards were analyzed by sparging 5 ml of the standard, and the 50 ppt standard was analyzed using 20 ml. The 50 ppt concentration is equivalent to 67.5 pg/ml of water for methylchloroform and 73.3 pg/ml for trichloroethylene.

These calibration curves were not used in the analysis of samples since the HP 3380A integrator is a dedicated computer which retains data input on a standard sample and the amount of a compound per area can be listed for each standard peak in the chromatogram. If the amount per area shows a sudden change under the same operating conditions, this would indicate operation beyond the linearity range of the detector, a poor standard or that some other instrument trouble exists. A sample run is automatically computed from the standardization data retained by the computer. A multiplication factor to adjust for sample size or amount of dilution can be added to the stored data at the time each sample is injected. electron-capture detector was used for all sample analyses with a maximum standard concentration of 50 ppb. If the first run on a sample indicated that the concentration was much higher than 50 ppb, the sample was diluted to bring it into range or minor adjustment was made by reducing the quantity of sample. A 5-ppb standard was generally used for low concentrations and up to 20 ml of sample.

The initial objective was to obtain chlorinated hydrocarbons through perchloroethylene without use of our program temperature facilities; however, as shown in Figure 4.6, the resolution would not be adequate to elute perchloroethylene in 13 minutes with methylchloroform, carbon tetrachloride, and other possible impurities in industrial waters. The flow and temperature were reduced slightly to provide a retention time of about 13 minutes for trichloroethylene as shown in Figure 4.10. The relative concentrations of carbon tetrachloride in the samples were not as high as shown in Figure 4.10; therefore, many samples were analyzed with the trichloroethylene retention time set at 10 minutes.

Preparation of Standards—Standards are prepared from the specially prepared water described earlier. A 1-liter volumetric flask is filled with the special water and a hypodermic syringe is used to inject a known quantity of the compounds. The flasks are placed on a shaker for 16 hours (overnight) and this forms the base standards which are diluted to lower concentrations. Base standards containing 5 μ g/ ℓ of water (5 ppm) and 2 μ g/ ℓ of water have been rediluted and analyzed on the electron-capture detector. Good agreement was obtained that would indicate that the measurements of these small quantities were reproducible and also that these quantities were completely soluble in 1 liter of water. The base standards and diluted standards were protected from light at all times. The base standards were used for 2 to 3 weeks before any concentration deterioration was noted, but the diluted standards were made fresh daily.

<u>Precision and Accuracy</u>—The precision of the method was tested using 10 determinations of a standard containing 50 ppb by volume of both

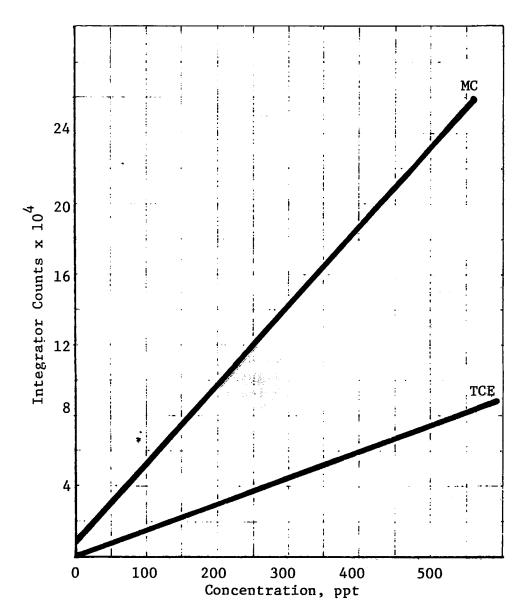


Figure 4.9. Calibration curves for the determination of trichloroethylene and methylchloroform using the electron capture detector.

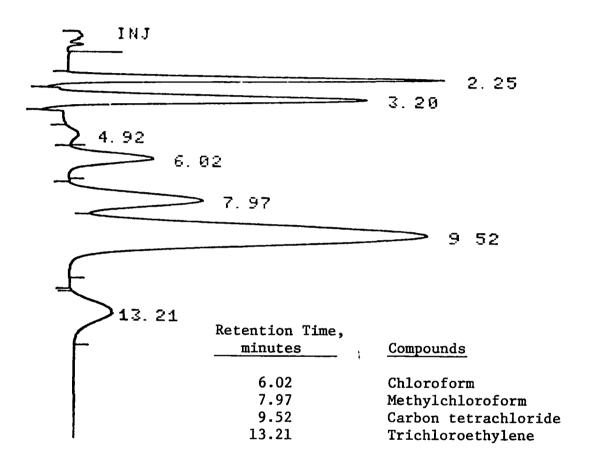


Figure 4.10. Chromatogram of several chlorinated hydrocarbons using the electron-capture detector after completion of development work.

methylchloroform and trichloroethylene. The following precision data were obtained:

	Methylchloroform	Trichloroethylene
Average	49.75	50.29
Sigma (σ)	1.65	1.68
Coefficient of variation	3.3	3.4

Five of the above analyses were made on different standards and the other five on the same standard. Two different operators were involved.

Since no primary standards exist for this type work or no crosslaboratory analyses among several laboratories have been performed to our knowledge, the absolute accuracy is not known.

Water Sample Data to be Presented--In addition to the concentration of trichloroethylene and methylchloroform in each sample, the following information is given:

- (1) Date sampled
- (2) Data analyzed
- (3) Amount of sediment in the sample
- (4) Sparging characteristics
- (5) Rough quantitative values for chloroform and carbon tetrachloride
- (6) Comments which indicate which samples are composites, tap waters, or required unusually high sample dilutions, and other miscellaneous remarks.

The sediment in the samples was classified as C=clear, L=light, M=medium, and H=heavy. The sediment concentrations were judged before shaking the samples prior to analysis. If no particles had settled on the bottom, they were classified as clear; any observable particles on the bottom were noted as light; if the bottom was nearly covered, it was classified as medium; and if the bottom was entirely covered, this was considered heavy. The samples classified as heavy contained only a very thin coating on the bottom. Some of these sediments appeared to be a gelatin-like substance.

The column headed "sparging foam" indicates the degree of foam generated while sparging the compounds from the water samples. These are designated as ND=none detected, L=light, M=medium, and H=heavy. Blank areas indicate that we made no observation. None of the samples produced

sufficient foam to cause trouble with carry-over to the collection trap; however, many of the samples were diluted before analysis, which would reduce foaming.

The results reported for chloroform and carbon tetrachloride were obtained by analyzing a standard mixture containing these two compounds plus the methylchloroform and trichloroethylene. Sensitivity ratios were calculated based on trichloroethylene as 1 and this permitted a rough quantitative estimation of these compounds.

Determination of Methylchloroform in Soil and Sediment

Methylchloroform is expected to be present in soil and sediment samples at levels of the order of 10^{-1} to 10^3 ppb by weight. The analysis technique must, therefore, be capable of detecting 10^{-1} to 10^{-2} ng of each substance in reasonably sized samples of 0.1 to 1.0 g. Furthermore, a high level of specificity is required to avoid interferences from the many other organic substances commonly present in soil and sediment samples.

Electron-capture gas chromatography (EC/GC) is ideally suited to detection of these volatile chlorinated hydrocarbons because of its very specific response to electrophilic substances at the required concentration levels. However, before EC/GC can be applied to such samples, the trichloroethylene and methylchloroform must be extracted to a phase suitable for injection into the chromatograph. Either gaseous or liquid samples can be handled by the chromatograph. The three methods used for these types of samples therefore involve a preliminary conversion of the methylchloroform sorbates to either gaseous or solution forms.

<u>Extraction Methods</u>--Basically three different methods for methyl-chloroform extraction have been considered:

- (1) Thermal desorption—A sample of soil is heated while being purged by a stream of nitrogen. The eluted methylchloroform is trapped on Tenax or other suitable sorbents and then injected into the chromatograph by flash heating of the trap.
- (2) Liquid extraction—The methylchloroform is solvent extracted using acetone and/or hexane. The resulting solution can then be injected directly into the chromatograph.
- (3) Aqueous sparging—Inasmuch as methylchloroform has low solubility in water, this substance can be used to disperse soil and sediment samples to render them susceptible to purging by nitrogen. The effluent methylchloroform is then handled much the same as with the thermal desorption method.

Method (1) has been shown to be useful for analysis of trichloroethylene and certain other chlorinated hydrocarbons in dry or only slightly wet samples. However, the excessive amounts of water likely to be present with sediment samples render this approach difficult at best. Furthermore, it has been shown that certain chlorinated hydrocarbons, such as chloroform and methylchloroform, are not recovered efficiently by this method. Indeed, results with some model soils suggest that methylchloroform is chemisorbed and can be recovered only as vinylidene chloride by this method.

Method (2) is efficient and satisfactory providing care is taken to minimize sample losses during the extraction and subsequent concentration steps of the procedure. (If aliquots of solution are analyzed, the sensitivity of the method is reduced.)

Method (3) also suffers from poor recovery of methylchloroform that is chemisorbed in the soil surface. However, this procedure more closely imitates the probable mechanism for mobilization of methylchloroform and trichloroethylene in the environment. Furthermore, Method (3) is an "online" procedure with little or no chance for either losses or gains of methylchloroform and trichloroethylene due to exposure of the sample to laboratory air. The method is equally applicable to wet and dry samples. The results of its application reflect the availability of methylchloroform and trichloroethylene to the environment rather than total methylchloroform and trichloroethylene exposure.

Apparatus--A schematic representation of the apparatus used for sparging of soil samples is shown in Figure 4.11. In use, presparged water (3 to 4 cc) is loaded into the fritted glass vessel, the soil sample injector is mounted, and the sparger is attached to the sample trap valve. The sample side of the system is then flushed with zero nitrogen until a suitable blank reading for methylchloroform and trichloroethylene is obtained. Usually, this is possible within about 10 minutes or less; but flushing is continued for approximately the 30 minutes required for the blank analysis. sample is then injected into the water and the effluent trichloroethylene and methylchloroform are trapped on Tenax maintained at room temperature. During sparging of the soil-water mixture, the sample is agitated by immersion in an ultrasonic bath. This serves to rapidly disperse the soil and facilitate sparging. Sparging periods of 10 minutes at 30 to 40 cc/min are sufficient. Following the sparging period, the by-pass around the sparger is opened to permit flushing of the water vapor from the Tenax trap. A flushing period of 5 to 6 minutes is sufficient to remove the water vapor without removing trichloroethylene or methylchloroform from the Tenax. trap valve is then switched to permit flushing by the zero-nitrogen GC carrier gas and the trap is heated rapidly (at ∿500 C/min) to 190 C to inject the methylchloroform and trichloroethylene into the chromatograph.

