SDMS Document
115269

OLEAN WELL FIELD
TOWN OF OLEAN, CATTARAUGUS COUNTY, NEW YORK
START THREE
ADMINISTRATIVE RECORD FILE
INDEX OF DOCUMENTS

1.0 FACTUAL INFORMATION/DATA

1.2 Site Investigations

P. 100001 - 100191 Report: Hydrogeologic Investigation at the AVX Facility, Olean, New York, prepared by Geraghty & Miller, Inc., prepared for AVX Corporation, January 1985.

1.3 POLREPs

100192 - 100194 Pollution Report Seven (7), Removal Action, Olean, New York, prepared by Mr. John Witkowski, OSC, Response and Prevention Branch, U.S. EPA, Region II, Recipients: See Distribution List, April 12, 1985.

1.7 Sampling Data/Data Summary Sheets/Chain of Custody Forms

P. 100195 - 100195 The actual document is available for review in the Olean Well Field Site File. Site Files are located at the United States Environmental Protection Agency, Region II Superfund Removal Records Center Edison, NJ.

3.0 PUBLIC PARTICIPATION

3.2 Community Relations Plan

P. 300001 - 300011 Plan: Community Relations Plan, Olean Wellfields, Olean, New York, prepared by Ms. Anne Tischbein, Weston/SPER Division, prepared for Mr. Robert Cobiella, OSC, Emergency and Remedial Response Division, Response and Prevention Branch, U.S. EPA, Region II Site Mitigation Section, undated.

HYDROGEOLOGIC INVESTIGATION

AT THE AVX FACILITY

OLEAN, NEW YORK

January 1985

Prepared for AVX Corporation

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Ground-Water Consultants
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HYDROGEOLOGIC INVESTIGATION AT THE AVX FACILITY, OLEAN, NEW YORK

INTRODUCTION

In August 1984 the AVX Corporation retained Geraghty & Miller, Inc. to implement a hydrogeologic investigation at the AVX facility in Olean, New York. This investigation was undertaken in response to an Administrative order (Index No. II-RCRA-3013-40202) from the U.S. Environmental Protection Agency's (USEPA) Region II office. The Administrative Order was signed on October 9, 1984 and the project was started soon after. The work plan on which this investigation is based was approved and incorporated into the Administrative Order.

The study was designed to assess hydrogeologic, ground-water quality, and soil-quality conditions in the vicinity of the AVX facility. This project is one of several investigations that have been carried out to determine the effect of volatile organic chemicals on The Olean "City Aquifer".

The investigation at the AVX facility included the following work:

- Installation of three 4-inch diameter monitoring wells. Two monitoring wells (AVX-1 and AVX-2) were screened in the City Aquifer, and are located east and south of the existing AVX production well. The third monitoring well (AVX-3), installed adjacent to AVX-1, was screened in a shallow water-table zone. Soil samples were collected during the drilling of the deep wells and analyzed for volatile organic compounds. Water samples were collected from all three monitoring wells and from the AVX production well and were also analyzed for volatile organic compounds. The locations of these wells are shown on Figure 1.

- Surface soil-sampling program. Soil samples were collected to a depth of 18 inches below ground surface at one hundred locations in the southern portion of the AVX facility and analyzed for volatile organic compounds.

- Sampling and analysis of AVX and off-site monitoring wells for landfill leachate parameters. Part of the property now owned by AVX was used as a municipal landfill prior to 1960. The

approximate extent of this former landfill is shown on Figure 1. In order to evaluate possible impacts on ground-water quality from former activities at this landfill, water samples from two of the three AVX monitoring wells and from eight off-site monitoring wells installed by others during previous investigations were analyzed for leachate indicators.

- Recovery test of the AVX production well. The AVX production well was shut down for 48 hours and water levels were measured in all wells at the AVX facility and in several off-site wells. The recovery test data were used to calculate hydraulic characteristics of the City Aquifer and to evaluate the effects of the AVX production well pumpage on water levels in the City Aquifer.

FIELD PROGRAM

The field program was carried out in accordance with the final Work Plan and the Quality Assurance/Quality Control (QA/QC) Document prepared by Geraghty & Miller, Inc. and approved by the USEPA. The QA/QC Document is appended to this report (Appendix A).

Monitoring Well Drilling and Installation

Between October 22 and October 27, 1984, three monitoring wells were installed by Buffalo Drilling Company, Inc. of Kenmore, New York, under the direction of Geraghty & Miller, Inc. The wells were drilled by the hollow-stem auger method using augers with an inside diameter of 6.25 inches and an outside diameter of approximately 10 inches. The monitoring well locations are shown on Figures 1 and 2.

Split-spoon samples were collected at five-foot intervals during the drilling of the two deep monitoring wells (AVX-1 and AVX-2). The samples were examined and described by the Geraghty & Miller, Inc. field hydrogeologist. The intervals between split-spoon samples were logged on the basis of drill cuttings and drilling characteristics. The split-spoon samples were subsequently examined by another Geraghty & Miller, Inc. hydrogeologist

to verify the field interpretations of the field hydrogeologist. The geologic logs for the deep AVX monitoring wells are presented in Appendix B.[1]

The four-inch diameter monitoring wells are constructed of 4-inch diameter black steel pipe and stainless steel screen. During well installation the casing and screen assembly was installed inside the hollow-stem augers. The lowermost section of the black steel pipe was threaded and screwed into the 10-foot section of stainless steel screen (0.010 inch slot). Subsequent lengths of steel pipe were welded together. Cape May No. 2 gravel was poured down the inside of the augers as the augers were extracted from the bore hole. This procedure was repeated until the annular space around the screen was filled with gravel to a level approximately two to four feet above the screen. The augers were then pulled back an additional two feet and bentonite pellets were poured down the inside of the augers to create a seal above the gravel pack. After the bentonite pellets were emplaced, the augers were removed from the borehole.[2]

1. The geologic materials penetrated in drilling AVX-3 are those described for the upper 12 feet of AVX-1 since these wells are approximately 10 feet apart.

2. The bentonite seal was emplaced in the annulus in AVX-3, as the top of the gravel pack is only 0.5 feet below land surface. The cement grout used to fix the protective pipe in place serves as a surface seal for this well.

The remainder of the annular space was then filled with a grout composed of approximately five parts Portland cement to one part bentonite. Table 1 summarizes the construction details of the completed monitoring wells and construction diagrams for each well are presented in Appendix B.

The three monitoring wells were developed from October 30 to November 2. Development of the wells was accomplished by pumping and agitation with compressed air as called for in the QA/QC Plan. The wells were pumped until they produced water that was relatively clear and free of sediment. Water levels were measured in the AVX monitoring and production wells during the field program and are summarized in Table 2.

A six-inch diameter steel protective pipe with a covering plate was cemented in place over each well. Each protective pipe has two tabs sticking up through the plate; padlocks were placed through holes in these tabs to protect the wells. Keys to the locks were sent to EPA and DEC. The well number and screened interval were stamped on the inside cover of each well. A triangular array of 2-inch diameter G.I. pipe was also installed around each well for additional protection. The elevation of the top of each 4-inch monitoring well was determined by Michael Canada, a surveyor licensed in the State of New York. The surveyor also determined the elevation of a hole that was cut into the

TABLE 1. Construction Details for AVX Production Well and Monitoring Wells

WELL	DATE INSTALLED	CASING & SCREEN DIAMETER (Inches)	Feet Below Land Surface: TOTAL DEPTH OF BORING	SCREENED INTERVAL	SCREEN SLOT SIZE (Inches)	ARTIFICIAL GRAVEL PACK	BENTONITE SEAL	CEMENT GROUT [1]	HEIGHT OF MEASUREMENT POINT (STICK-UP) ABOVE LAND SURFACE (feet)	Feet Above Mean Sea Lev: ELEVATION OF MEASUREMENT POINT (TOP OF 4" CASING)	ELEVATIO OF GROUN SURFACE BASE OF W
AVX-1	10/24/84	4	79.0	68.55 - 78.77	0.010	65.0 - 78.77	62.0 - 65.0	0 - 54.0 [2]	2.8	1,440.49	1,437.7
AVX-2	10/27/84	4	84.0	70.0 - 80.0	0.010	66.0 - 80.5	63.0 - 66.0	0 - 63.0	3.6	1,437.38	1,433.8
AVX-3	10/25/84	4	15.0	1.5 - 11.7	0.010	0.5 - 12.0	None [3]	0 - 0.5	3.3	1,439.81	1,436.5
AVX-PW	1959	12	76.8	61.0 - 65.0 65.0 - 67.0 67.0 - 72.0 72.0 - 74.0 74.0 - 76.0	0.010 [4] 0.040 0.060 0.035 0.016	Natural	?	?	2.9	1,442.29 [5]	1,439.4

1) 5:1 Mixture of Portland/Bentonite.

2) Formation collapse from 54' to 62' below land surface.

3) A bentonite seal was not emplaced due to proximity of gravel pack to the land surface (0.5 ft. BLS). Some space was needed to allow for protective pipe to be cemented in place. The grout mixture used to cement the protective pipe in place serves to seal the top of the annulus.

4) Screen slot size information provided by well driller (W.E. Lanphere, Shinglehouse, PA).

5) Measuring point on production well is a hole cut into the pump base.

100011

TABLE 2. Water Levels in AVX Production Well and Monitoring Wells.

WELL	ELEVATION OF MEASURING POINT (ft, msl)	DEPTH TO WATER (ft)	WATER LEVEL ELEVATION (ft, msl)	DATE	COMMENTS
AVX-PW	1442.29	32.56	1409.73	11/07/84	Pumping
		36.17	1406.12	11/09/84	Pumping
		31.16	1411.13	11/11/84	After 48 hrs of recovery
		36.15	1406.14	11/12/84	After 11 hrs of pumping
		35.85	1406.44	11/13/84	Pumping
AVX-1	1440.49	29.27	1411.22	11/07/84	AVX-PW pumping
		29.05	1411.44	11/08/84	AVX-PW pumping
		29.85	1410.64	11/09/84	AVX-PW pumping
		28.52	1411.97	11/11/84	AVX-PW off 48 hr
		29.77	1410.72	11/12/84	AVX-PW pumping 11 hrs
		29.78	1410.71	11/13/84	AVX-PW pumping
AVX-2	1437.38	26.80	1410.58	11/07/84	AVX-PW pumping
		26.97	1410.41	11/08/84	AVX-PW pumping
		27.19	1410.19	11/09/84	AVX-PW pumping
		26.15	1411.23	11/11/84	AVX-PW off 48 hr
		27.05	1410.33	11/12/84	AVX-PW pumping 11 hrs
		27.15	1410.23	11/13/84	AVX-PW pumping
AVX-3	1439.81	5.07	1434.74	11/07/84	
		5.50	1434.31	11/08/84	
		5.37	1434.44	11/09/84	
		3.40	1436.41	11/10/84	Response to rainfall event
		3.68	1436.13	11/11/84	
		3.79	1436.02	11/12/84	
		4.05	1435.76	11/13/84	

pump base of the AVX production well through which water levels were measured during the recovery test.