The soil or sediment injector is constructed as a syringe-like device with its open end capped by a tight-fitting Teflon plug. The injector is weighed and then used to core the analysis sample directly from the bulk as-received sample. Reweighing and capping of the injector are done rapidly to minimize contact with the laboratory air. The injector is then kept closed

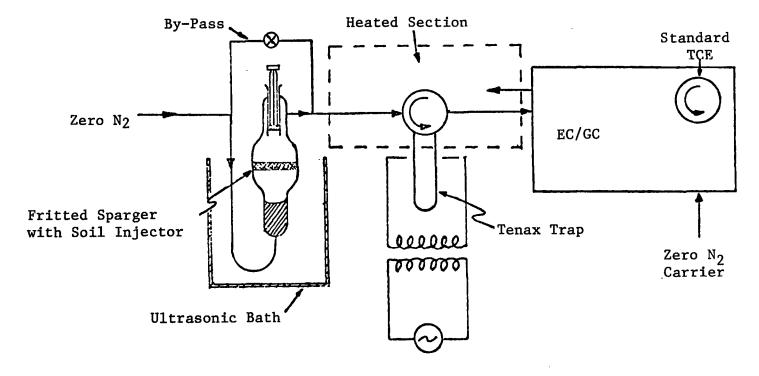


Figure 4.11. Schematic of soil and sediment analysis apparatus.

until injection of the soil into the sparger, at which time both the soil and Teflon plug are manually ejected.

The chromatograph is a Varian 1200 equipped with a $Ti(H^3)$ detector. The column is a 1/8-inch by 10-foot stainless steel column packed with SP-2100 (GP 20 percent SP-2100/1 percent Carbowax-1500 on 100-120 mesh Supelcoport). Output signals are quantified using an Infotronics Model CRS 204 integrator coupled to a TTY output.

Standardization is accomplished using a precalibrated gas standard of TCE in nitrogen. Approximately 4-ng samples of trichloroethylene are usually used for standardization. Such samples yield peak areas on the order of $10^4~\mu v/sec$, and peaks on the order of $10^2~to~10^3~\mu v/sec$ can be separated from the inherent background noise. Comparative calibrations with methylchloroform and trichloroethylene indicate a relative response of 3.36 for MC/TCE at equal concentrations. The response curve of the detector is not perfectly linear but rather varies with $C^{1.05}$ in the concentration range of interest. A sample chromatogram showing response obtained with one sediment sample is shown in Figure 4.12.

Quality of Results--There are several points that must be recognized in discussion of the significance of the results of trichloroethylene and methylchloroform analyses on soil and sediment samples. Ideally, standardization should be performed using well-characterized standards of trichloroethylene and methylchloroform on substrates that closely simulate those of subject samples, and these standards should be traceable to primary standards established by independently certified means. Such standards are not available for methylchloroform and trichloroethylene in soils and sediments, nor is it possible to reliably prepare such standards because of the inherent instability of this type of specimen. Because of the general stability of trichloroethylene in a nonoxidizing atmosphere, we have chosen to use trichloroethylene in nitrogen as the reference standard for the current work. Analysis of the standard was made by the manufacturer and has been crosschecked with samples of the same concentration prepared by injection of liquid samples into the Battelle smog chamber. The standard being used appears to be accurate to within a few percent.

A second infringement on the quality of the results is related to the basic heterogeneity of the samples. Any soil sample is likely to be a composite of various organic and inorganic structures, e.g., sand particles, clays, organic residues, plant fragments, etc. Such local heterogeneity is likely to be reflected in appreciable local gradients in the distribution of methylchloroform and trichloroethylene. These local-gradient tendencies are likely to be superimposed on the natural vertical and horizontal gradients that are caused by temporal and spatial variation in the flux of methylchloroform or trichloroethylene to a given sample area. Because of the limited size of the analytical sample, the results must therefore be considered as point analyses rather than as representative analyses. This situation is magnified further with the sediment samples. With sediments, the fraction of the sample that is present as a liquid phase is much larger than with the soil samples. Results can vary considerably thereby reflecting the partitioning of methylchloroform and trichloroethylene between the solid and liquid phases.

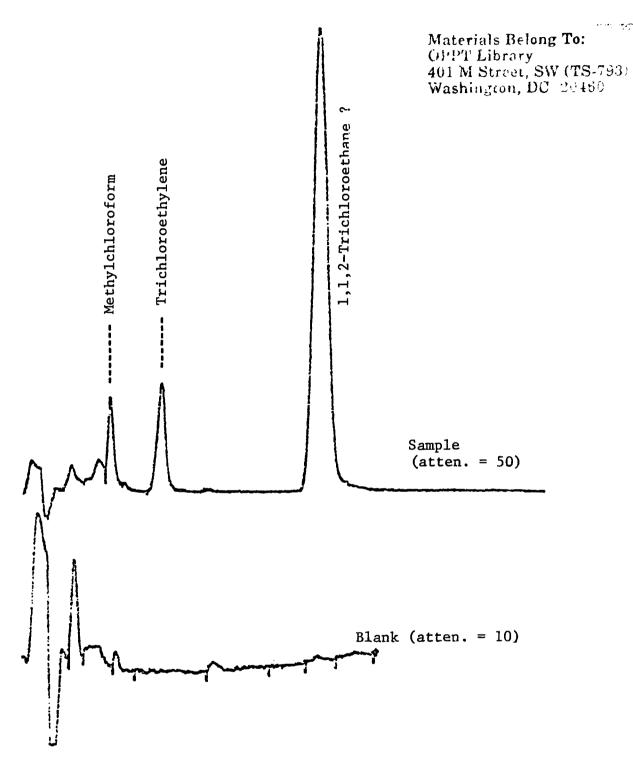


Figure 4.12. Sample chromatogram for sediment.

5. MONITORING DATA

The sampling rationale, sampling protocol, and analytical methods have been described. The results are presented as a series of maps and tables which describe the locations, the nature of the samples, and the concentrations of methylchloroform in the samples. A separate set of data is presented for each site.

Production Sites Monitored

For each methylchloroform production site a map is presented with sampling points indicated (Figures 5.1 to 5.4). The results from the analysis of the samples and detailed descriptions of the sampling locations are presented (Tables 5.1 to 5.12).

User Site Monitored

The data obtained at a methylchloroform user site are presented in Figure 5.5 and Tables 5.13 to 5.15.

Background Site Monitored

The data obtained at St. Francis National Forest near Helena, Arkansas, are presented in Tables 5.16 to 5.18. This site represents a rural background site and is removed from known sources of methylchloroform and major industrial activity (see Figure 5.6).

Discussion of Results

The ambient air concentration profiles around all facilities monitored are characterized by increased concentrations of methylchloroform in the downwind direction from the source. Upwind measurements, which showed significantly lower methylchloroform concentrations, do not give any evidence of other methylchloroform sources which would contribute to the observed downwind levels.

Considerable variation was observed in the maximum downwind levels of methylchloroform at the various production plants. Maximum concentrations ranged from 12 to 155 ppbv at the methylchloroform production facilities. The variations in the observed maximum concentrations among plants may be due to differences in (1) production processes, (2) emission control equipment, (3) meteorological conditions, and (4) distance from plant. Higher production capacity apparently does not necessarily imply higher emissions since the maximum concentrations observed at the larger plants were lower than those observed at the smaller operations.

Very large temporal variations were generally observed at a given site downwind from the methylchloroform production facilities. Changes in meteorological conditions (wind speed and direction) and/or variations in the process emissions may account for this phenomenon. Less temporal variation was noted in the ambient air concentrations downwind from the methylchloroform solvent cleaning facility. Due to the nature of solvent cleaning operations, more uniform emission rates might be expected. In addition, meterological conditions (essentially no wind) during much of the sampling period reduced dispersion of the plume.

Duplicate analyses on some of the soil samples suggest that sample heterogeneity may contribute 30 to 50 percent deviation in the reported values for individual analyses. It is interesting to note that both soil and sediment samples from the background site display methylchloroform content equal to or greater than many of the plant-site samples. However, if it is assumed that a true average background level can be obtained by averaging all results equal to or less than the background-site levels, there still remains a number of samples containing significantly more methylchloroform than the background.

The highest levels of methylchloroform are generally associated with sediment samples. Chromatograms of these high-concentration-level samples also show the presence of appreciable quantities of other EC-sensitive compounds. For example, the same chromatogram shown in Figure 4.12 indicates the presence of other chlorinated hydrocarbons including a relatively large amount of what is believed to be 1,1,2-trichloroethane. A peak believed to be due to perchloroethylene was present in some chromatograms and small amounts of carbon tetrachloride were detected in some of the samples. Other peaks were sometimes present but none of these interfered with the methylchloroform analyses.

Although the results for the sediment samples generally reflect average concentrations of methylchloroform present in both the liquid and solid phases of the samples, with two samples, F-1-S and F-3-S, an attempt was made to distinguish between the two phases. In these experiments, a portion of each sample was centrifuged to permit sampling of the liquid phase. Analyses of the resulting water samples differed considerably from the bulk sample analyses. In both cases, the methylchloroform concentration was approximately the same as in the bulk sample.

However, the trichloroethylene content of the water was nearly an order of magnitude smaller in the water than in the bulk sample. While these experiments are not conclusive, they suggest that the trichloroethylene was truly associated with the solid phase and that the observed methylchloroform was primarily associated with the liquid phase. In other words, if there is any methylchloroform absorbed on the solid phase of these samples, it is present in a form that is not readily mobilized.

MONITORING DATA

PRODUCTION SITE 1

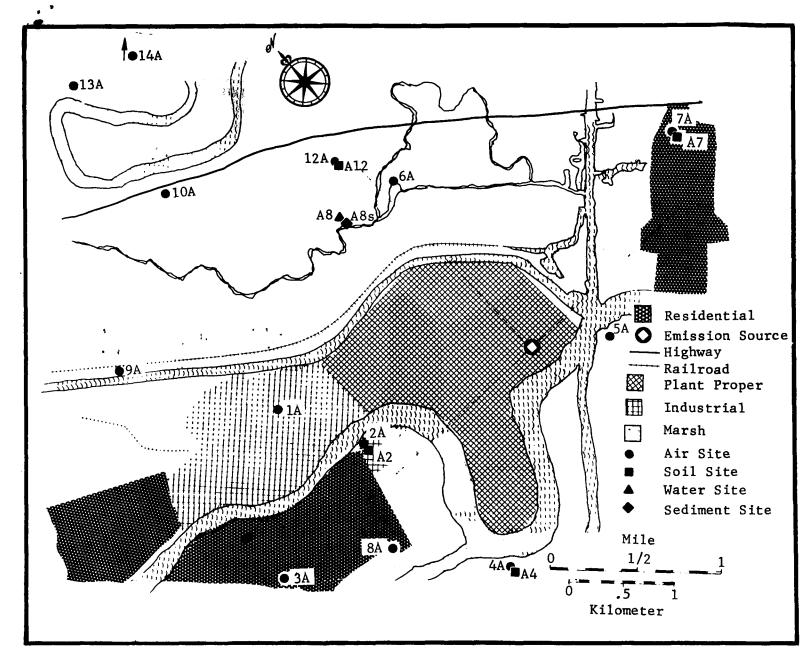


Figure 5.1. Sampling locations at Dow Chemical Plant A, Freeport, Texas--methylchloroform production site.