Soil and Water Samples from the AVX Monitoring Wells

As specified in the EPA Work Plan, portions of each split-spoon sample collected from the boreholes of monitoring wells AVX-1 and AVX-2 were transferred to sample bottles for analysis of volatile organic compounds (VOCs). The samples were collected and transferred in accordance with the relevant protocol specified in Appendix A, and sent to Cambridge Analytical Associates (CAA) of Boston, Massachussetts for the analysis of VOCs by EPA Method 601.

Duplicate samples were collected from the split-spoon for each five-foot interval and labeled "A" and "B". The three samples collected over a fifteen-foot interval were placed together in a plastic zip-lock bag for shipment. The three "A" samples, collected over each fifteen-foot interval, were composited in the laboratory prior to analysis. As specified in the Work Plan, one "B" sample group, corresponding to the "A" sample group with the highest combined concentration of VOCs, was analyzed using EPA Method 624.

Water samples were collected from the three AVX monitoring wells and the AVX production well on November 8, 1984 and were analyzed for VOCs by EPA Method 624. The

wells were sampled in accordance with the pertinent protocol in Appendix A. Replicate samples, labeled MW-4 and MW-5, were collected from monitoring well AVX-1 and the AVX production well, respectively.

The results of all of the soil and water analyses are discussed in a later section of this report.

Surface Soil Samples

Samples of the upper soils were collected at one hundred locations in the undeveloped, southern portion of the AVX property. A grid measuring 1,000 feet in an east-west direction and 200 feet in a north-south direction was established. This grid was divided into 20 square subgrids measuring 100 feet on a side. The configuration of the soil sampling grid is shown on Figure 2.

Soil samples were collected at five locations within each subgrid. These locations were approximately one quarter of the distance from each corner on the diagonal and at the intersection of the two diagonals of each subgrid. At each location a hole 12 to 18 inches deep was made with a hand auger and two soil samples were collected from the bottom of each hole in accordance with the pertinent protocol in Appendix A.

Duplicate samples collected at each sampling location were labeled "C" and "D". The five "C" samples within each subgrid were composited in the laboratory and analyzed for VOCs by EPA Method 601. Two "D" samples, corresponding to the two groups of "C" samples with the highest combined concentration of VOCs, were composited in the laboratory and analyzed by EPA Method 624.

Water Samples from Off-Site Monitoring Wells

In an effort to evaluate the potential impacts of the former municipal landfill on ground-water quality, water samples were collected on October 27 and 28, 1984 from eight monitoring wells installed by others during previous investigations of the City Aquifer. The off-site wells sampled were D&M(Dames & Moore)-7, D&M-7A, D&M-9, D&M-9A, D&M-10, D&M-10A, D&M-10B, and NUS-1. Samples from monitoring wells AVX-1 and AVX-3 were also collected for the analysis of landfill indicator parameters on November 8, 1984.

These water samples were collected in accordance with the relevant protocol in Appendix A. The samples were analyzed for pH, specific conductance, alkalinity, ammonia, chloride, sulfate, potassium, and iron. In addition, the samples from the off-site wells were analyzed for VOCs using EPA Method 624.

GROUND-WATER AND SOIL QUALITY

Ground-Water Analyses

Volatile Organic Compounds: Water samples were collected from eight off-site monitoring wells (D&M-7, D&M-7A, D&M-9, D&M-9A, D&M-10, D&M-10A, D&M-10B, and NUS-1) on October 27 and 28, 1984 and from the AVX monitoring wells and the AVX production well (AVX-PW) on November 8, 1984. These samples, along with a trip blank, were sent to CAA for VOC analysis. The results of this work are summarized in Table 3 and the complete laboratory report is provided in Appendix C.

As part of the QA/QC plan, replicate water samples were collected from AVX-1 (labeled MW-4) and AVX-PW (labeled MW-5) and were analyzed for VOCs utilizing EPA Method 624. Neither of the two samples collected from AVX-1 had detectable amounts of any of the VOCs tested for, and there was excellent agreement between the analytical results (51 vs. 52 ppb of trichloroethylene) for the two samples from the AVX production well. In order to evaluate the potential for contamination of the samples during shipment, an unopened trip blank was packaged with the water samples and analyzed for the same parameters. No VOCs were detected in the trip blank. In addition to analyzing for the volatile organic compounds listed in EPA Method 624, all of the water

samples were analyzed for other major volatile organic peaks. No other major peaks were identified in any of the water samples.

Inorganic Parameters: Water samples collected from the eight off-site wells listed above and AVX-1 and AVX-3 were analyzed for inorganic parameters that might be indicative of landfill leachate. The results of the inorganic analysis are summarized in Table 4. The complete laboratory report is included in Appendix C.

Soil Analyses

Monitoring Well Soil Samples: As discussed earlier, portions of the split-spoon samples from the boreholes of AVX-1 and AVX-2 were transferred to 40 ml vials and sent to CAA for VOC analysis. Duplicate samples were collected and labeled "A" or "B". The "A" samples from each 15-foot interval were composited in the laboratory and analyzed for VOCs by EPA Method 601. The results of this analysis are summarized in Table 5. The complete laboratory report is included in Appendix C.

As specified in the Work Plan, one "B" sample group, corresponding to the "A" sample group with the highest combined concentration of VOCs, was to be composited in the laboratory and analyzed by EPA Method 624. The "B" samples

for the interval of 0 to 15 ft from AVX-1 were selected for the EPA Method 624 analysis. The results of this analysis are presented in Table 5 and the complete laboratory report is included in Appendix C.

Surface-Sampling Grid: Soil samples were collected at a depth of 12 to 18 inches in 20 subgrids measuring 100 feet by 100 feet and sent to CAA for VOC analysis. The locations of the grids are shown on Figure 2. As discussed in an earlier section of this report, two samples were collected at five discrete locations within each subgrid. The two samples were labeled "C" and "D". The five "C" samples from each subgrid were composited in the laboratory and analyzed by EPA Method 601. The concentrations of VOCs detected in these surface soil samples are summarized in Table 6. Only those subgrids in which VOCs were detected are listed. Samples from subgrids not listed in Table 6 had no detectable VOC concentrations. The complete laboratory report is included in Appendix C.

As specified in the Work Plan, the two "D" sample groups corresponding with the two "C" sample groups having the highest combined concentrations of VOCs were composited in the laboratory and analyzed using EPA Method 624. The results of this EPA Method 624 analysis are summarized in Table 6. The complete laboratory report is included in Appendix C.

REVIEW OF SELECTED REPORTS FROM PREVIOUS INVESTIGATIONS

Aerial Photograph Analysis

Aerial photographs of the AVX/former landfill site and surrounding areas taken in 1956, 1966, and 1980 were ordered by Geraghty & Miller, Inc. from the U.S. Department of Agriculture's Aerial Photography Field Office in Salt Lake City, Utah on October 2, 1984. Due to backlogs in processing requests at this agency, the photographs had not been received as of January 14, 1984.

During the field investigation at the AVX site, EPA representatives made available for inspection aerial photographs taken of the AVX area in 1955 (believed to be the first aerial photograph of this site), 1960, and 1966. The 1955 photograph shows that the landfill was active at that time. The extent of the landfill shown on Figure 1 was determined from this photograph. By 1960 the landfill was no longer active and was completely covered by vegetation. It is not possible to determine from the 1955 photograph what type of refuse was disposed of in the landfill.

The aerial photographs indicate an excavated area 300 to 600 feet east of the intersection of Seneca Avenue and

Dugan Road. This area is not owned by AVX. The photographs taken in 1955 and 1960 show pools of standing liquid within this excavated area.

Review of the Technos Geophysical Study

Technos, Inc. carried out a geophysical investigation in the East Olean area. The study consisted of shallow high resolution electromagnetic profile mapping and a high density resistivity sounding program. A preliminary report, dated March 1983, was submitted by Technos, Inc. to Engineering Science, Inc. This report was reviewed by Geraghty & Miller, Inc. for any data concerning the AVX site and the former landfill site.

The electromagnetic (EM) mapping measured the bulk electrical conductivity of the upper twenty feet of soil. According to Technos, the mapping revealed an increase in EM values from the McGraw Edison site to the west. This may indicate a decreasing permeability of the shallow sediments in this direction. An area of anomalously high EM values between Butler Avenue and AVX corresponds with the area occupied by the old landfill. The EM mapping revealed little or no evidence of a conductive plume near the edges of the landfill. However, it should be noted that the area between the railroad tracks and East State Street was not investigated due to the concentration of buildings, buried

utility lines, and other obstacles.

The resistivity soundings, as interpreted by Technos, Inc., indicate that, in some areas of Olean, the subsurface materials are composed of two permeable sand and gravel zones separated by a clay layer of varying thickness. The soundings made in the vicinity of AVX and the old landfill indicate that the upper sand and gravel zones are missing in this area and that the AVX/landfill area is underlain directly by substantial thicknesses of clay and till. Technos, Inc. further states that "both upper and lower (sand and gravel) units are thin or absent in the area around the AVX and municipal landfill sites". Geraghty & Miller, Inc.'s review of the Technos study indicates that the resistivity soundings may be less reliable with increased depth, as the geologic logs of the deep AVX monitoring wells and CDM(Camp, Dresser & McKee)-14 indicate a deep sand and gravel zone, the top of which was encountered at depths from 46 to 69 feet in this area.

Previously Collected Geologic and Water-Quality Data

Geologic logs for CDM-14 and CDM-9 (located halfway between D&M 10 and East State Street) were compared to the logs of the AVX monitoring wells. The logs of CDM-14 and AVX-2 are very similar in that the deep sand and gravel zone is split by a 20-foot thick zone of silty till

(56-76 feet deep) at CDM-14 and 13 feet thick (61 to 74 feet deep) at AVX-2. The upper part of the deep sand and gravel zone is apparently absent at AVX-1.

The log of CDM-9 indicates that the deep sand and gravel zone thickens to the south; it occurs from a depth of 46 to at least 86 feet below land surface and is not split by a till layer such as was found at CDM-14 and AVX-2. An upper sand and gravel zone was encountered at CDM-9 at a depth of 36 to 46 feet. As the logs of the Dames & Moore wells (referred to in the Work Plan as CW-7, CW-9, CW-10 and BP-1) were not made available by USEPA or the N.Y. Department of Environmental Conservation (NYDEC), these logs could not be used to aid in the interpretation of the AVX field data.

Water-quality data for the CDM monitoring wells and 36 domestic, industrial, and public-supply wells were reviewed as part of this study. Results of the water-quality analyses from the Dames & Moore wells (referred to in the Work Plan as CW-7, CW-9, DW-10 and BP-1) were not available at the time this report was prepared. The available water-quality data must be viewed in light of the fact that the various monitoring and supply wells are screened at several different intervals, and few data are available concerning construction details of many domestic wells.

The available water-quality results indicate that trichloroethylene is the major contaminant in both the City Aquifer and shallower aquifer units. The area in which detectable amounts of trichloroethylene were noted extends from Dugan Avenue to beyond Clark Street. In general, the area in which concentrations of trichloroethylene exceeded 1,000 ppb occupies a band extending from Dugan Avenue (approximately 1,000 feet south of Seneca Avenue) to Public-Supply Wells 37M and 38M.