TABLE 5.1 AMBIENT AIR MEASUREMENTS AT DOW CHEMICAL PLANT A (METHYLCHLOROFORM PRODUCER)

	Distance	Direction								Meteoro1	ogical 0	bserva	tionsc
	from	from				entrat		_	Wind	Wind	Temper-		
Sit	e Plant,	Plant,	Date		Ambie	nt Air	, ppb	vb	Speed,	Direction,	ature,	RH,	Barometer,
No.	km	degreesa	1976	Time	MC	CC14	TCE	PCE	m/s	degreesa	C	%	mm Hg
1A	2.6	300	11/8	0940	2.3	0.30	≤1	NDd	5-10	080	17	$_{ m ND}$ d	772
				1020	≤0.3	0.25	≤1	ND	5-10	080	17	ND	772
2A	1.9	285	11/8	1055	≤0.3	1.4	≤1	ND	5-9	080	18	ND	772
				1110	1.1	2.1	≤1	ND	5-9	080	18	ND	772
			11/9	1536	0.7	0.15	≤1	ND	4-7	180	24	ND	764
			11/10	1346	0.5	0.15	≤1	ND	6-7	155	24	ND	761
3A	3.2	265	11/8	1140	1.2	0.60	≤1	ND	5-9	080	19	ND	771
				1155	≤0.3	1.1	≤1	ND	5-9	080	19	ND	771
4A	2.1	225	11/8	1330	≤0.3	0.15	≤1	ND	4-7	080	21	ND	770
				1345	≤0.3	0.15	≤1	ND	4-7	080	21	ND	770
5A	0.8	120	11/8	1420	≤0.3	Ø.15	≤1	ND	4-7	080	21	ND	769
				1433	≤0.3	0.15	≤1	ND	4-7	080	21	ND	769
			11/9	1614	0.6	0.15	≤1	ND	4-7	-180	24	ND	764
			11/10	1420	0.7	0.14	≤1	ND	6-7	155	24	ND	761
6A	1.9	010	11/8	1610	≤0.3	0.14	≤1	ND	3-7	065	21	ND	769
			•	1625	≤0.3	0.15	. ≤1	ND	3-7	065	21	ND	769
7A	2.6	080	11/8	1523	≤0.3	0.15	≤1	ND	4-7	080	21	ND	769
			·	1535	≤0.3	0.16	≤1	ND	4-7	080	21	ND	769
			11/9	1450	1.1	0.14	≤1	ND	5-8	185	24	ND	764
				1746	0.8	0.14	≤1	ND	2-4	165	23	ND	763
1			11/10	1309	0.7	0.13	≤1	ND	5	155	23	ND	761
A8	2.6	260	11/8	1712	≤0.3	0.19	≤1	ND	3-9	075	20	ND	769
				1738	0.8	0.24	≤1	ND	3-9	075	20	ND	769
				1745	≤0.3	0.18	≤1	ND	3-9	075	20	ND	769
				1758	2.2	0.58	≤1	ND	4-5	070	19	ND	769
				1812	0.8	0.38	≤1	ND	4-5	070	19	ND	769 ≈
				1825	0.4	0.22	≤1	ND	4-5	070	19	ND	769
10.	A 3.2	350	11/9	0924	≤ 0.3	0.22	≤1	ND	2-4	150	21	ND	767
			•	0940	≤0.3	0.17	≤1	ND	2-4	150	21	ND	767
				1015	≤0.3	0.14	≤ 1	ND	2-4	150	21	ND	767
	_			1030	≤0.3	0.54	≤1	ND	2-4	150	21	ND	767

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TABLE 5.1. (Continued)

	Distance	Direction									ogical O	bserva	tionsc
	from	from				entrat			Wind	Wind	Temper-		
Site	Plant,	Plant,	Date		Ambie	nt Air	, ppb	vp	Speed,	Direction,	ature,	RH,	Barometer,
No.	km	degrees ^a	1976	Time	MC	CC1 ₄	TCE	PCE	m/s	degrees ^a	С	% 	mm Hg
10A	3.2	350	11/9	1900	≤0.3	0.25	≤1	ND	3–5	165	22	ND	763
(Con	t)			1915	≤0.3	0.22	≤1.	ND	3-5	165	22	ND	763
				2200	≤0.3	0.22	≤1	ND	3-4	170	21	ND	763
			11/10	1430	≤0.3		≤1	ND	6	150	23	ND	761
				1445	≤0.3	0.15	≤1	ND	6	150	23	ND	761
				1500	≤0.3	0.15	≤1	ND	6	160	23	ND	761
.2A	2.6	005	11/9	1155	7.6	0.27	≤1	ND	4-6	180	23	ND	765
•				1210	6.2	0.48	≤1	ND	4-6	180	23	ND	765
				1220	5.8	0.27	≤1	ND	4-6	180	23	ND	765
				1235	4.2	0.21	≤1	ND	3-6	185	24	ND	764
				1250	9.4	0.58	≤1	ND	3-6	185	24	ND	764
				1305	0.9	0.16	≤1	ND	3-6	185	24	ND	764
				1320	4.2	0.50	≤1	ND	3-6	185	24	ND	764
				1335	1.5	0.18	≤1	ND	4-7	180	24	ND	764
				1350		0.48	≤1	ND	4-7	180	24	ND	764
				1405	1.4	0.23	≤1	ND	4-7	180	24	ND	764
				1420	2.2	0.28	≤1	ND	4-7	180	24	ND	764
				1450	2.4	0.31	≤1	ND	5-8	185	24	ND	764
				1505	≤0.3	0.26	≤1	ND	5-8	185	24	ND	764
				1630	≤0.3	0.16	≤1	ND	3-5	175	24	ND	763
				1645	0.7	0.27	≤1	ND	3-5	175	24	ND	763
				1755	≤0.3	0.15	≤1	ND	2-4	165	23	ND	763
				2300	7.2	0.94	≤1	ND	2-3	170	21	ND	763
				2315	6.5	2.4	≤1	ND	2-3	170	21	ND	763
				2330	9.8	3.3	≤1	ND	2-3	170	21	ND	763
				2345	3.0	1.2	≤1	ND	2-3	185	21	ND	763
				2400	9.8	3.8	≤1	ND	2-3	185	21	ND	763
			11/10	0020	2.2	0.48	≤1	ND	2-3	185	21	ND	763
				0035	11.5	1.8	≤1	ND	1-2	170	21	ND	763
				0050	6.5	1.3	≤1	ND	1-2	170	21	ND	763
				0105	7.6	3.4	≤1	ND	1-2	170	21	ND	763

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TABLE 5.1. (Continued)

	Distance	Direction									ogical 0	bserva	tions ^c
	from	from				entrat			Wind	Wind	Temper-		
Site	Plant,	Plant,	Date		_Ambie	nt Air	, ppb	vp	Speed,	Direction,	ature,	RH,	Barometer
No.	km	degrees ^a	1976	Time	MC	CC1 ₄	TCE	PCE	m/s	degrees ^a	C	<u></u> %	mm Hg
12A	2.6	005	11/10	0120	1.1	1.2	≤1	ND	1-2	170	21	ND	763
			•	0135	≤0.3	0.33	≤1	ND	2	160	21	ND	763
				0150	≤0.3	0.58	≤1	ND	2	160	21	ND	763
				0210	≤0.3	0.84	≤1	ND	2	160	21	ND	763
				0225	≤0.3	0.18	≤1	ND	2	160	21	ND	763
				0245	1.4	0.84	≤1	ND	2	155	21	ND	763
				0315	≤0.3	0.14	≤ <u>1</u>	ND	2	155	21	ND	763 `
				0345	2.2	0.14	≤1	ND	2	· 170	21	ND	762
				0415	2.6	0.14	≤1	ND	2	170	21	ND	762
				0445	1.1	0.10	≤1	ND	2-3	155	21	ND	762
				0515	1.8	0.14	≤1	ND	2-3	155	21	ND	762
				0545	0.7	0.14	≤1	ND	2	140	21	ND	762
				0615	≤0.3	0.10	≤1	ND	2	140	21	ND	762
				0645					2-3	120			
				0715					2-3	120			
				0745					2-3	110			
				0815					2-3	110			
				0845					4-5	155			
				0915					4-5	155			
				0945	≤0.3	0.14	≤1	ND	2-5	155	22	ND	763
				1015	≤0.3	0.14	≤1	ND	2-5	155	22	ND	763
				1045	≤0.3	0.18	≤1	ND	4-6	165	23	ND	763
				1115	≤0.3	0.14	≤1	ND	4-6	165	23	ND	763
				1145	≤0.3	0.14	≤1	ND	4	165	23	ND	763
				1215	≤0.3	0.14	≤1	ND	4	165	23	ND	762
				1245	≤0.3	0.14	≤1	ND	4	165	23	ND	762
13A	5.1	345	11/10		≤0.3	0.11	≤ <u>1</u>	ND	2-5	155	22	ND	761
			•	1645	0.5	0.10	≤1	ND	2-5	155	22	ND	761
14A	7.8	355	11/10		≤0.3	0.13	≤ <u>1</u>	ND	2-5	155	22	ND	761
			•	1715	_≤0.3	0.15	≤1	ND	2-5	155	22	ND	761

FOOTNOTES FOR TABLE 5.1

aNorth - 360°.

bTo convert to $\mu g/m^3$ at 25 C multiply ppbv by MC 5.46 CCl₄ -- 6.29 TCE -- 5.37.

^CGeneral weather conditions:

11/18/76 Clear, sunny, no precipitation 11/9/76 Clear, sunny, no precipitation 11/10/76 Slightly cloudy in morning, clearing in afternoon, no precipitation.

d_{ND} = not determined.

TABLE 5.2. ANALYSIS OF WATER, SOIL, AND SEDIMENT SAMPLES FROM DOW CHEMICAL PLANT A (METHYLCHLOROFORM PRODUCER)^a

		ppb by			Spargin	Sediment		Date	Date	ample
Comment	CC1 ₄	CHC1 ₃	TCE	MC	Foam	in Sample		Analyzed	ampled	No. S
				<u>er</u>	Wat					
Surface	116	82	126	117	Light	Clear		11/23/76	1/9/76	A-1 1
Bottom	32	25	122	119	Light	Light		11/28/76	1/9/76	A-2 1
Surface	5	1	5	0.8	Light	Heavy		11/23/76	1/9/76	A-5 1
Bottom	7	3	13	1	Light	Heavy		11/23/76	1/9/76	A-6 1
Surface	0.3	1	0.9	0.1	ND	Heavy		11/17/76	1/9/76	A-7 1
Surface	0.3	· 2	2	12	Medium	Medium		11/19/76	1/10/76	A-8 1
Tap wate	<0.1	<0.1	19	17	ND	Clear		11/30/76	1/13/76	A-9 1
Composit	24	12	76	35	Medium	Light		12/1/76	1/12/76	1 1
		diment	Se					<u>Soil</u>		
	Concent	ater	nple W	9	1,	Concentration	(Water	Sample	
	pp	ontent,	ght, C	Sample W		ррьь		Content,	Weight,	Sample
TCE	MC	%	5	No.	<u> </u>	MC TC		<u></u> %	<u>g</u>	No.
0.21	6.1	71.3	616	A-1-S).20 ND	(11.4	0.166	A-2
ND	0.34	46.8	240	A-5-S	2	NDc ND		7.8	0.141	A-4
0.036	0.31	49.0	244	A-7-S	2	ND 0.23		21.3	0.155	A-7
					45	0.04	(20.5	0.628	A-12

a Notes: ND = none detected. See "Determination of Methylchloroform in Water" for description of terms.

bDry basis, ppb by weight.

^CPractical detection limits: MC = 6 pg; TCE = 10 pg.

TABLE 5.3. DESCRIPTIONS OF SAMPLING LOCATIONS AT DOW CHEMICAL PLANT A, FREEPORT, TEXAS (NOVEMBER 9-12, 1976)

WATER

- (Water sample sites A1, A2, A5, A6, and A7 are not shown on the map, Figure 2.32. These sites are upstream, west of the boundaries of this map, and are upstream and downstream of the location where the canal from Dow Chemical Plant A discharges into the Brazos River.)
- A1 Surface sample, effluent canal from Plant A taken approximately 10 meters upstream in canal from confluence with Brazos River--moderate current, turbid.
- A2 Bottom sample (approximately 2-1/2 meters deep) -- same location as A1.
- A5 Surface sample, 400 meters downstream of plant outfalls in Brazos River--taken in center of channel--30 meters wide, 5-6 meters deep--swift current, turbid.
- A6 Bottom sample, same location as A5--taken 4 meters deep.
- A7 Surface sample, 800 meters upstream from plant outfalls in Brazos River--30 meters wide, 5-6 meters deep--steep banks bounded by recreational areas--swift current, turbid.
- A8 Shoreline surface sample in East Union Bayou--corresponds to air sampling site 12A--bayou bounded by dredge spoils--40 meters wide--moderate current, clean.
- A9 Tap water from Lake Jackson, Holiday Inn, Freeport, Texas.
- One 24-hour composite effluent sample collected November 11-12, 1976.

SEDIMENT

AlS - Effluent canal from Plant A, 10 meters upstream in canal from confluence with Brazos River-light tan, compacted sheet-like clay.

TABLE 5.3. (Continued)

SEDIMENT

- A5S 400 meters downstream of plant outfalls in Brazos River--fine textured, dark loam.
- A7S 800 meters upstream plant outfalls in Brazos River -- brown, fine textured silt/clay.

SOIL

- A2 Northeast of Freeport, south of turning basin on flood control levee--corresponds to air sampling site 2A--light industrial and commercial area--dredge spoil/gumbo--hard sand/silt over hard, compact clay.
- A4 400 meters east of Phillips petroleum plant--corresponds to air sampling site 4A--sand/shell spoil and root-bound sandy silt.
- A7 Overgrown vacant lot 200 meters south of Shrimp Hut in Surfside--residential area on Gulf---fine sand, some roots.
- Al2 -- 250 meters south off Route 332 near East Union Bayou--corresponds to air sampling site 12A-open dredge spoil area--fine sand eroded from spoil piles and roadway, some roots.

MONITORING DATA

PRODUCTION SITE 2

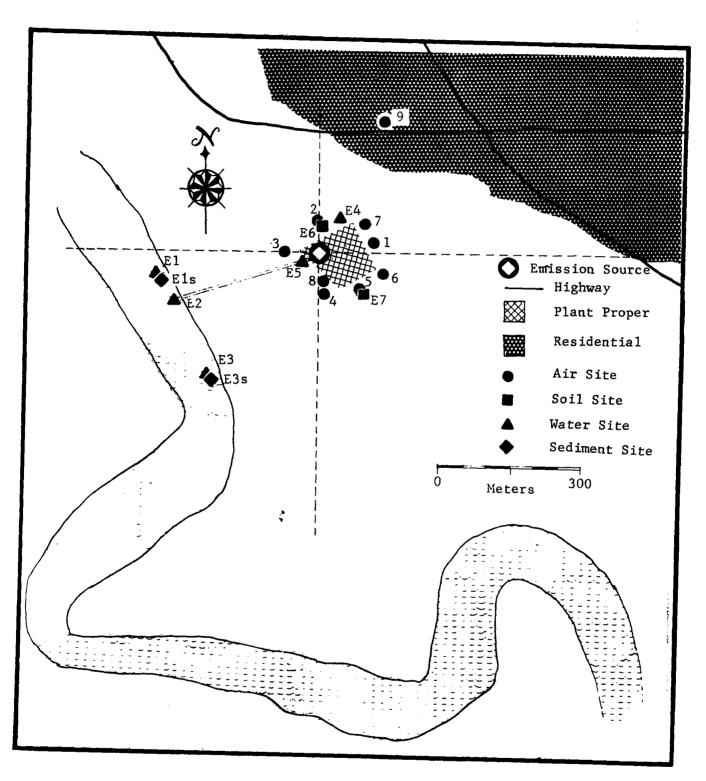


Figure 5.2. Sampling locations at Vulcan Materials Company, Geismar, Louisiana—methylchloroform production site.

TABLE 5.4. AMBIENT AIR MEASUREMENTS AT VULCAN MATERIALS (METHYLCHLOROFORM PRODUCER)

	Distance	Direction								Meteoro1	ogical 0	bserva	itions ^d
	from	from				entrat			Wind	Wind	Temper-		
Site	Plant,	Plant,	Date		Ambie	nt Air	, pp	bv ^c	Speed,	Direction,	ature,	RH,	Barometer
No.	km a	degrees b	1976	Time	MC	CC14	TCE	PCE	m/s	degrees ^a	C	% 	mm Hg
1	0.4	090	12/2	0950	≤0.3	0.42	≤1	≤0.3	5	320	12	51	765
				1015	≤0.3	0.30	≤1	≤0.3	5	320	12	51	765
				1040	≤0.3	0.22	≤1	≤0.3	5	340	12	52	765
				1135	≤0.3	0.17	≤1	≤0.3	4	360	13	50	765
				1150	≤0.3	0.73	≤1	≤0.3	4	360	13	50	765
				2025	2.0	5.7	≤1	0.8	0		4	71	764
				2055	0.8	3.7	≤1	≤0.3	0		4	71	764
•			12/3	0005	1.2	23	≤1	1.1	0		1	84	764
				0032	2.5	18	≤1	1.1	0		1	84	764
				0101	0.8	8.0	≤1	0.5	0		1	84	764
				0129	1.6	8.0	≤1	0.8	0		_	84	764
				0156	18	20	≤1	7.2	1	010	0	84	764
				0225	3.8	11	≤1	1.1	1	010	0	84	764
				0252	8.8	14	≤1	3.0	0		0	86	764
				0320	5.0	8.0	≤1	1.4	0		0	86	764
				0348	17	15	≤1	2.3	0		-1	88	764
				0415	16	15	≤1	2.3	0		-1	88	764
				0443	8.8	8.2	≤1	3.6	1	070	1	88	764
				0511	7.0	7.6	≤1	1.9	1	070	1	88	764
				0539	6.7	6.8	≤1	1.0	0		1	89	764
				0606	3.5	4.6	≤1	0.8	0		1	89	764
				0634	2.7	4.8	≤1	0.8	1	060	2	90	764
				0702	2.4	5.3	≤1	0.5	1	060	2	90	764
				0734	2.2	4.3	≤1	0.5	2	060	2	90	764
				0802	1.6	3.5	≤1	≤0.3	2	060	2	90	764
_				1035	0.6	6.3	≤1	0.5	1	060	14	63	764
2	0.3	010	12/2	1215	≤0.3	0.20	≤1	≤0.3	4	260	13	50	765
				1230	≤0.3	0.17	≤1	≤0.3	4	260	13	50	765
				2115	2.7	24	≤1	1.5	0		3	76	764
				2145	7.0	20	≤ 1	2.3	0		3	76	764
			12/3	1030	140	22	_ [≤] 1	2.3	_ <u>1</u>	060	14	63	764

5-15

TABLE 5.4. (Continued)

	Distance	Direction								Meteorol	ogical O	bserva	tions
	from	from			Conc	centrat	ion i	ln	Wind	Wind	Temper-		
Site	Plant,	Plant,	Date			ent Air			Speed,	Direction,	ature,	RH,	Barometer
No.	km a	degrees b	1976	Time	MC	CC1 ₄	TCE		m/s	degreesa	С	%	mm Hg
2	0.3	010	12/3	1057	77	8,8	≤1	3.6	1	060	14	63	764
,				1150	31	6.4	≤ <u>1</u>	≤0.3	4	140	15	63	764
				1220	9.2	3.4	≤ <u>1</u>		4	140	15	63	764
				1330	5.6	1.7	≤ <u>1</u>	1.1	2	290	14	62	763
	4			1357	≤0.3	0.18	≤ <u>1</u>	≤0.3	2	180	13	62	762
				1425	27	4.5	≤ <u>1</u>	1.1	2	180	13	62	762
				1453	1.2	4.1	≤ <u>1</u>	0.6	3	170	18	38e	762
				1520	92	10	≤ <u>1</u>	2.3	3	170	18	38 ^e	762 ·
				1545	140	12	≤ ₁	4.2	3	190	18	46 ^e	762
3	0.3	230	12/2	1252	≤0.3	0.20	≤ <u>1</u>	0.3	4	260	13	51	764
				1303	≤0.3	0.20	≤ <u>1</u>	0.3	4	260	13	51	764
			12/3	0928	≤0.3	4.2	≤ 1	0.3	4	100	13	60	764
				1000	≤0.3	0.88	≤ <u>1</u>	0.3	4	100	13	60	764
4	0,4	150	12/2	1330	≤0.3	0.73	≤ <u>1</u>	0.3	3	290	14	52	764
				1355	75	10	≤1	23	3	290	14	52	764
				1425	8.6	2.2	≤1	7.5	4	360	14	53	764
				1451	41	68	≤1	2.0	4	360	14	53	764
				1520	8.8	1	≤ <u>1</u>	19	4	360	14	53	764
5	0.6	140	12/2	1555	155	10	≤ 1	7	4	280	15	54	764
				1625	5.5	0.28	≤1	0.3	4	280	15	54	764
6	0.6	120	12/2	1645	11	11	≤1	4.3	3	. 280	14	54	766
				1720	14	10	≤1	6.2	3	280	14	54	766
				1745	4.2	3.6	≤ 1	0.8	0		9	58	764
				1815	3.8	7.0	≤ 1	0.5	0	olica destr	9	58	764
				1845	1.4	10	≤1	0.3	0		6	64	764
				1910	1.2	6.3	≤ 1	0,3	0	-	6	64	764
7	0.4	045	12/2	2215	4.0	10	≤ 1	2.3	0		3	78	764
			•	2240	1,4	5.9	≤ 1	3.6	0	00	3	78	764
				2310	2.7	52	≤ 1	6.7	0		1	80	764
				2335	1,4	50	≤ 1	3.6	0	-	1	80	764

TABLE 5.4. (Continued)

	Distance	Direction								Meteorol	ogical Ob	serva	tions d
Site	from Plant,	from Plant,	Date			entrat ent Air			Wind Speed,	Wind Direction,	Temper- ature,	RH,	Barometer,
No.	km a	degrees ^b	1976	Time	MC	CC1 ₄	TCE	PCE	m/s	degrees ^a	С	%	mm Hg
8	0.3	190	12/3	0835	0.8	6.7	≤1	1.0	3	110	10	82	764
				0902	≤0.3	1.4	≤1	≤0.3	3	110	10	82	764
				1252	≤0.3	0.23	≤1	≤0.3	2	290	17	58	763
9	3	030	12/3	1615	0.5	1.2	≤1	≤0.3	3	150	18	46	762
				1645	≤0.3	0.7	≤1	≤0.3	3	150	18	46	762

^aDistance and direction estimated scaled map unavailable.