RECOVERY TEST

A controlled aquifer recovery test was carried out on the AVX production well (AVX-PW) between 9:00 pm on November 9, 1984 and 9:00 pm on November 11, 1984 after turning off the well. Automatic water-level recorders were installed on AVX-1 and AVX-2, and water levels were also measured in AVX-3. Periodic water-level measurements were made in eight off-site wells (D&M-19, D&M-19A, CDM-14, D&M-9, D&M-9A, D&M-10, D&M-10A and D&M-10B) in order to measure the lateral extent of aquifer response.

Pre-Test Measurements

Water levels were measured three or four times in AVX-PW, AVX-1, AVX-2, and AVX-3 in the eight-hour period prior to the beginning of the recovery test. During this period there were minor water-level fluctuations in these wells, the maximum being 0.04 feet in AVX-PW over the eight-hour period.

Water levels were measured once in the eight off-site wells prior to shutdown. These measurements were made between 2:00 pm and 4:00 pm on November 9, 1984, five to seven hours before turning off AVX-PW.

Recovery Test Procedures

AVX-PW was shut off at 9:00 pm on November 9, 1984. Water level measurements were taken every 30 seconds in AVX-PW for the first ten minutes after shutdown and at increasing intervals thereafter. Water levels were measured with an M-scope (a battery-operated electric sensing device) through a hole cut into the pump base.

Automatic water-level recorders were installed on AVX-1 and AVX-2. The recorders were set up so that the pen moved across the length of the drum in 12 hours, and one complete turn of the drum was equivalent to a change in water level of 5.0 feet. The depth to water from the top of the well casing was measured with a chalked steel tape prior to the test and noted on the recorded chart. As a check on the recorder clock accuracy, the recorders were "ticked" approximately every two hours and the actual time noted. The depth to water was measured with a chalked steel tape approximately every twelve hours, when the pen on the recorder was reset.

Water levels were measured in the eight off-site wells two to three hours after AVX-PW was shut off. The water levels in these wells were measured three times on November 10, and once on November 11 while AVX-PW was shut down. A USEPA geologist who was on-site during most of the field

work, measured water levels in other off-site wells periodically during both the recovery and pumping periods. The field data sheets of water-level measurements in AVX-PW, AVX-3, the eight off-site wells, and the recorder charts for AVX-1 and AVX-2 are included in Appendix D.

A recording barometer was set up at the AVX site eight hours prior to the start of the recovery test and was monitored during the test to detect relative changes in barometric pressure. The barometer pressure dropped approximately 0.5 inch from 1 pm on November 9 to 7:30 am on November 12. The chart from the recording barometer is included in Appendix D.

Pump Start-Up and Post-Test Measurements

AVX-PW was started up at 9:00 pm on November 11, 1984, after being shut down for 48 hours. The recorders installed on AVX-1 and AVX-2 were recalibrated approximately two and one half hours prior to start-up. Water levels in AVX-PW were measured on the same logarithmic time scale employed in the recovery test for three-hours after the well was turned on, and water levels in AVX-3 were measured five times during this three hour interval.

Water levels in the eight off-site wells were measured approximately two hours after AVX-PW was turned back on and remeasured the following morning, approximately 12 hours after start-up.

The water level in AVX-PW was measured at 8:00 am on November 12, approximately 11 hours after start-up, and the pumping level was almost identical to the pumping level immediately prior to the recovery test on November 9. Soon after this measurement on November 12, an attempt was made to determine the pumping rate. The discharge of AVX-PW cannot be measured directly, as the well pumps water into the plant where it is used for cooling in various processes. After going through the plant processes, the water from AVX-PW is discharged through a corrugated metal pipe outside (south of) the south fence. The open end of this corrugated pipe is only two feet above the ground, which places a restriction on the size of a container that can be placed underneath it.

A seven-gallon bucket and a stopwatch were used to measure the flow from the corrugated pipe. A total of six measurements were made; it took an average of 1.1 seconds to fill the seven-gallon bucket. This is equivalent to a flow rate of 380 gallons per minute (gpm). AVX personnel stated that all of the water discharged from the corrugated pipe originated from the production well.

Recovery Test Analysis

Recovery Data: The water-level information collected from AVX-PW, AVX-1, and AVX-2 during the 48-hour recovery test was used to construct semi-logarithmic plots of water-level recovery versus time. These plots are shown on Figures 3, 4, and 5. The water level recovery at a particular instant was calculated by subtracting the depth to water at that instant from the depth to water at the start of the test, under pumping conditions. The assumption was made that water levels in these three wells had stabilized with respect to pumpage of AVX-PW prior to the beginning of the recovery test.

Drawdown Data: The water-level information collected from AVX-PW, AVX-1, and AVX-2 after AVX-PW was turned back on was similarly used to construct semi-logarithmic plots of water-level drawdown versus time. These plots are shown on Figures 6, 7, and 8.

Aquifer Parameters

Aquifer hydraulic characteristics were calculated using the data collected during the recovery and drawdown periods. The recovery data were plotted (Figures 3 through 5) on semi-logarithmic graph paper with water-level recovery on the arithmetic scale and time on the logarithmic scale. The

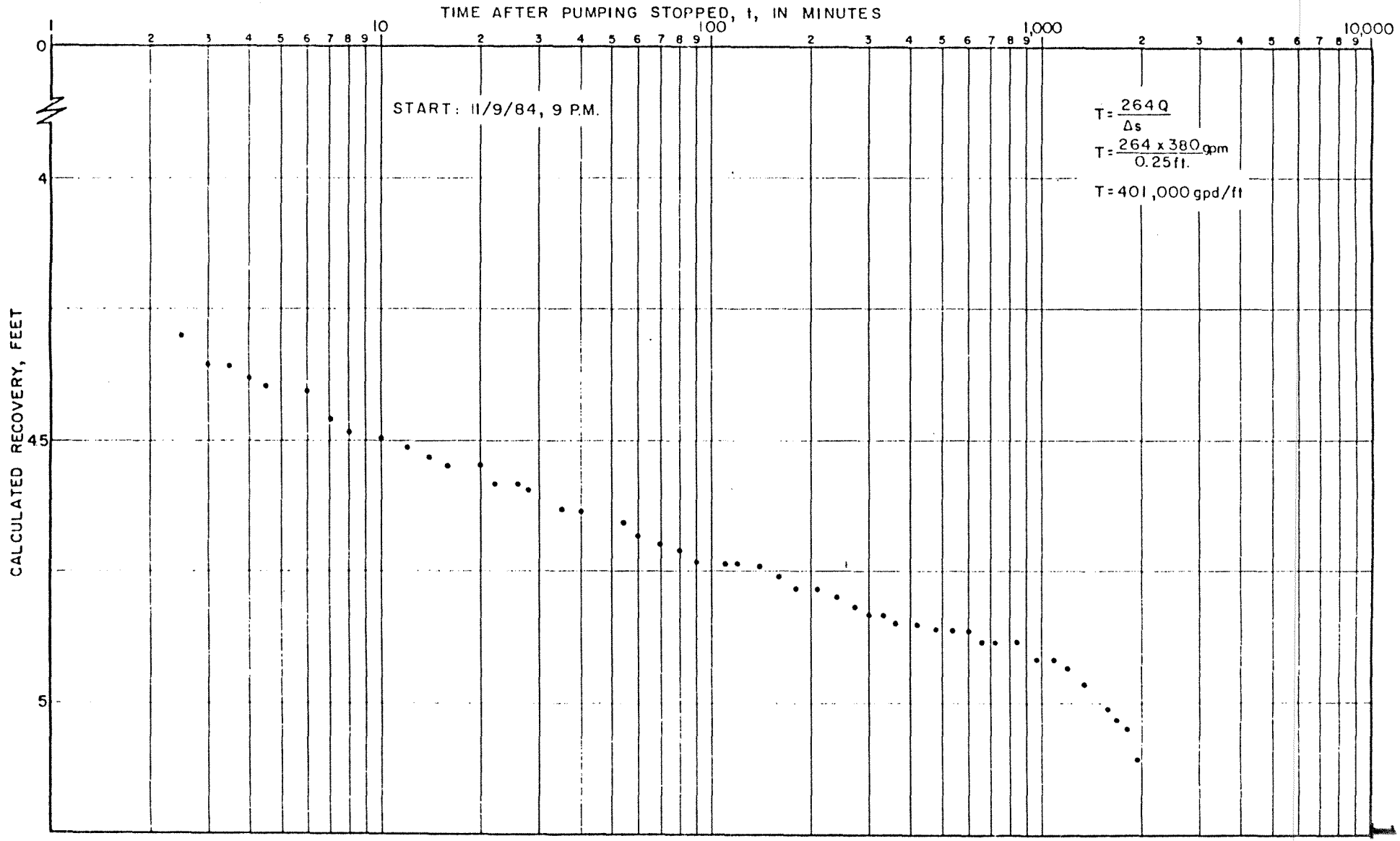

FIGURE 3 - SEMI-LOGARITHMIC PLOT OF RECOVERY VERSUS TIME FOR AVX-PW

100029

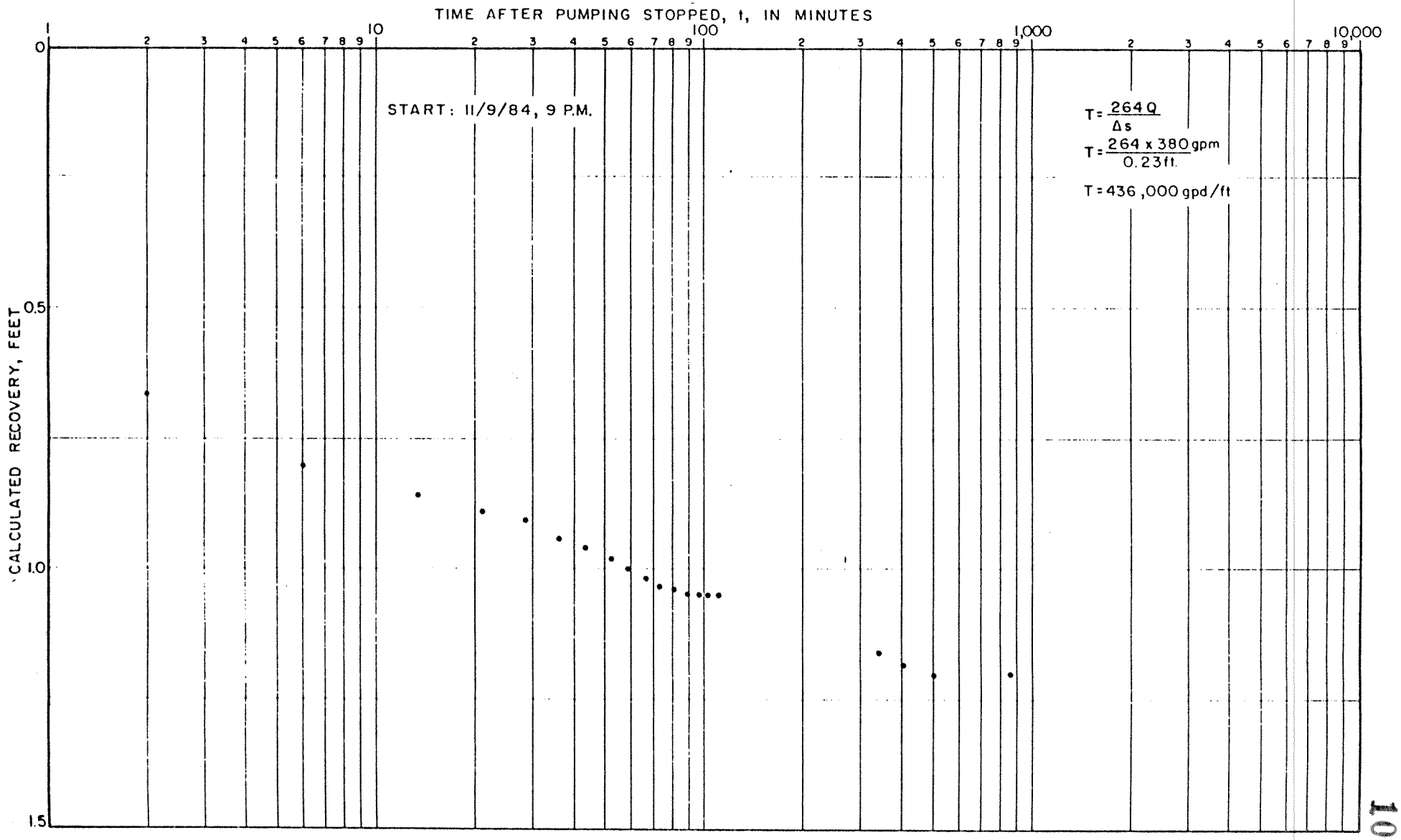

FIGURE 4 - SEMI-LOGARITHMIC PLOT OF RECOVERY VERSUS TIME FOR AVX-1

100030

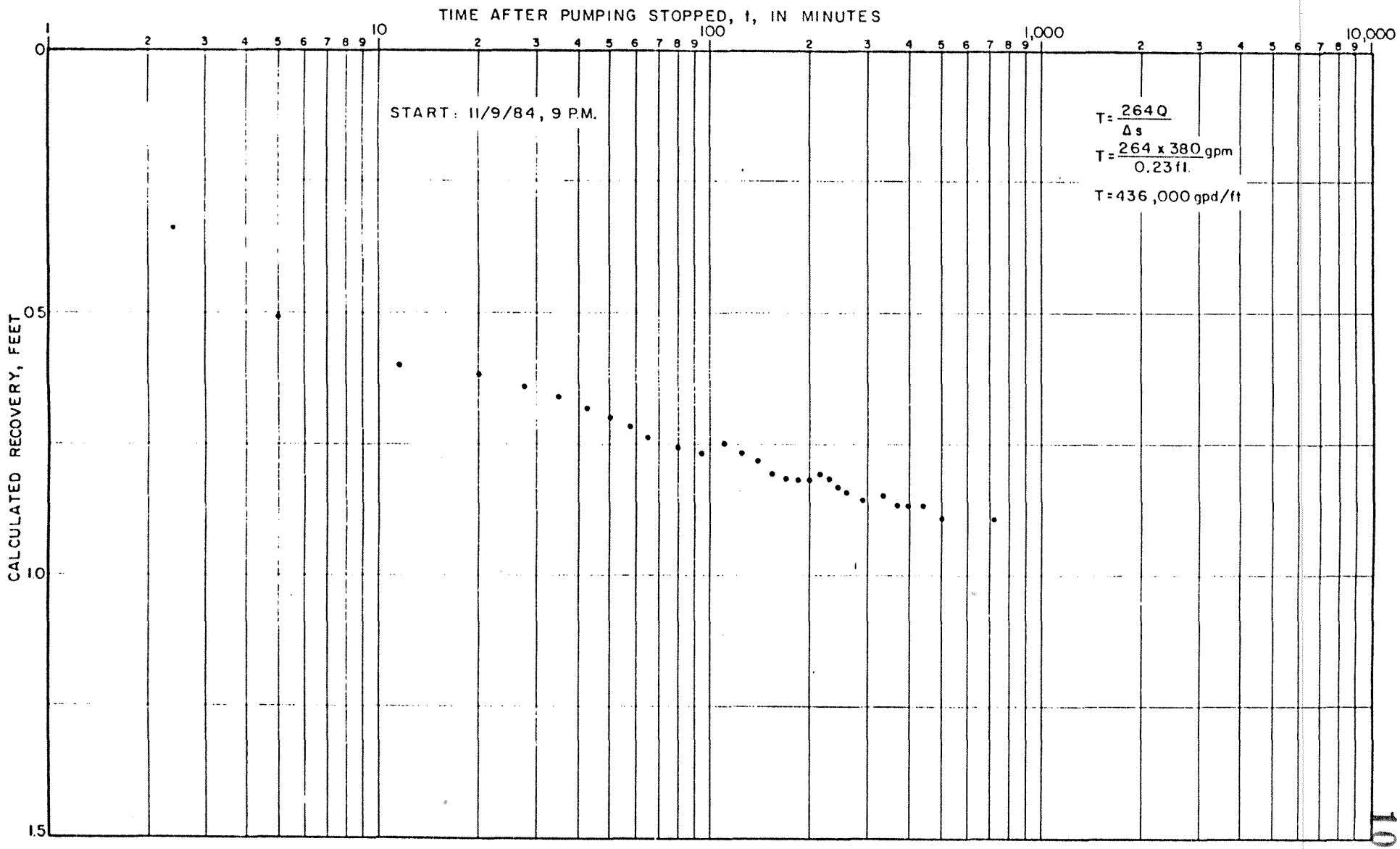

FIGURE 5 - SEMI-LOGARITHMIC PLOT OF RECOVERY VERSUS TIME FOR AVX-2

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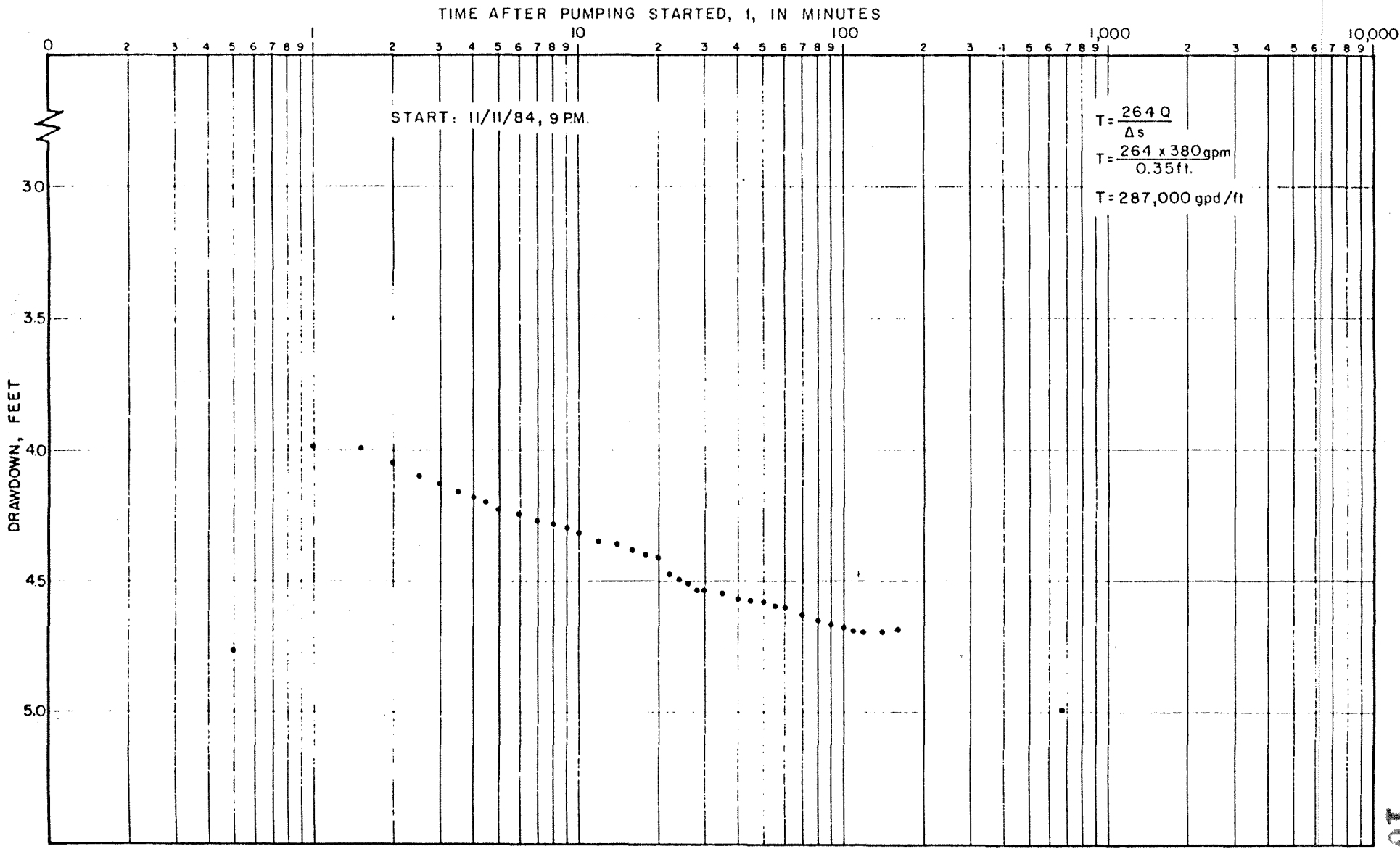