 $^{\text{C}}$ To convert to $\mu\text{g/m}^3$ at 25 C multiply ppbv by $^{\text{MC}}$ -- 5.46 $^{\text{CCl}_4}$ -- 6.29 $^{\text{TCE}}$ -- 5.37 $^{\text{PCE}}$ -- 6.78.

d General weather conditions: 12/2/76 Partly cloudy in morning becoming clear about 1600 hours, no precipitation 12/3/76 Clear, sunny, no precipitation.

^bNorth - 360°.

e Possible malfunction of RH instrument.

TABLE 5.5. ANALYSIS OF WATER, SOIL, AND SEDIMENT SAMPLES FROM VULCAN MATERIALS PLANT (METHYLCHLOROFORM PRODUCER)^a

Sample No.	Date Sampled	Date Analyzed	Sediment in Sampl			centrati TO		weight CC14	Comments
				<u>W</u> :	ater				
E-1	12/2/76	12/30/76	Light	ND	2	2	5 6	2	
E-2	12/2/76	12/28/76	Light		344		4 394	193	
E-3	12/2/76	12/27/76	Ticht		169	9 2	4 . 226	92	
E-4	12/2/76	12/29/76	Medium	Heavy	3,314	4 36	0 152	629	Diluted 1→100
E-5	12/2/76	12/29/76	Clear	ND	16,500	4,30	0 31,675	9,060	Composite 1→50
		<u>Soil</u>					Sediment		
	Sample	Water	Concentra			Sample	Water	Concent	
Samp1	e Weight,	Content,	ppbb		Sample	Weight,	Content,	pp	
No.	g	%	MC	TCE	No.	g	%	MC	TCE
E-6	0.298	26.3	0.45	0.62	E-1-S	0.209	54.3	0.13	0.25
E-7	0.304	20.4	0.94	0.18	E-3-S	0.255	27.7	2.6	3.2

Notes: ND = none detected. See "Determination of Methylchloroform in Water" for description of terms.

bDry basis, ppb by weight.

TABLE 5.6. DESCRIPTIONS OF SAMPLING LOCATIONS AT VULCAN MATERIALS, GEISMAR, LOUISIANA (DECEMBER 2, 1976)

WATER

- E 1 Surface sample taken from bank of Mississippi River 30 meters upstream from plant outfall-area used for barge mooring--moderate current, turbid.
- E 2 Surface sample taken at end of submerged outfall pipe in Mississippi River--effluent discharged subsurface--moderate current, turbid.
- E 3 Surface sample from bank of Mississippi River 75 meters downstream from plant outfall--barge moored within 20 meters of sampling point--moderate current, turbid.
- E 4 Roadside ditch 60 meters north of Vulcan office (1 meter wide, 1-3 centimeters deep)--ditch received runoff from heavily trafficked road.
- E 5 Twenty-four hour composite effluent sample from inside plant.

SEDIMENT

- ElS Shoreline sample in Mississippi River 30 meters upstream from plant outfall--gray-black silt/ sand, oil texture.
- E3S Shoreline sample in Mississippi River 75 meters downstream from plant outfall--gray-black silt/sand, oily texture.

SOIL

- E 6 100 meters east of Vulcan office--road cut in front of plant--sandy soil.
- E 7 200 meters south of plant--strip between service road and railroad--sandy silt, little humus or roots.

MONITORING DATA

PRODUCTION SITE 3

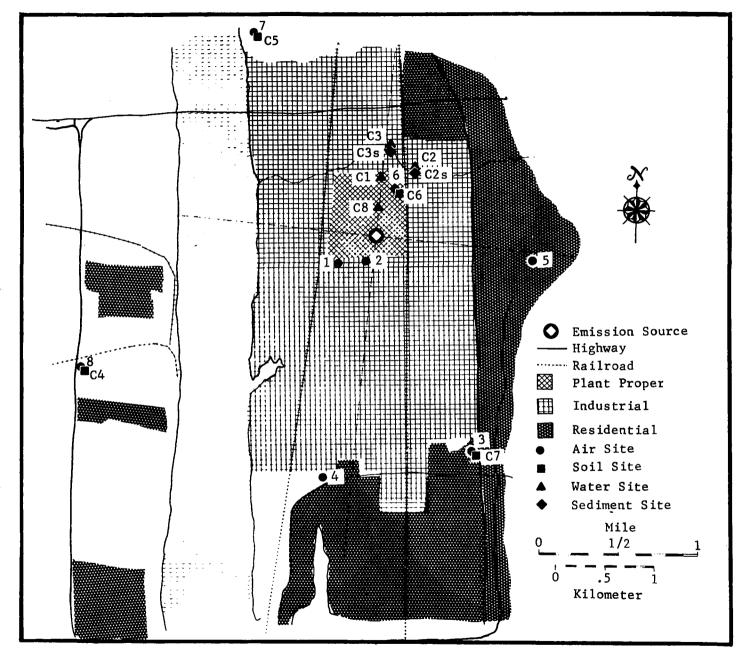


Figure 5.3. Sampling locations at Ethyl Corporation, Baton Rouge, Louisiana--methylchloroform production site.

TABLE 5.7. AMBIENT AIR MEASUREMENTS AT ETHYL CORPORATION (METHYLCHLOROFORM PRODUCER)

	Distance	Direction										Observat:	ions ^C
	from	from				centra			Wind	Wind	Temper		
Site	•	Plant,	Date			ent Air			Speed,	Direction,	ature,		Barometer,
No.	km	degreesa	1976	Time	MC	CC1 ₄	TCE	PCE	m/s	degrees ^a	С	cm)	mm Hg
1	0.4	240	11/18	0815	≤0.3	7.3	5.6	5.2	4	020	14	$\mathtt{ND}^{\mathbf{d}}$	761
				0845	≤0.3	1.9	1.9	1.6	4	020	14	ND	761
2	0.2	195	11/18		≤0.3	0.80	≤ 1	_0.7	4	060	16	ND	761
			,	0945	≤0.3	4.8	≤ 1	3.2	4	060	16	ND	761
			11/19		≤0.3	1.1	<u>≤1</u>	≤0.3	3	120	12	(trace)	758
				1014	≤0.3	2.4	5.4		4	030	11	(trace)	758
				1100	≤0.3	47	7.2	37	4	060	11	(0.1)	758
				1200	≤0.3	0.90	2.4		4	030	11	(0.4)	758
3	2.4	150	11/18		≤0.3	0.15	<u>-</u> 1.	≤0.3	4	060	18	ND	761 `
			•	1730	≤0.3	0.16	≤ <u>1</u>	≤0.3	0	·360	17	ND	759
				1800	≤0.3	0.12	_ ≤1	≤0.3	Ö	360	17	ND	759
				1930	≤0.3	0.12	≤1	0.5	2	360	17	ND .	759
				2000	0.6	0.13	≤1	0.6	2	360	17	ND	759
				2030	1.1	0.15	≤1	0.6	0	360	16	ND	759
				2100	1.0	0.15	≤1	0.6	0	360	16	ND	759
				2130	1.5	0.21	≤ <u>1</u>	1.0	Ö	360	16	ND	759
				2200	1.0	0.33	≤1	1.0	Ö	360	16	ND	759
				2230		0.26	≤1	0.8	0	360	16	ND	759
				2300	2.2		≤1	0.6	0	360	16	ND	759
				2330	3.9	0.52	≤1	0.4	0	360	16	ND	759
				2400	0.6	0.50	≤1	0.4	1	140	14	ND	759
			11/19	0030	1.5	2.7	≤1	0.5	0	360	14	ND	759
				0100	0.9	1.9	≤1	≤0.3	0	360	14	(0.02)	759
				0130	0.9	1.3	≤1	≤0.3	0	360	12	ND	759
				0200	1.6	0.9	≤1	≤0.3	1	120	12	(0.02)	758
				0545	≤0.3	0.33	≤1	≤0.3	2	090	12	(0.02)	759
				0615	≤0.3	0.37	≤1	≤0.3	2	090	12	ND	759
				0700	≤0.3	0.32	≤1	≤0.3	4	060	10	(0.02)	758
				0730	≤0.3	0.26	≤1	≤0.3	4	070	10	ND	758
				0800	≤0.3	0.26	≤1 .	≤0.3	3	060		(trace)	758
		<u> </u>		0830	0.5	0.22	≤1	≤0.3	3	060	11	ND	758

5-2

TABLE 5.7 (Continued)

	Distance from	Direction from			Con	entrat	don d	în	Wind	<u>Meteorol</u> Wind	ogical (Temper-		ionsc
Site		Plant,	Date			ent Air		-	Speed,	Direction,	ature,		Barometer,
No.	km	degrees ^a	1976	Time	MC	CC1 ₄	TCE	PCE	m/s	degrees ^a	C ,	cm)	mm Hg
3	2.4	150	11/19	0900	≤0.3	0.21	≤1	≤0.3	4	060	11	(trace)	758
				0930	≤0.3	0.19	≤1	≤0.3	4	060	11	ND	758
				1000	≤0.3	0.18	≤1	≤0.3	4	030	11	(trace)	758
4	2.6	180	11/18	1055	≤0.3	0.12	≤1	≤0.3	2	020	18	ND	761
				1125	≤0.3	0.12	≤1	≤0.3	2	020	18	ND	761
5	2.2	095	11/18	1155	≤0.3	0.11	≤1	≤0.3	1	070	18	ND	761
				1225	≤0.3	0.17	≤1	≤0.3	1	070	18	ND	761
6	0.7	010	11/18	1255	≤0.3	0.85	≤1	0.5	1	240	19	ND	761
•				1355	≤0.3	0.36	≤1	0.4	2	340	19	ND	761
			11/19		≤0.3	0.38	≤1	≤0.3	2	350	12	(trace)	
				0415	≤0.3	1.3	≤1	0.6	2	350	12	ND .	758
				0445	≤0.3	0.18	≤1	≤0.3	1	340	12	(0.07)	
				0515	≤0.3	0.43	≤1	≤0.3	1	340	12	ND	758
7	2.2	330	11/18		≤0.3	0.16	≤1	≤0.3	0	360	18	ND	761
8	3.2	240	11/18		≤0.3	0.14	≤1	≤0.3	2	080	18	ND	761
				1615	≤0.3	0.13	≤1	≤0.3	2	080	18	ND	761
			11/19		≤0.3	0.67	≤1	≤0.3	4	040	11	(0.7)	757
				1330	≤0.3	0.17	≤1	≤0.3	4	040	11	ND	757

a_{North - 360°.}

CC1₄ -- 6.29

TCE -- 5.37

PCE -- 6.78.

^CGeneral weather conditions: 11/18/76 Clear in morning becoming cloudy about 1400 hours with light intermittent rain beginning about 2200 hours 11/19/76 Cloudy all day, intermittent rain throughout the day.

 $^{^{}b}$ To convert to μ g/m 3 at 25 C multiply ppbv by MC -- 5.46

d_{ND} = not determined.

TABLE 5.8. ANALYSIS OF WATER, SOIL, AND SEDIMENT SAMPLES FROM ETHYL CORPORATION (METHYLCHLOROFORM PRODUCER)^a

Sample	Date	Date	Sediment	Spargi			ion, ppb t		
No.	Sampled	Analyzed	in Sample	Foan	n MC	T(CE CHC1	CC1 ₄	Comments
				Wa	iter				
C-1	11/18/76	12/22/76	Light	ND	74	128	3 105	67	
C-2	11/18/76	12/22/76	Heavy	Heavy	0.	4 ().4 6	0.1	Surface
C-3	11/18/76	12/21/76	Heavy	Heavy	20	37	37	23	Surface
C-8	11/18/76	12/22/76	Clear	Light	10	10	32	12	Composite
C-9	11/19/76	12/22/76	Clear	ND	0.	05 (2	0.2	Tap water
		<u>Soil</u>	* 3				Sediment	<u>:</u>	
Samp1	Sample Weight,	Water Content,	Concentrat	ion,	Sample	Sample Weight	Water Content		tration,
No.	g	%	MC	TCE	No.	g		MC	TCE
C-4	0.246	26.3	0.20	NDC	C-2-S	1.07	27.2	0.81	ND
C-5	0.261	20.5		ND	C-3-S	0.330	77.5	ND	116
C-6	0.274	27.5	0.13	ND					
C-7	0.192	17.3	0.28	ND					

Notes: ND = none detected. See "Determination of Methylchloroform in Water" for description of terms.

bDry basis, ppb by weight.

^CPractical detection limits: MC = 6 pg; TCE = 10 pg.

TABLE 5.9. DESCRIPTIONS OF SAMPLING LOCATIONS AT ETHYL CORPORATION, BATON ROUGE, LOUISIANA (NOVEMBER 18-19, 1976)

WATER

- C1 Effluent sample taken immediately above the settling pond weir to receiving bayou--strong aromatic odors--light blue-green color, very slippery feel.
- C2 Surface sample taken in receiving bayou 200 meters upstream from plant outfall (7 meters wide, 0.5 1.5 meters deep) -- moderate current; anaerobic odor; black murky color with oil slick and tar globules on surface (bayou flows through heavily industrialized area and under railroad tracks).
- C3 Surface sample taken in receiving bayou 300 meters downstream from plant outfall (7 meters wide, 1-2 meters deep)--moderate flow; water quality appearance same as at C2 with additional slippery feel.
- C8 24-hour composite effluent sample--6:00 a.m. November 18 to 6:00 a.m. November 19, 1976.

SEDIMENT

- C2S Upstream in receiving bayou 200 meters from plant outfall; same location as water sample site C2; taken 1 meter from bank; aerobic, black, oily ooze.
- C3S Downstream in receiving bayou 300 meters from plant outfall; same location as water sample C3; taken 0.5 meters from bank; anaerobic, black, oily ooze.

SOIL

- C4 Grove Plantation approximately 0.8 kilometers west of Mississippi River--corresponds to air sampling site 8--taken at edge of cane field; wet silt/gumbo.
- C5 0.5 kilometers east of dead end of Mengel Road (some industry and a tank-washing operation within 0.5 kilometer radius)--corresponds to air sampling site 7--5 meters north off road in open field; loam.
- C6 Northeast corner of Ethyl parking lot on steep banks of bayou--much railroad traffic in immediate vicinity--hard clay.
- C7 Narrow (0.6 meter) edge of parking lot at Istrauma Baptist Church--residential area approximately 10 meters from street--gravel, sand/rock.

MONITORING DATA

PRODUCTION SITE 4

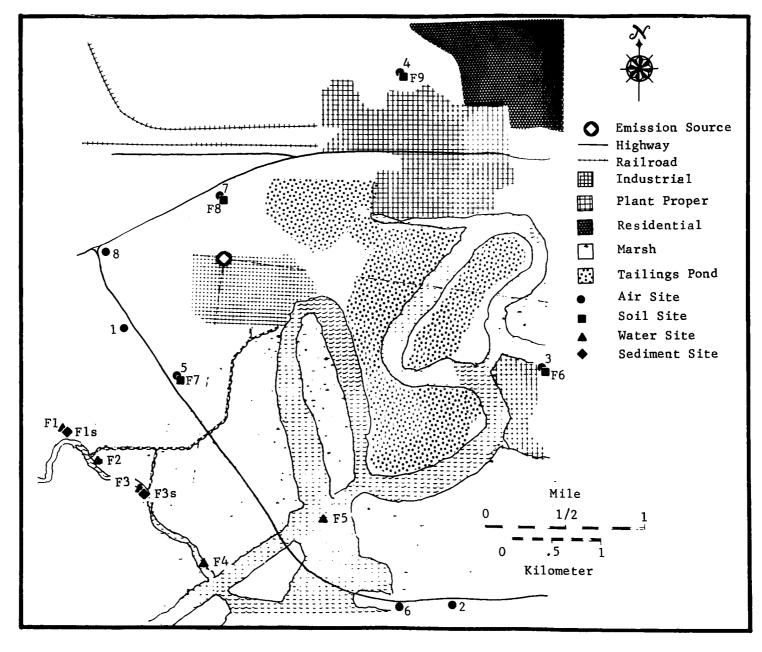


Figure 5.4. Sampling locations at PPG Industries, Lake Charles, Louisiana--methylchloroform production site.

TABLE 5.10. AMBIENT AIR MEASUREMENTS AT PPG INDUSTRIES (METHYLCHLOROFORM PRODUCER)

	Distance	Direction									ogical O	bserva	tionsc
	from	from				centrat		_	Wind	Wind	Temper-		
Site	•	Plant,	Date			ent Air			Speed,	Direction,	ature,	RH,	Barometer
No.	km	degrees ^a	1976	Time	MC	CC1 ₄	TCE	PCE	m/s	degrees ^a	C	%	mm Hg
1	1.3	215	12/6	1014	1.3	0.24	2.7	0.4	8	150	15	87	755
				1043	0.8	0.17	2.2	0.3	8	150	15	87	755
2	4.2	140	12/6	1123	≤0.3	0.13	≤1	≤0.3	7	160	17	90	754
				1152	≤0.3	0.19	≤1	≤0.3	7	160	17	90	754
3	3.5 i	85	12/6	1302	≤0.3	0.13	≤1	≤0.3	9	160	17	87	753
				1330	≤0.3	0.20	≤1	≤0.3	9	160	17	87	753
			12/7	0900	0.7	0.24	≤1	0.4	7	340	6	76	759
4	2.7	40	12/7	1512	0.5	0.21	≤1	≤0.3	7	330	8	60	760
				1540	≤0.3	0.17	≤1	≤0.3	7	330	8	60	760
5	1.4	195	12/6	2100	≤0.3	0.18	≤1	≤0.3	10	360	14	80	756 ·
				2129	≤0.3	0.15	≤1	≤0.3	10	·360	14	80	756
				2157	≤0.3	0.15	≤1	≤0.3	10	350	12	80	756
				2226	≤0.3	0.17	≤1	≤0.3	9	350	12	77	756
				2254	≤0.3	0.20	≤1	≤0.3	9	350	12	77	756
6	4.0	150	12/6	2331	≤0.3	0.17	≤1	≤0.3	7	350	12	76	756
				2400	≤0.3	0.31	≤1	≤0.3	7	350	12	76	756
			12/7	0028	≤0.3	0.28	≤1	≤0.3	6	360	12	74	756
				0056	0.4	0.20	≤1	≤0.3	6	360	12	74	756
				0125	1.7	0.88	15	3.8	6	360	12	74	756
				0153	≤0.3	0.19	≤1	0.4	7	360	11	80	757
				0222	≤0.3	0.20	≤1	≤0.3	7	360	11	80	757
				0249	1.5	0.64	6.6	2.5	6	340	9	80	757
				0317	5.0	0.21	≤1	≤0.3	6	340	9	80	757
				0345	8.5	0.31	2.2	0.7	7	340	8	80	757
				0413	0.7	0.19	≤1	≤0.3	7	340	8	80	757
				0441	≤0.3	0.20	≤1	≤0.3	7	340	7	79	758
				0509	6.5	0.92	12	5.0	7	340	7	79	758
				0537	≤0.3	0.15	≤1	≤0.3	6	350	6	79	758
				0605	1.4	0.17	≤1	≤0.3	6	350	6	79	^ 758
				0633	1.2	0.26	≤1	≤0.3	7	350	6	79	758
				0701	1.4	0.17	≤1	≤0.3	7	350	6	79	758

TABLE 5.10. (Continued)

	Distance	Direction								Meteorol	ogical Ob	serva	itions ^C
Site No.	from Plant, km	from Plant, degrees ^a	Date 1976	Time		centrat ent Air CC1 ₄	r, pph	_	Wind Speed, m/s	Wind Direction, degrees a	Temper- ature, C	RH,	Barometer mm Hg
		150	20/2	0707	0.6	0.65			_				
6	4.0	150	12/7	0727	2.6	0.65	5.8		7	350	6	79	758
				0755	5.0	0.96	8.0	3.2	6	350	6	76	758
				0823	5.3	0.73	5.8	3.2	6	350	6	76	758
				0855	≤0.3	0.20	≤1	≤0.3	7	340	6	76	759
				0958	≤0.3	0.15	≤1	≤0.3	8	320	5	76	759
				1015	≤0.3	0.15	≤1	≤0.3	8	320	5	76	759
				1030	≤0.3	0.13	≤1	≤0.3	7	320	5	73	760
				1045	≤0.3	0.15	≤1	<0.3	7	320	5	73	760
				1100	≤0.3	0.23	≤1	≤0.3	7	320	5	73	760
				1205	2.4	0.40	4.6	1.8	6	320	6	73	760
				1232	≤0.3	0.20	≤1	≤0.3	7	340	6	70	760
				1259	5.0	0.64	7.0	≤0.3	7	340	6	70	760
				1328	2.1	0.26	4.6	≤0.3	6	340	7	68	760
				1358	≤0.3	0.20	≤1	_ ≤0.3	6	340	7	68	760
7	0.6	360	12/7	1030	≤0.3	0.21	≤1	≤0.3	7	320	6.7	73	760
8	1.3	265	$\frac{12}{7}$	1052	0.4	0.75	 ≤1	0.4	7	320	6.7	73	760

^aNorth - 360°.

b To convert to g/m^3 at 25 C multiply ppbv by MC -- 5.46 CC1₄ -- 6.29 TCE -- 5.37 PCE -- 6.78.