FIGURE 6 - SEMI-LOGARITHMIC PLOT OF DRAWDOWN VERSUS TIME FOR AVX-PW

100032

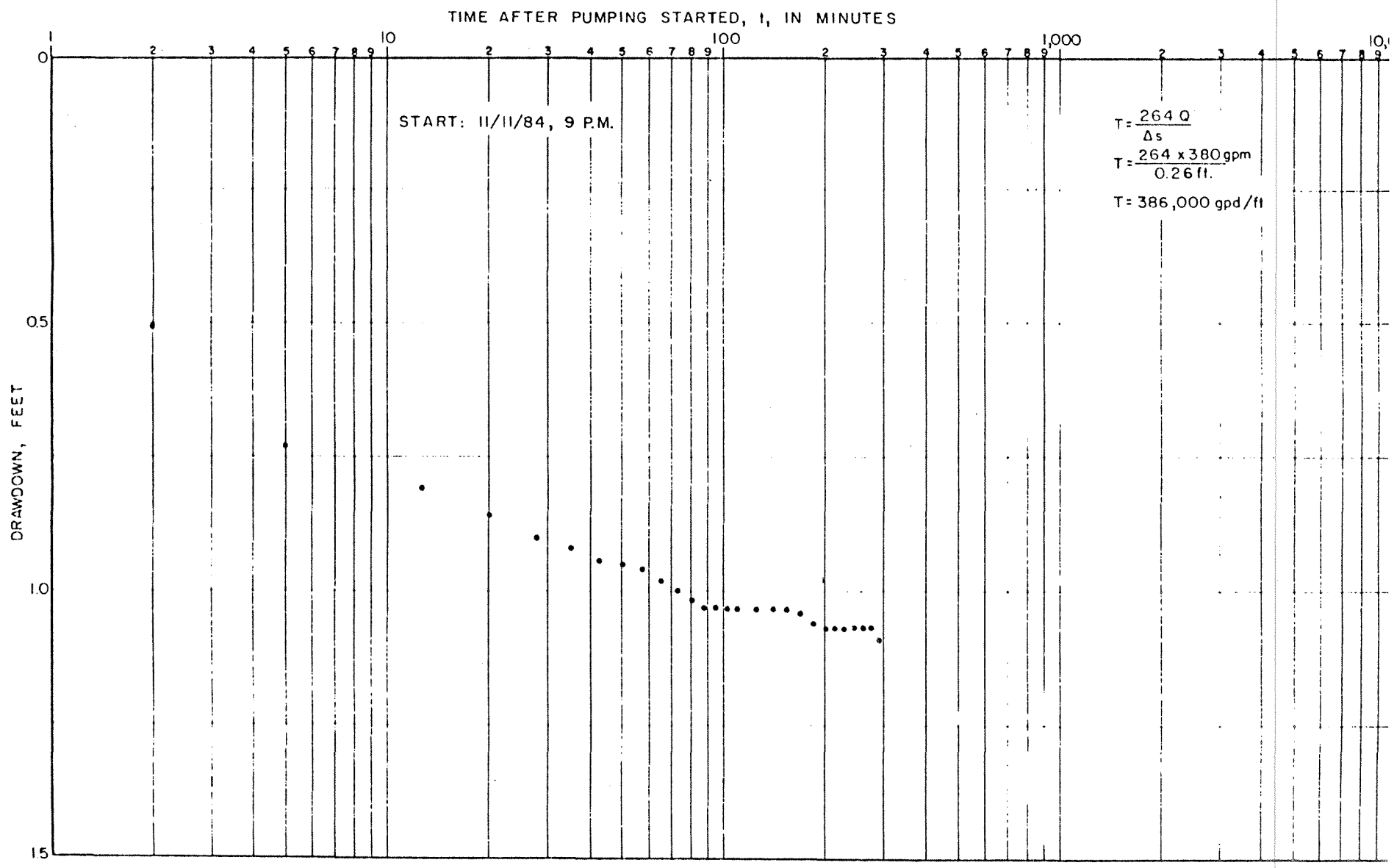

FIGURE 7 - SEMI-LOGARITHMIC PLOT OF DRAWDOWN VERSUS TIME FOR AVX-1

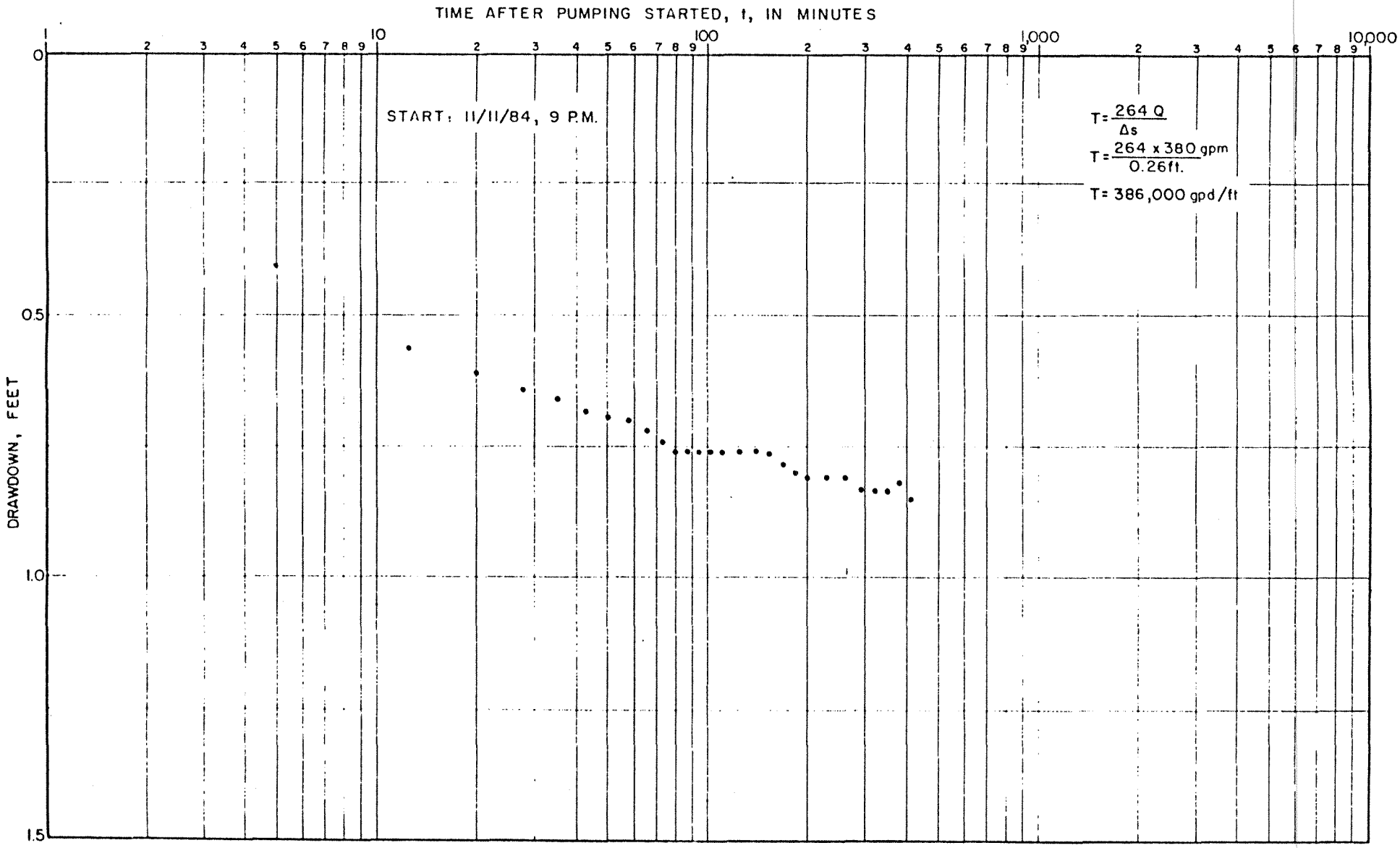

FIGURE 8 - SEMI-LOGARITHMIC PLOT OF DRAWDOWN VERSUS TIME FOR AVX-2

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100035

calculated values of aquifer transmissivity are shown on these figures.

The water-level drawdown data were similarly plotted on semi-logarithmic graph paper (Figures 6 through 8) and the Cooper-Jacob non-equilibrium method was used to calculate aquifer transmissivity and storativity. The calculated values of transmissivity and storativity are presented in Table 7.

Response of AVX-3 (Water Table Well)

The water level in the well screened in the shallow water-table zone (MW-3) did not show an immediate response to the shut-down of AVX-PW at 9:00 pm on November 9, 1984. The water level in this well, however, did rise rapidly from 11:45 pm on November 9 until 9:15 am the following morning. This rise of approximately 2.0 feet in water level is interpreted as being in direct response to a rainfall event. Rain, intermittently heavy, fell from 11:00 pm on November 9 to 7:30 am on November 10.

After rising in response to this rainfall event, the water level in MW-3 slowly dropped from 9:15 am on November 10 until 3:00 am on November 11. Another rainfall event, from 2:00 am to 8:00 am on November 11, caused the water level in MW-3 to rise again, this time by only 0.25 foot,

TABLE 7. Summary of Aquifer Hydraulic Characteristics (Calculated from Recovery and Pumping Test Data).

WELL	TYPE OF DATA ANALYZED	TRANSMISSIVITY (gpd/ft)	STORATIVITY (dimensionless)
AVX-PW	Recovery	401,000	
AVX-PW	Drawdown	287,000	
AVX-1	Recovery	436,000	1.6×10^{-5}
AVX-1	Drawdown	386,000	4.6×10^{-5}
AVX-2	Recovery	436,000	5.1×10^{-5}
AVX-2	Drawdown	386,000	1.5×10^{-4}

from 3:00 am to 1:00 pm on November 11. At its highest, the water level in MW-3 was 0.5 foot below ground surface.

Response of Off-Site Wells

Water levels in seven of the eight off-site wells responded rapidly to the turning off and starting up of AVX-PW. The only well that did not respond was D&M-10B, which is screened in the shallow water-table zone. The maximum water-level recovery in each well (after approximately 43 hours of recovery) and the maximum subsequent water-level drawdown (measured after approximately 13 hours of resumed pumping of AVX-PW) are summarized in Table 8.

Respectfully Submitted,

GERAGHTY & MILLER, INC.

Daniel A. Nachman,
Staff Scientist

Vincent W. Uhl, Jr.,
Senior Scientist

William J. Seevers,
Vice President

TABLE 8. Response of Off-Site Wells to Shut-Down and Subsequent Start-Up of AVX-PW.

WELL	APPROXIMATE DISTANCE FROM AVX-PW (ft)	INITIAL RECOVERY (ft) (AVX-PW Off 2 hours)	MAXIMUM RECOVERY (ft) (AVX-PW Off 43 hours)	MAXIMUM SUBSEQUENT DRAWDOWN (ft) (AVX-PW pumping 13 hours)
CDM-14	300	0.50	0.86	0.76
D&M-19	600	0.38	0.71	0.65
D&M-19A	600	0.22	0.58	0.54
D&M-9	700	0.35	0.60	0.46
D&M-9A	700	0.36	0.61	0.46
D&M-10	1000	0.33	0.60	0.43
D&M-10A	1000	0.34	0.60	0.46
D&M-10B	1000	0.01	0.01	0.06

I certify that I have personally examined and am familiar with the information submitted above this certification. Based upon my own knowlege and upon my inquiry of those individuals responsible for obtaining the information presented, the foregoing information is true, accurate and complete. I am aware that this information is being requested for the purpose of determining compliance with local, state and federal laws and may be submitted to appropriate governmental regulatory agencies for those purposes. I am aware that there are significant penalties for submitting false information to such agencies, including the possibility of fine and imprisonment.

V. W. Uhl

Vincent W. Uhl, Jr.,
Authorized Representative

January 1985

APPENDIX A

Quality Assurance/Quality Control Document

100041

QUALITY ASSURANCE/QUALITY CONTROL

DOCUMENT

AVX FACILITY
OLEAN, NEW YORK

Geraghty & Miller, Inc.
Ground-Water Consultants
7 Atlantic Street
Hackensack, New Jersey 07601

QUALITY ASSURANCE / QUALITY CONTROL DOCUMENT

1. Project Name: AVX, Olean, New York.
2. Project Requested By: U. S. Environmental Protection Agency.
3. Date of Request:
4. Date of Project Initiation: October 9, 1984
5. Project Officer: William J. Seevers
6. Quality Assurance Officer: Vincent W. Uhl, Jr.

7. Project Description

A. Objective, Scope, Design, and Rationale

Geraghty & Miller, Inc. has prepared this Quality Assurance/Quality Control document for the hydrogeologic investigation to be carried out at the AVX facility in Olean, New York. This work was mandated by a Consent Order issued by the U. S. Environmental Protection Agency. The project was initiated on October 9, 1984 with the approval of the revised Work Plan by the USEPA. William J. Seevers, Vice- President of the firm, will serve as Project Officer. The Quality Assurance Officer will be Vincent W. Uhl, Jr., Manager of the firm's New Jersey Office.

In general the investigation will consist of the following phases (refer to Appendices and Work Plan for details):

1) The installation of three monitoring wells at the AVX facility. Monitoring well AVX-1 will be located east of the AVX production well, between the production well and the former municipal landfill. Monitoring well AVX-2 will be drilled approximately 480 feet south of the north property line and approximately 420 feet east of the western property boundary. Monitoring wells AVX-1 and AVX-2 will be drilled to a depth of approximately 80 feet, the assumed depth of the AVX production well. Monitoring well AVX-3 will be drilled approximately 10 feet west of well AVX-1, and will be drilled to the lower part of the upper aquifer, or to a depth of at least 6 feet below the top of the water table if no confining layer is encountered. It is estimated that AVX-3 will have a final depth of approximately 40 feet. Soil samples from monitoring wells AVX-1 and AVX-2 will be composited and analyzed for chlorinated volatile organics.

2) Sampling of the AVX production well, the three monitoring wells, and other existing monitoring wells in the vicinity of the AVX facility. The wells at the

AVX facility will be sampled for volatile organics and landfill indicator parameters; some of the existing monitoring wells surrounding the AVX site will be sampled for landfill indicator parameters and volatile organics. (See appendix B for list of parameters).

3) Soil samples will be collected in the undeveloped area in the southern part of the AVX facility for analysis of chlorinated volatile organics. The soil samples will be collected from the upper 18 inches of soil in a grid extending one thousand feet in an east-west direction and two hundred feet in a north-south direction.

4) A water-level recovery test will be carried out at the AVX facility. The production well, which currently operates 24 hours a day, will be shut down for approximately 48 hours. Water levels will be measured in the production well, the AVX monitoring wells, and selected existing monitoring wells surrounding the AVX facility. Water levels in all of these wells will be measured periodically before and after the recovery test.

B. Data Usage

The design and rationale of the project was developed by EPA after discussion with both AVX and their consultants. In accordance with the EPA Work Plan, the data collected during this project will be submitted to EPA. It is understood that EPA will turn it over to consultants to the New York State Department of Environmental Conservation investigating ground water contamination in the vicinity of Olean, New York.

This Quality Assurance/Quality Control document has been prepared to ensure consistency, accuracy and reliability of the data collected during the field investigation. The Quality Assurance/Quality Control document consists of the following elements:

Appendix A - Specifications for the Installation of Monitoring Wells.

Appendix B - Ground-Water Sampling Protocol.

Appendix C - Soil Sampling Protocol for Drilled Samples.

Appendix D - Soil Sampling Protocol for Surface Samples.

Appendix E - Protocol for Water-Level Recovery Test.

Appendix F - Document and Data Management.

Appendix G - Cambridge Analytical Associates QA/QC Procedures.

8. Schedule of Tasks and Products

The field investigation and report preparation is expected to follow the schedule contained in Part VI of the EPA work plan. The actual dates during which field work is carried out will be recorded on appropriate field logs and other control paperwork. This control paperwork will be used to determine if the project is on schedule.

9. Project Organization and Responsibility

Project Officer - William J. Seevers - (516) 921-6060
Project Manager - Vincent W. Uhl, Jr. - (201) 646-1400
Sampling Operations - Daniel Nachman - (201) 646-1400
Sampling Q. C. - Daniel Nachman - (201) 646-1400

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Laboratory Analysis - Mr. Ed Lawler

Laboratory Q. C. Senior Analytical Chemist

Cambridge Analytical Assoc.

1106 Commonwealth Ave.

Boston, Mass. 02215

(617) 232-2207

Data Processing Activities - Daniel Nachman - (201) 646-1400

Data Processing Q. C. - Daniel Nachman

Data Quality Review - Vincent W. Uhl, Jr. - (201) 646-1400

Overall Q. A. - Vincent W. Uhl, Jr.

Overall Project Coordination - Vincent W. Uhl, Jr.

Drilling Contractor - Buffalo Drilling Co., Inc.

1965 Sheridan Drive

Kenmore, New York 14223

(716) 875-0906

10. Data Quality Requirements and Assessments

The validity of the ground-water quality data must be confirmed by quality-control measures instituted during sampling, sample handling, and analysis. Contradictory results are inevitable in a program of this size, and without check on the procedures, incomplete or incorrect conclusions may result.

Quality control begins with proper sampling procedures, which include specifications for well development, well purging on the day of sampling, methods of sample removal from the well (including acceptable materials for any equipment or supplies that are in contact with samples: pumps, tubing, bailers, etc.), sample processing (for example, filtration, preservation, bottle filling), and sample shipment (Appendix B). For all sampling activities, we will prepare these specifications, which will be in accord with technically sound standard practice (for example, EPA-600 4-82-029, "Handbook for Sampling and Sample Preservation of Water and Wastewater"), subject to revisions based on local regulatory or field requirements.

Samples taken for volatile organic analysis are the most fragile and the use of extra quality control samples is necessary to monitor sampling and laboratory performance. A trip blank (a vial filled in the laboratory with organic-free water that travels unopened to the site and back to the laboratory) should accompany each round and preferably each day's shipment of samples. Detection of contamination during shipment or in the laboratory often results from analysis of trip blanks. In addition, reproducibility of results can be estimated by splitting one well's sample in the field (field replicates) and sending the differently labeled samples to the laboratory. A comparison of field replicates can provide the

information needed to determine whether the ground-water quality in two wells is significantly different and whether ground-water quality is changing with time. In general, we recommend that 10 to 20 percent of wells be replicated (minimum of two wells replicated) with higher percentage of wells replicated in crucial areas or when particularly important decisions must be made. Our recommendation encompasses a compromise of data certainty requirements and cost for the general case based on our experience. We will adjust the extent of quality-control sampling to meet the needs of specific parts of the program as they evolve.

Laboratory quality control includes established routines for instrument standardization and data workup. Geraghty & Miller, Inc.'s geochemists will work closely with the laboratory to assure that samples are reaching the laboratory in proper condition and to solve analytical problems resulting from complex groundwater samples. In addition Geraghty & Miller, Inc. will assess the reported data for internal consistency and interpret the results in light of constituents expected from previous land use and from chemical and biochemical transformations that may have occurred in the aquifer.

11. Data Representativeness - The principal concern with respect to data representativeness relates to the representativeness of ground water samples collected during the sampling program. In this regard the wells will be pumped (purged) prior to sampling and at least 5 casing volumes will be removed prior to sample collection. Since the work plan dictates the location of the various soil samples, representativeness is not an issue.

12. Data Comparability - Since there will be only one sampling event as per the EPA work plan, this is not an issue or concern. However sampling methodology, analytical procedures, etc. are specified herein for the purpose of data comparison with other projects that have been carried out within the overall problem area.

13. Data Completeness - The QA/QC plan and work outlined herein relates specifically to a work plan mandated by EPA and we have within this QA/QC plan addressed pertinent issues related to the work plan.

14. Sampling Procedure

Refer to Appendices B, C, and D.

15 Sample Custody Procedures

Chain of custody documentation and preparation and handling of sample containers is provided in Appendix G.

16. Calibration Procedures and Preventive Maintenance

Field Equipment

Instrument

PH meter:	Clean and calibrate after each sample
Conductivity Meter:	Clean and calibrate after each sample

Calibration procedures with respect to laboratory equipment is outlined in Appendix G.

17. Documentation, Data Reduction, and Reporting

Refer to Appendix F.

18. Data Validation

Refer to Section 11 and Appendices B & G with respect to water sample collection and laboratory data validation.

In terms of field operations, transmittal of both soil and water samples will be field checked by two individuals. Geologic logs developed for the monitoring wells will be

independently checked by a Geraghty & Miller geologist not involved otherwise in the project. Pumping test data will be checked both in the field and office.

19. Performance and System Audits

Laboratory performance will be checked with Trip Blanks and Replicate Samples. The work will be conducted by a laboratory ascribing to EPA QA/QC requirements.

The drilling of monitoring wells will be audited by on-site geologists. Prior to the final selection of a drilling subcontractor, reference checks were made.

The pumping test and soil sampling will be audited and performed by on-site geologist(s).

20. Corrective Action - The work and data generated during the project will be continually reviewed by Vincent W. Uhl, Jr. and if any problems occur during the project, they will be addressed in a timely matter and an appropriate corrective action plan will be formulated.

APPENDIX A. MONITORING WELL SPECIFICATIONS

1. GENERAL

1.01 THE CONTRACT DOCUMENTS apply to this Section.

1.02 DESCRIPTION OF THE SYSTEM

A. THE MONITORING WELLS described herein are part of a program to monitor ground-water quality at the AVX facility in Olean, N.Y. It is proposed to drill three test borings and install three monitoring wells. The three test borings in which the monitoring wells will be installed will be used to obtain soil samples at five foot intervals.

1.03 DRILLING SITE

A. DRILLING will be carried out at the AVX plant in Olean, N.Y.

B. THE WELL locations will be shown to the driller by the Consultant.

1.04 LOCAL GEOLOGY

A. THE AVX SITE is reportedly underlain by a clayey and silty till with sand layers of varying thickness (50-60 feet thick). The till overlies an artesian zone of sand and gravel averaging 20 feet in thickness.

B. INFORMATION regarding subsurface conditions is intended to assist the Driller in preparing his bid. The Consultant does not guarantee its accuracy or that it is necessarily indicative of conditions to be encountered in drilling the wells. The Driller shall satisfy himself regarding all local conditions affecting this work by personal investigation and neither the information on local geology nor that derived from maps or drawings, nor from the Consultant or his agents or employees shall act to relieve the Driller of any responsibility hereunder or from fulfilling all of the terms and requirements and Specifications.

2. PRODUCTS

2.01 GRAVEL

A. THE GRAVEL shall be free of foreign material and water washed prior to emplacement.

B. THE GRAVEL shall be clean quartz gravel free of silt or clay.

C. THE SIZE of the gravel shall be determined by the Consultant prior to the startup of drilling.

2.02 BENTONITE PELLETS

A. BENTONITE PELLETS shall be not larger than 1/2 inch in diameter and must be dry prior to placement in the well annulus.

2.03 GROUT

A. GROUT shall consist of a mixture of 6 parts Portland Type 1 cement to 3 parts water to 1 part bentonite.

2.04 CASINGS

A. THE WELL CASINGS shall be 4-inch diameter black steel pipe having wall thickness of 1/4-inch (schedule 80). No grease shall be used when joining casing sections together.

2.05 SCREENS

A. THE WELL SCREEN shall be 4-inch diameter UOP Johnson wire-wound stainless steel screen, having openings of 0.010 inch.

3. EXECUTION

3.01 DRILLING METHOD

A. THE CONVENTIONAL hollow-stem-auger method of drilling using 6 1/4-inch ID augers shall be employed. However, when the Consultant determines that the hollow-stem-auger method is ineffective due to local geologic conditions, then the mud rotary method will be employed. Any drilling fluid utilized must be approved by AVX and EPA prior to its use. The diameter of the finished hole must be a minimum of 8 inches and a maximum of 12 inches.