^cGeneral weather conditions: 12/6/76 Cloudy, rainfall recorded from 1000 to 2100 hours, very heavy at times 12/7/76 Cloudy with very slight clearing in late afternoon, no precipitation.

TABLE 5.11. ANALYSIS OF WATER, SOIL, AND SEDIMENT SAMPLES FROM PPG INDUSTRIES (METHYLCHLOROFORM PRODUCER)^a

Sample No.	Date Sampled	Date Analyzed	Sedimer in Samp	-	ging <u>Con</u> am MC		on, ppb by CHCl ₃	weight CC14	Comments
			····	w	ater			.	
									
F-1	12/7/76	1/5/77	Heavy		132	353	11	29	
F-2	12/7/76	1/7/77	Heavy		181	. 447	85	40	
F-3	12/7/76	1/7/77	Heavy		58	179	30	12	
F-4	12/7/76	1/6/77	Heavy		161	403	34	38	
F-5	12/7/76	1/4/77	Heavy		5	29	12	<0.1	
F-10	12/7/76	12/13/76	Clear		C	0.3	1 <0.1	<0.1	Tap water
		Soil		4 y			Sediment		
	Sample	Water	Concentr	ation,		Sample	Water	Concen	tration,
Samp]	le Weight,	Content,	ppb	b	Sample	Weight,	Content,	p 1	pb ^b
No.	g	%%	MC	TCE	No.	g	%	MC	TCE
F-6	0.337	12.2	0.14	0.11	F-1-S	0.263	72.6	2.2	146
F-7	0.286	22.5	1.0	$ND^{\mathbf{C}}$	F-2-S	d			
F-8	0.232	19.6	0.61	0.077	F-3-S	0.265	71.0	1.1	15
F-9	0.782	28.3	0.22	ND	F-4-S	d			

anotes: ND = none detected. See "Determination of Methylchloroform in Water" for description of terms.

bDry basis, ppb by weight.

^cPractical detection limits: MC = 6 pg; TCE = 10 pg.

d Samples damaged in shipping (including duplicates).

TABLE 5.12. DESCRIPTIONS OF SAMPLING LOCATIONS AT PPG INDUSTRIES, LAKE CHARLES, LOUISIANA (DECEMBER 7, 1976)

WATER

- F1 Surface sample 50 meters upstream from plant outfall in Bayou d'Inde (10 meters wide, 2.5 3 meters deep)--slow current; high conductivity (>8000 microohms/cm); dark colored, turbid.
- F2 Surface sample--confluence of northernmost PPG effluent canal in Rayou d'Inde--5 meters wide, 1 meter deep.
- F3 Surface sample--confluence of southernmost PPG effluent canal in Bayou d'Inde--200 meters below confluence of first canal (F2); 5 meters wide, 1 meter deep.
- F4 Surface sample--50 meters downstream of southernmost PPG effluent canal (F3) in Bayou d'Inde-10 meters wide, 2-3 meters deep.
- F5 Surface sample taken at mouth of Calcasieu River in Prien Lake, downstream of PPG outfall.
- F10 Top water taken from the Sheraton Motel, Lake Charles, Louisiana.

SEDIMENT

- F1S 50 meters upstream from plant outfall in Bayou d'Inde--black ooze.
- F2S Confluence of northernmost PPG effluent canal in Bayou d'Inde--black, oily ooze.
- F3S Confluence of southernmost PPG effluent canal in Bayou d'Inde--black, oily ooze.
- F4S 50 meters downstream of southernmost PPG effluent canal (F3) in Bayou d'Inde--black, oily ooze.

SOIL

- F6 Lake Shore Drive near Port of Lake Charles -- residential area -- sand/clay roadfill.
- F7 I210 drainage ditch 400 meters north of bridge over Prien Lake--ditch composed of both concrete and sandy clay.

MONITORING DATA
USER SITE

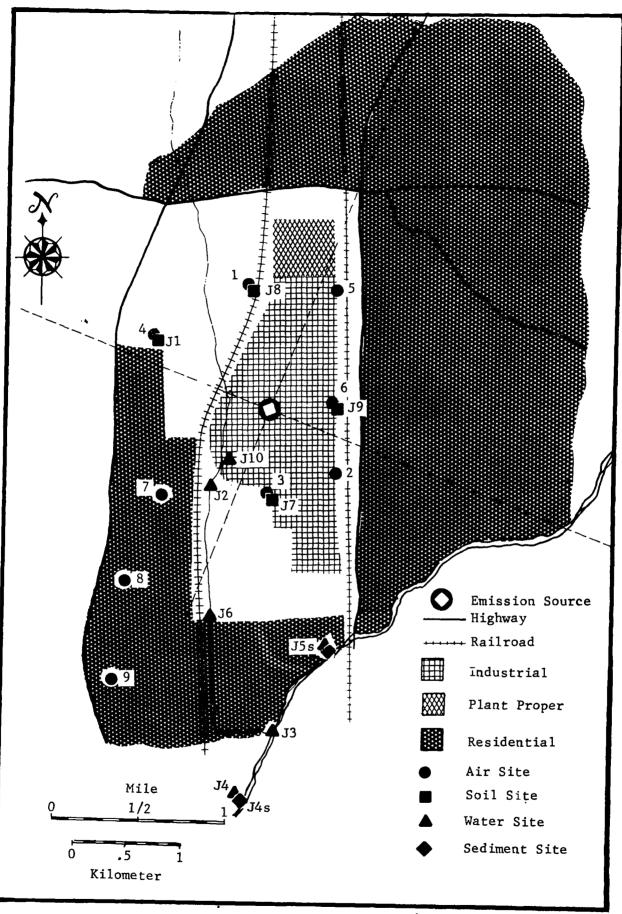


Figure 5.5 Sampling locations at Boeing Company, Auburn, Washington--methylchloroform user site. 5-33

TABLE 5.13. AMBIENT AIR MEASUREMENTS AT BOEING COMPANY, AUBURN PLANT (METHYLCHLOROFORM USER)

	Distance	Direction							-		ogical O	serva	itions ^C
0.1.	from	from	n .			centrat			Wind	Wind	Temper-	2011	D
Site No.	Plant, km	Plant, degrees ^a	Date 1977	Time	MC MC	CC1 ₄	TCE	PCE	Speed, m/s	Direction, degrees ^a	ature, C	RH, %	Barometer, mm Hg
1	0.7	335	1/10	1330	10.0	0.11	0.38	0.29	0.8	180	-1	86	761
			·	1350	4.6	0.10	0.15.	0.18	0.8	180	-1	86	761
				1410	6.8	0.11	0.15	0.29	1.4	180	0	86	761
				1925	0.8	0.11	0.14	0.29	d	d	-1	88	761
			1/11	0750	3.8	0.09	0.64	0.20	d	d	-1	92	758
			ŕ	0810	5.0	0.06	0.64	0.25	d	d	0	92	758
				0900	2.0	0.09	0.26	0.69	d	d	0	92	758
				1630	1.6	0.10	0.26	0.69	0.6	150	3	93	754
2	0.6	125	1/10	1455	0.4	0.11	0.34	0.08	1.4	180	0	86	760
			•	1515	0.5	0.13	0.14	0.11					
3	0.9	165	1/10	1542	0.4	0.13	0.14	0.06	1.2	180	-2	87	761
			•	1602	0.4	0.13	0.14	0.06	1.2	1:80	-2	87	
			1/11	1006	2.3	0.10	1.8	0.98	0.6	50	1	92	757
			•	1027	3.0	0.07	0.92	1.4	0.6	50	1	92	757
				1655	4.5	0.10	0.83	0.60	0.6	150	3	93	753
				1715	4.9	0.11	0.80	0.66	0.6	150	3	93	753
				1735	5.0	0.11	0.90	0.66	0.5	150	2	93	753
4	1.1	280	1/10	1630	0.6	0.14	0.14	0.08	0.7	150	2	87	761
			•	1655	0.6	0.13	0.14	0.11	0.7	150	2		
			1/11	1550	6.2	0.11	0.90	0.94	0.5	210	3	94	754
			•	1610	5.2	0.11	0.76	0.94	0.5	210	3	94	754
				1825	5.2	0.14	0.92	0.69	d	d	2	94	753
5	0.9	005	1/10	1945	0.9	0.15	0.18	0.42	d	d	1	88	761
6	0.4	050	1/11	0923.	1.6	009	0.18	0.65	d	d	-1	92	758
				0943	2.3	0.13	0.30	0.76	d	d	-1	92	758
7	1.2	215	1/11	1120	6.9	0.09	0.80	0.90	d	d	<u> </u>	92	757
				1140	7.3	0.09	0.84	0.73	d	d	1	92	757
				1200	7.3	0.08	0.76	0.69	d	d	$\bar{\overline{1}}$	92	757
	•			1220	7.4	0.10	1.0	0.73	0.5	360	2	93	757
				1240	6.9	0.10	1.0	0.78	0.5	360	2	93	757
				1325	7.3	0.10	1.0	0.82	d	d	2	93	757

TABLE 5.13. (Continued)

	Distance	Direction								Meteorol	ogical Ob	serva	tionsc
Site	•	from Plant,	Date	en t	Ambie	entrat	, ppb	_v b	Wind Speed,	Wind Direction,	Temper-	RH,	Barometer,
No.	km	degrees ^a	1977	Time	MC	CC1 ₄	TCE	PCE	m/s	degrees ^a	C	%	mm Hg
7	1.2	215	1/11	1805	4.8	0.10	0.80	0.66	d	d	2	93	753
8	2.0	210	1/11	1345	7.8	0.10	1.0	0.90	-	d	2	93	756
			·	1405	8.4	0.09	1.1	1.0	d	d	2	93	756
9	2.9	200	1/11	1430	4.4	0.11	0.82	0.69	0.6	180	3	93	756
				1450	4.0	0.11	0.78	0.65	0.6	180	3	93	756
10	1.1	255	1/11	1510	7.0	0.10	1.2	0.97	0.5	210	3	93	756
				1530	8.1	0.10	1.1	1.0	0.5	210	3	93	756

^aNorth - 360°.

 b To convert μ g/m 3 to ppbv multiply by MC $\stackrel{--}{}$ 5.46 c CC1 $_{4}$ $\stackrel{--}{}$ 6.29 c TCE $\stackrel{--}{}$ 5.37

 $^{\mathbf{c}}$ General weather conditions: 1/10/77 Heavy overcast of fog; no precipitation

PCE -- 6.78.