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3.02 DRILLING

A. THREE MONITORING WELLS will be installed to the approximate depths specified below:

WELL NO.	APPROXIMATE DEPTH (ft)
MW - 1	80
MW - 2	80
MW - 3	15

3.03 FORMATION SAMPLES

A. SOIL SAMPLES will be taken during the drilling of each monitoring well at 5-foot intervals or at intervals specified by the Consultant. The driller will be required to use a standard 2-inch split spoon sampling device to obtain soil samples.

B. ACTUAL SAMPLING DEPTHS shall be determined from information derived during drilling operations and as directed by the Consultant.

3.04 SCREEN AND CASING

A. THE CASING AND SCREENS shall be installed to depths designated by the Consultant. Alternate depths suggested because casing cannot be set to depth, because of equipment problems, hole straightness problems, or for any other reason will not be given consideration. The Driller must insure that the hole stays open to the final depth during the installation of casing, screen, gravel and grout.

B. IT IS ESTIMATED that ten feet of screen will be installed in each of the monitoring wells.

3.05 SAND BACKFILLING AND CEMENTING, SCREEN AND CASING

A. AFTER SETTING THE SCREEN AND CASING to the bottom of the hole, gravel will be placed in the annulus between the auger flight and the casing/screen to 3 to 5 feet above the top of the screen in the wells. A three-foot thick layer of bentonite pellets will be placed over the sand backfill. The gravel will be emplaced by carefully pouring in by hand and washed down with water or by means of a one-inch diameter tremie pipe, if the method of drilling is changed to mud rotary.

B. CEMENT GROUT will be emplaced in the hole from the top of the bentonite seal to land surface. The cement will be mixed to a density approved by the Consultant and emplaced by means of a tremie pipe.

C. THE WELL shall be completed with a six-inch diameter, locking well head assembly set in a concrete pad. A protective triangular array made of 2-inch diameter G.I. pipe will be installed around each well head for additional protection.

3.06 DEVELOPING THE WELL

A. AFTER THE GROUT has set for 12 hours in the well annulus, the well shall be developed to the satisfaction of the Consultant. The well will be developed using compressed air and/or a suitable pump. The Driller may also be required to surge the well with a surge-block plunger of the appropriate diameter.

3.07 DECONTAMINATION OF EQUIPMENT

A. AFTER THE DRILLING, SAMPLING AND INSTALLATION of each well, and prior to the drilling of the first well, the Driller shall decontaminate all augers, rods, split-spoon samplers, tremie pipes and any other tools to be used in the drilling operations.

B. THE DRILLER shall have a steam-cleaning unit on the work site to be used for the decontamination of his equipment.

C. ALL CASING AND SCREEN shall be steam cleaned prior to being emplaced in the drilled hole.

D. ALL SPLIT-SPOON samples shall be scrubbed and washed between each use. The samplers will be washed in Micro solution and then rinsed in fresh water of known quality.

3.08 ABANDONMENT OF WELLS

A. IN THE EVENT that the Driller shall fail to drill a well or place the casings to the depth specified or to such lesser depths as ordered by the Consultant or should abandon the well because of loss of tools or for any other cause, he shall, if requested and as directed by the Consultant, remove salvageable casings and equipment and fill the abandoned hole in accordance with state and/or local regulations. The Driller shall then move over and drill a new hole at a location approved by the Consultant. This work shall be done at the Driller's expense employing the use of salvaged materials, if usable, at the discretion of the Consultant.

3.09 BORING LOG

A. THE DRILLER shall maintain a detailed boring log of the operations during all drilling procedures. The log shall give a complete description of all formations encountered, footage, size of the hole drilled, depth, sizes of all casings installed, water-level changes and depths at which they occurred, a description of cementing operations, and other such pertinent data as may be requested by the Consultant. The daily log shall be maintained on the site and be available for inspection by the Consultant at all times.

APPENDIX B. GROUND-WATER SAMPLING PROTOCOL

AVX FACILITY

COLLECTION OF WATER SAMPLES

Water samples will be collected from the existing production well and the proposed new monitoring wells for volatile organic compounds (VOCs) and landfill indicator parameters. In addition, water samples will be collected from existing monitoring wells 10A, B, C, and 9A and B, 7A and B, and MW-1 for an analysis of landfill indicator parameters and volatile organic compounds.

The samples collected for volatile organic analysis will be analyzed with the procedures, and for the parameters, set forth in EPA Method 624 (other major peaks will also be identified and quantified). The landfill indicator parameters to be analyzed for are: conductance, pH, temperature, alkalinity, chloride, potassium, sulfate, ammonia, and iron.

In addition to the samples outlined above, we will split samples from two wells (field replicates) and send the differently labeled samples to the laboratory for VOC analysis. This procedure serves as a check on the precision of the analytical procedures used by the laboratory.

A rigorous protocol will be followed prior to and during sampling to insure that all samples represent conditions in the formation tapped by the well. Prior to sampling, five volumes of

standing water will be removed from each monitoring well capable of sustaining a significant (measurable) yield. This will be accomplished using a centrifugal pump for the wells where the depth to water does not exceed 20 feet and a submersible pump for wells with a water levels below suction limits. Samples from the monitoring wells will be collected with a teflon bailer. The sample from the production well will be collected from the pump discharge, during normal pumping conditions.

1.0 Well Evacuation Procedures

1.1 Identify the well and record its number.

1.2 Clean the top of the well with a clean cloth.

1.3 Remove the well cap or plug, wipe the inside of the casing with a clean cloth and place the cap on plastic material.

1.4 Clean the first 5 feet of the steel measuring tape with distilled water and then measure the depth to water from the top of the well casing.

1.5 Compute the volume of water in the 4-inch diameter well (height of water column (feet) x 0.65 gallons/foot).

1.6 Remove five times the volume of standing water in the well using a centrifugal pump or a submersible pump, depending upon the depth to water.

1.6.1 The intake opening of the pump hose should be positioned and maintained just below the water surface in the well casing to ensure that the well is properly flushed.

If there is a slight decrease in the well's water level as a result of pumping, the intake line should be lowered as needed. This procedure does not have to be followed for those wells having a low specific capacity, which is indicated by a rapid and pronounced decline in the water level, even to the point where the well is pumped dry.

1.6.2 If the well has been pumped or developed recently, the water level may not yet have recovered or returned to its normal level. This does not require a change in the evacuation procedures outlined above. Although the actual volume of water in the casing under such conditions is less than normally found, the removal of five times this volume is sufficient to provide samples for analysis that are representative of the water in the surrounding formation.

1.6.3 If the well is pumped dry during this procedure and shows essentially complete recovery within 15 minutes, removal of water should continue for four additional pump down and recovery periods. If recovery is less than 75 percent during the 15 minutes after complete evacuation, sampling can begin with the next appearance of water. However, the initial volume which eventually becomes available may not be large enough to complete the sampling in the brief period of time normally required.

2.0 Well Sampling Procedure

2.1 Sample the wells with a teflon bailer. Each AVX well will have a dedicated bailer to avoid cross contamination. In addition, a disposable plastic line will be used to lower the bailer. This will reduce the chance of introducing foreign objects into the bailer.

2.2 Samples will be collected in a glass bottle that is large enough to fill all of the individual sample containers. The large collection bottle will be thoroughly rinsed with distilled water and then rinsed with water from the well prior to the actual collection of the samples.

2.2.1 If a well will not yield the volume of water necessary to immediately fill all of the same containers, each container should be filled in succession as ground water enters the well.

2.3 Once samples have been collected they will be prepared and preserved in the following manner:

Constituent	Container/Size	Preparation	Acidification
pH, specific conductance, temperature	Beaker	Analyze in field	None
Iron	Plastic 0.5-1 liter	Filter/cool to 4 degrees C	pH <2 with nitric acid

Constituent	Container/Size	Preparation	Acidification
Chloride, sulfate, potasium, alkalinity	Plastic/glass 1 liter	Cool to 4 degrees C	None
Ammonia	Plastic/glass 1 liter	Cool to 4 degrees C	PH <2 with sulfuric acid
VOCs	Two 40 ml bottles with teflon septa and screw caps	Cool to 4 degrees C	None

2.4.1 All water samples designated for iron analysis will be filtered through a 0.45 micrometer membrane filter prior to acidification. This is done to ensure that only iron ions initially in solution will be measured. (acidification can displace metal ions absorbed on particles in the sample that are not initially removed. Therefore, the unfiltered sample usually shows much higher metal concentrations; the higher value corresponds to the "dissolved plus displaceable" metal-ion concentration). As the membrane may become clogged by sample turbidity, prefiltration through paper and/or fiberglass may be required to expedite the filtration process.

APPENDIX C. SOIL SAMPLING PROTOCOL FOR DRILLED SAMPLES

Split spoon samples will be taken during the drilling of monitoring wells AVX-1 and AVX-2. Samples will be collected at five-foot intervals up to the screen setting. The samples will be collected according to the following procedure:

1.0 Sample Handling and Bottling

1.1 Each split spoon will be opened on a clean piece of plastic sheeting, and handled with disposable plastic gloves.

1.2 The split spoon core will be trimmed with a clean knife and spatula. The outside of each core will be trimmed off, and two samples will be cut to the size of a 40 ml VOC bottle.

1.3 Two samples will be collected from each split spoon core and placed within separate 40 ml glass VOC bottles. The two samples will be identified with the well number and the depth sampled. In addition, one of the two samples will be labeled "A" and the second "B".

1.4 The samples will be handled and bottled as efficiently and expeditiously as possible, in order to minimize loss of volatile organic compounds.

2.0 Sampling Packaging and Analysis

2.1 The split spoon samples for each 15-foot interval (three samples) will be placed in a self-sealing plastic bag. The "A" samples will be bundled separately from the "B" samples.

2.2 The "A" samples bagged together will be composited in the laboratory and will be analyzed by EPA method 601 for trichloroethylene, 1,1,1-trichloroethane, and tetrachloroethylene.

2.3 The "B" samples are to be retained by the laboratory until the "A" samples have been analyzed and the results evaluated. The "B" sample corresponding to the composited "A" sample with the highest combined concentration of the three compounds will be analyzed for volatile organic compounds using EPA Method 624 (with additional major peaks identified and quantified).

3.0 Decontamination Procedures

3.1 The split-spoon samplers, knives, and spatulas used in the collection and trimming of the soil samples will be thoroughly cleaned before the collection of each sample.

3.2 The samplers, knives and spatulas will be rinsed and scrubbed in Micro solution, followed by a rinse in distilled water.

3.3 The geologist bottling the samples will use clean disposal gloves for handling each sample, and the sheet of plastic covering the work surface will be cleaned between each sample.

APPENDIX D. SOIL SAMPLING PROTOCOL FOR SURFACE SAMPLING

Surface soil samples will be collected in the undeveloped area in the southern part of the AVX facility. The soil samples will be collected according to the following protocol:

1.0 Soil Sampling Grid

Beginning at a point along the western property line, approximately 450 feet south of the north property line (or approximately 50 feet north of the south fence line), a grid 1,000 feet long (extending toward the east property line) and 200 feet wide (extending toward the south property line) will be established. This grid will be broken into 20 subgrids each 100 feet by 100 feet in dimension. Shallow borings (12 to 18 inches in depth) will be collected within each subgrid. The approximate location of the borings will be on the intersecting diagonals drawn from each corner of the subgrid. Four samples will be taken within each subgrid, at points approximately 1/4 of the distance from each corner on the diagonal, and a fifth sample will be taken at the approximate location of the intersection of the two subgrid diagonals.