1/11/77 Heavy overcast of clouds, fog; light rain from 0600 to 1500 hours.

 $^{
m d}_{
m Wind}$ speed below starting threshold of 0.75 mph for MRI 1071 weather station.

5-36

ANotes: ND = none detected. See "Determination of Methylchloroform in Water" for description of terms.

Dry basis, ppb by weight,

Sample primarily roots and peat moss--could not be run as soil sample.

dPractical detection limits: MC = 6 pg; TCE = 10 pg.

TABLE 5.15. DESCRIPTIONS OF SAMPLING LOCATIONS AT BOEING COMPANY, AUBURN, WASHINGTON (JANUARY 10-12, 1977)

WATER

- J2 Surface sample--Boeing outfall from settling pond--taken as effluent spilled from concrete culvert--water clear, warm, smelled of chlorine, slippery to touch.
- J3 Surface sample 1 meter upstream in outfall canal to Stuck River 3 kilometers downstream of plant outfall (1 meter wide, 0.5 meter deep)--clear, moderate current.
- J4 Surface sample 100 meters downstream from plant outfall in Stuck River (50 meters wide, 0.5 1 meter deep) -- swift current, turbid, very cold, ice cover near shore; receives runoff from land-moving operation near banks.
- J5 Surface sample 30 meters upstream from plant outfall in Stuck River (40 meters wide, 0.5 1 meter deep) -- turbid, very cold, ice cover near bank, swift current.
- J6 Surface sample--Boeing outfall canal 1.5 kilometers downstream from plant outfall--residential area; 3-4 meters wide; 0.5 meter deep; taken below bridge; clear, moderate current.
- J10 5-hour composite effluent sample--undiluted--first 5 hours of 8-hour discharge.

SEDIMENT

- J4S 100 meters downstream from plant outfall in Stuck River--compact, fine sand.
- J5S 30 meters upstream from plant outfall in Stuck River -- compact, fine sand.

SOIL

- J1 dead end road northwest of plant--corresponds to air sampling site 4; overgrown vacant lot at side of road--20 meters north of intersection, light to moderate traffic--root-bound sandy clay.
- J7 0.5 kilometers southwest of southern plant guard gate--corresponds generally to air sampling site 3--taken 10 meters east off moderately trafficked blacktop road--wet, sandy clay.
- J8 Ditch paralleling railroad tracks on northwest corner of plant property, 50 meters from plant fence--corresponds to air sampling site 1--loam.
- J9 Ditch between C Street and railroad tracks east of plant, 30-40 meters from plant fence--periodically heavy train and auto traffic--corresponds to air sampling site 6--loose textured, rootbound loam.

MONITORING DATA

BACKGROUND SITE

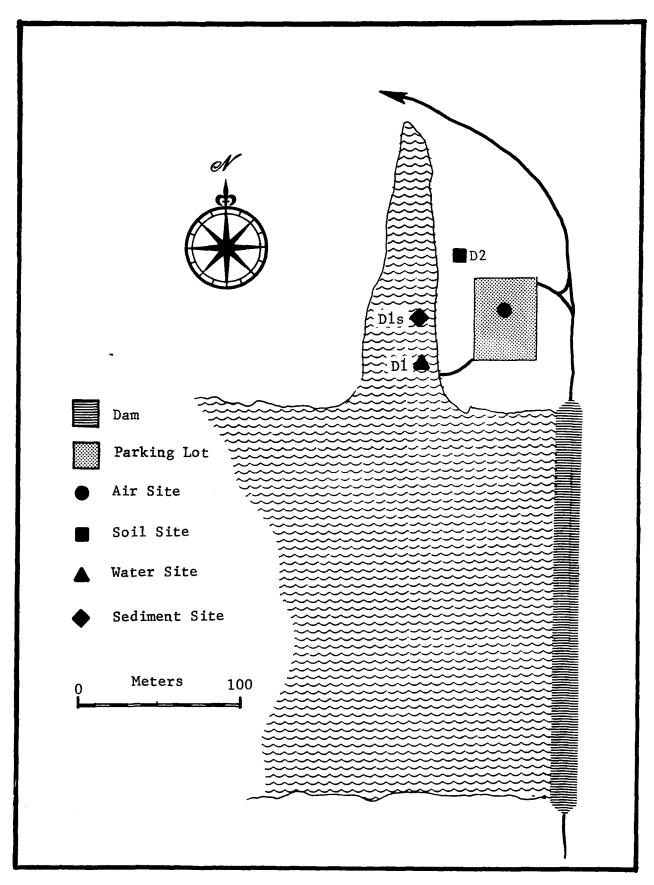


Figure 5.6. Sampling locations at St. Francis National Forest, Helena, Arkansas--background site.

TABLE 5.16. AMBIENT AIR MEASUREMENTS AT ST. FRANCIS NATIONAL FOREST (RURAL BACKGROUND)

			ncentr					logical Observ		
			ient A			Wind Speed,		-		Barometer
Date ———	Time	MĈ	CC1 ₄	TCE	PCE	m/s	degrees ^b	C	% 	mm Hg
11/30/76	1030	≤0.3	0.15	≤1	≤0.3	1	170	3	41	761
	1055	≤0.3	0.15	≤1	≤0.3	1	170	3	41	761
	1125	≤0.3	0.13	≤1	≤0.3	1	170	3	41	761
	1146	≤0.3	0.14	≤1	≤0.3	2	210	5	38	760
	1216	≤0.3	0.15	≤1	≤0.3	2	210	5	38	760
	1243	≤0.3	0.12	≤1	≤0.3	3	230	4	39	760
	1308	≤0.3	0.11	≤1	≤0.3	3	230	4	39	760
	1334	≤0.3	0.14	≤1	≤0.3	3	225	5	42	759
	1400	≤0.3	0.14	≤1	≤0.3	3	225	5	42	759
	1426	≤0.3	0.13	≤1	≤0.3	3	225	4	44	759
	1452	≤0.3	0.15	≤1	≤0.3	4	220	4	44	759
	1517	≤0.3	0.14	≤1	≤0.3	4	220	· 4	44	759
	1545	≤0.3	0.15	≤1	≤0.3	3	160	4	49	759
	1611	≤0.3	0.14	≤1	≤0.3	3	160	4	49	759
	1636	≤0.3	0.14	≤1	≤0.3	2	165	2	58	759
	1700	≤0.3	0.15	≤1	≤0.3	2	165	2	58	759
	1730	≤0.3	0.15	≤1	≤0.3	2	165	1	66	759
	1800	≤0.3	0.14	≤1	≤0.3	2	165	1	66	759
	1825	≤0.3	0.15	≤1	≤0.3	2	165	1	66	759
	1850	≤0.3	0.14	≤1	≤0.3	2	160	1	70	759
	1917	≤0.3	0.14	≤1	≤0.3	2	160	1	70	759
	1955	≤0.3	0.15	≤1	≤0.3	4	170	2	73	758
	2022	≤0.3	0.15	≤1	≤0.3	4	170	2	73	758

^aTo convert to $\mu g/m^3$ at 25 C multiply ppbv by MC -- 5.46; CC1₄ -- 6.29; TCE -- 5.37; PCE -- 6.78.

b_{North} - 360°.

 $^{^{\}mathrm{c}}$ General weather conditions: Clear, sunny, no precipitation.

TABLE 5.17. ANALYSIS OF WATER, SOIL, AND SEDIMENT SAMPLES FROM THE BACKGROUND SITE^a

Sample No.	Date Sampled	Date Analyzed	Sediment in Sampl			TCE	cHC1 ₃	CC1 ₄	Comments
				<u>. </u>	<u>Vater</u>				
D-1	11/30/76	12/20/76	Clear	ND	0.4	<0.0	5 2	0.2	
D-3	11/30/76	12/14/76	Clear	ND	0.4	22	3	<0.1	Tap water
		Soil					Sediment		
	Sample	Water	Concentra			Sample	Water		tration,
Samp:	le Weight,	Content,	ppb ^b)	Sample	Weight,	Content,	p	pb ^b
<u>No</u>	g	%	MC	TCE	No.	g		MC	TCE
D-2	0.200	25.8	0.54	0.63	D-1-S	0.198	45.0	0.67	2.2
D-2	0.641	24.3	0.29	<0.42c	D-1-S	0.115	54.0	0.23	$_{ m ND}$ d

^aNotes: ND = none detected. See "Determination of Methylchloroform in Water" for description of terms.

bDry basis, ppb by weight.

 $^{^{\}mathrm{c}}$ Possible interference present.

dPractical detection limits: MC = 6 pg; TCE = 10 pg.

TABLE 5.18. DESCRIPTIONS OF SAMPLING LOCATIONS AT STORM CREEK LAKE, ST. FRANCIS NATIONAL FOREST, HELENA, ARKANSAS (NOVEMBER 30, 1976)

WATER

- D1 Surface sample taken from concrete boat dock on Storm Creek Lake 100 meters south of parking lot--little wave action; clear.
- D3 Top water taken from the Holiday Inn, Helena, Arkansas.

SEDIMENT

D1S - Taken from boat dock on Storm Creek Lake 100 meters south of parking lot--mud and sand with cover of light leaf litter; snail and mussel shells abundant.

SOIL

D2 - west-facing slope north of boat ramp 75 meters west-southwest of parking lot--sandy humus with decomposing leaf litter and many roots--dense undergrowth of honeysuckle vines.

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15. SUPPLEMENTARY NOTES

16. ABSTRACT

The levels of methylchloroform (MC) in various environmental media were determined at four production sites, one user site, and a background site. The ambient air level was determined on-site by direct injection of the ambient air into a gas chromatograph followed by detection and quantification with an electron capture detector. Water, soil, and sediment samples were returned to Battelle for analyses. For the analyses of water samples, MC was sparged from the water collected on a trap material using a commercial liquid sample concentrator. The trapped organic material was then backflushed onto a gas chromatograph column which was connected to an electron capture detector used to quantify the MC in the original sample. A similar technique was used for the quantification of MC in soil and sediment. results from the analyses and detailed descriptions of the sampling locations are given and keyed to site maps. Considerable variation was observed in the maximum downwind levels of MC at various production plants. Concentrations in ambient air ranged from less than 0.3 ppb to 155 ppb. Concentrations in surface water in vicinity of production and user plants was even more variable ranging from fractions of a ppb to over 16 ppm. Concentrations in soil and sediment range from the limits of detection to over 6.1 ppm.

17. KEY	WORDS AND DOCUMENT ANALYSIS	
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS c. COSATI Field/C	Group
Methylchloroform	Environmental monitoring	
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