2.0 Method of Sample Collection

2.1 At each sampling location, surface debris (leaves, twigs, etc.) will be cleared away.

2.2 Each boring will be advanced to a depth of 12 to 18 inches by means of a hand auger. The soil excavated will be piled at least two feet away from the hole. A plastic sheet with a hole pierced in the middle will be placed over the hole. The plastic sheeting is intended to provide a clean work place and a clean surface for sample bottles, tools, etc.

2.3 The soil samples will be collected from the excavated hole with a clean trowel. Two samples will be collected at each location, one labeled "C" and the other "D". The samples will be transferred directly to 40 ml VOC bottles, filled with as much soil as possible.

2.4 The geologist collecting the soil samples will wear disposable plastic gloves during the sampling. The samples will be transferred to the VOC bottles as expeditiously as possible to minimize volatilization of organic compounds.

2.5 The trowel used to collect the soil samples will be cleaned between each sampling location, employing the decontamination procedures described in Appendix C.

3.0 Sample Packaging and Analysis

3.1 Each sample bottle will be labeled with the subgrid number, the sampling location within the subgrid (NE, NW, SE, SW, or C for center), and either "C" or "D".

3.2 The five "C" samples collected within each subgrid will be bagged together for shipping to the laboratory. The "D" samples will be similarly packaged.

3.3 The five "C" samples from each subgrid will be composited in the laboratory and analyzed for trichloroethylene, 1,1,1-trichloroethane and tetrachloroethylene in accordance with EPA Method 601.

3.4 The "D" samples are to be retained by the laboratory until the "C" samples have been analyzed and the results reviewed. The "D" samples corresponding to the two composited "C" samples with the highest concentrations of the three compounds will be composited in the laboratory and analyzed for volatile organic compounds by EPA Method 624 (with additional major peaks identified and quantified).

APPENDIX E. PROTOCOL FOR WATER-LEVEL RECOVERY TEST

A water-level recovery test will be carried out by shutting off the AVX production well and measuring water levels in the production well, the AVX monitoring wells, and other monitoring wells in the vicinity of the AVX facility. The other monitoring wells to be measured during the test are Wells 19A, 19B, 14, 10A, 10B, 10C, 9A and 9B.

A rigorous protocol will be followed prior to, during and after the test to insure that the water-level data collected are accurate and consistent. All measurements will be made to the nearest 0.01 foot.

Prior to Shut Down

1.0 Surveying

1.1 The three monitoring wells to be installed at the AVX facility will be surveyed by a licensed surveyor in order to convert all water levels to msl.

1.2 A point on the top of the 4-inch well casing will be identified with paint. This point will be surveyed to the nearest 0.01 foot.

1.3 A point on the pump base or cement block of the AVX production well will be identified with paint. This point will also be surveyed to the nearest 0.01 foot.

1.4 The other monitoring wells that are to be measured during the test will be resurveyed by the same surveyor who surveys the AVX monitoring wells and production wells.

2.0 Pre-Test Measurements

2.1 Water-level recorders will be installed on monitoring wells AVX-1, AVX-2, and AVX-3 as soon as those wells are developed. The recorders will be equipped with 24-hour clocks. The data from these recorders will provide information to aid in the assessment of regional trends and the effects of pumping wells in the area.

2.2 Pumping water levels will be periodically measured in the AVX production well.

2.3 Water-levels will be periodically measured in the other monitoring wells (19A, 19B, 14, 10A, 10B, 10C, 9A, and 9B. The static water level in these wells will be measured within three hours of the beginning of the test.

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2.4 A barometer will be installed near the AVX production well at least 24 hours prior to the beginning of the test. The barometer will either be of the recording type, or readings will be taken every 30 minutes during the test. The barometric pressure will also be recorded for 24 hours following the test.

During the Test

3.0 AVX Monitoring Wells

3.1 Eight-hour clocks will be put on the recorders previously installed on the AVX monitoring wells. These recorders will be periodically monitored during the test, and the charts will be changed every eight hours.

4.0 AVX Production Well

4.1 The water-level in the AVX production well will be measured just prior to shut-down. Once the well is shut down, the water level will be measured on a regular schedule, set forth on page E-5. The water levels will be recorded on a standardized form, an example of which is shown on page E-6.

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5.0 Other Monitoring Wells

5.1 The water levels in monitoring wells 19A, 19B, 14, 10A, 10B, 10C, 9A and 9B will be measured once every hour for the initial six hours of the recovery test, and every two hours after that. Water-level measurements will be measured to the nearest 0.01 foot, and each measurement will be repeated twice to ensure accuracy.

Post-Test Measurements

6.0 AVX Monitoring Wells

6.1 The recorders on the monitoring wells will be equipped with 8-hour clocks for the 24-hour period after the production well is put back in service.

6.2 The 8-hour clocks will be replaced with 8-day clocks for the week following the test.

7.0 AVX Production Well

7.1 Frequent water-level measurements in the production well will be taken for the 3-hour period after the well is put back in service.

7.2 The water-level in the production well will be measured hourly from 3 hours to 8 hours following the test, and after that it will be checked every 2 or 3 hours until 24 hours after the well is put back in service.

8.0 Other Monitoring Wells

8.1 The water-levels in the monitoring wells listed in 5.0 will be measured hourly in the 3-hour period following the end of the recovery test, and every 2 or 3 hours after that until 24 hours after the production well is put back in service.

8.2 Periodic water-level measurements will be taken in these wells during the second and third day after the AVX production well is put back in service.

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OLEAN, NEW YORK

2 day test

Elapsed Time (minutes)	Frequency of Measurements
1 - 5	Every 30 seconds
5 - 10	Every minute
10 - 30	Every 2 minutes
30 - 60	Every 5 minutes
60 - 120 (2 hours)	Every 10 minutes
120 - 180 (3 hours)	Every 20 minutes
180 - 360 (6 hours)	Every 30 minutes
360 - 720 (12 hours)	Every hour
720 - 2,880 (48 hours)	Every 2 hours

Environmental Monitoring and Support Laboratory—Cincinnati, Ohio 45268, March 1979.
9. "EPA Method Validation Study 23, Method 601 (Purgeable Halocarbons)," Report for EPA Contract 68-03-2856 (In preparation).

Table 1. Chromatographic Conditions and Method Detection Limits

Parameter	Retention Time (min.) Column 1	Retention Time (min.) Column 2	Method Detection Limit µg/L
Chloromethane	1.50	5.28	0.08
Bromomethane	2.17	7.05	1.18
Dichlorodifluoromethane	2.62	nd	1.81
Vinyl chloride	2.67	5.28	0.18
Chloroethane	3.33	8.68	0.52
Methylene chloride	5.25	10.1	0.25
Trichlorofluoromethane	7.18	nd	nd
1,1-Dichloroethene	7.93	7.72	0.13
1,1-Dichloroethane	9.30	12.6	0.07
trans-1,2-Dichloroethene	10.1	9.38	0.10
Chloroform	10.7	12.1	0.05
1,2-Dichloroethane	11.4	15.4	0.03
1,1,1-Trichloroethane	12.6	13.1	0.03
Carbon tetrachloride	13.0	14.4	0.12
Bromodichloromethane	13.7	14.6	0.10
1,2-Dichloropropane	14.9	16.6	0.04
trans-1,3-Dichloropropene	15.2	16.6	0.34
Trichloroethene	15.8	13.1	0.12
Dibromochloromethane	16.5	16.6	0.09
1,1,2-Trichloroethane	16.5	18.1	0.02
cis-1,3-Dichloropropene	16.5	18.0	0.20
2-Chloroethylvinyl ether	18.0	nd	0.13
Bromoform	19.2	19.2	0.20
1,1,2,2-Tetrachloroethane	21.6	nd	0.03
Tetrachloroethene	21.7	15.0	0.03
Chlorobenzene	24.2	18.8	0.25
1,3-Dichlorobenzene	34.0	22.4	0.32
1,2-Dichlorobenzene	34.9	23.5	0.15
1,4-Dichlorobenzene	35.4	22.3	0.24

nd = not determined

Column 1 conditions: Carbopack B 60/80 mesh coated with 1% SP-1000 packed in an 8 ft × 0.1 in ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 45 °C for 3 min. then programmed at 8 °C/min. to 220 ° and held for 15 min.

Column 2 conditions: Porasil-C 100/120 mesh coated with n-octane packed in a 6 ft × 0.1 in ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 50 °C for 3 min then programmed at 6 °C/min to 170 ° and held for 4 min.

Table 2. Single Operator Accuracy and Precision

Parameter	Average Percent Recovery	Standard Deviation %	Spike Range (ug/L)	Number of Analyses	Matrix Types
Bromodichloromethane	100.9	5.0	0.43-46.7	21	3
Bromoform	89.5	9.0	1.45-50	20	3
Bromomethane	105.0	17.3	3.39-49.2	21	3
Carbon tetrachloride	82.5	25.6	0.55-50	19	3
Chlorobenzene	93.9	8.9	2.21-50	20	3
Chloroethane	91.5	22.4	3.95-50	21	3
2-Chloroethylvinyl ether	96.3	9.9	4.39-133	20	3
Chloroform	101.7	20.6	0.44-50	20	3
Chloromethane	91.4	13.4	0.55-23.9	21	3
Dibromochloromethane	98.3	6.5	0.75-93.0	21	3
1,2-Dichlorobenzene	10.20	2.0	4.89-154	21	3
1,3-Dichlorobenzene	91.6	4.3	2.94-46.7	21	3
1,4-Dichlorobenzene	97.5	9.3	2.99-51.6	21	3
Dichlorodifluoromethane	87.8	18.0	2.18-43.4	21	3
1,1-Dichloroethane	102.3	5.5	0.44-46.7	21	3
1,2-Dichloroethane	97.8	4.8	0.44-46.7	21	3
1,1-Dichloroethene	101.1	21.7	0.37-50	19	3
trans-1,2-Dichloroethene	91.0	19.3	0.44-98.0	20	3
1,2-Dichloropropane	97.7	8.8	0.29-39.0	21	3
cis-1,3-Dichloropropene	86.7	6.0	0.44-46.7	21	3
trans-1,3-Dichloropropene	73.5	17.2	0.43-50	20	3
Methylene chloride	97.9	2.6	0.73-46.7	21	3
1,1,2,2-Tetrachloroethane	91.9	15.0	0.46-46.7	21	3
Tetrachloroethene	94.1	18.1	0.50-35.0	21	3
1,1,1-Trichloroethane	75.1	12.5	0.37-29.0	21	3
1,1,2-Trichloroethane	91.0	25.1	0.45-50	21	3
Trichloroethene	106.1	7.4	0.38-46.7	21	3
Trichlorofluoromethane	89.3	13.9	149	14	2
Vinyl chloride	101.9	11.4	0.82-32.3	21	3

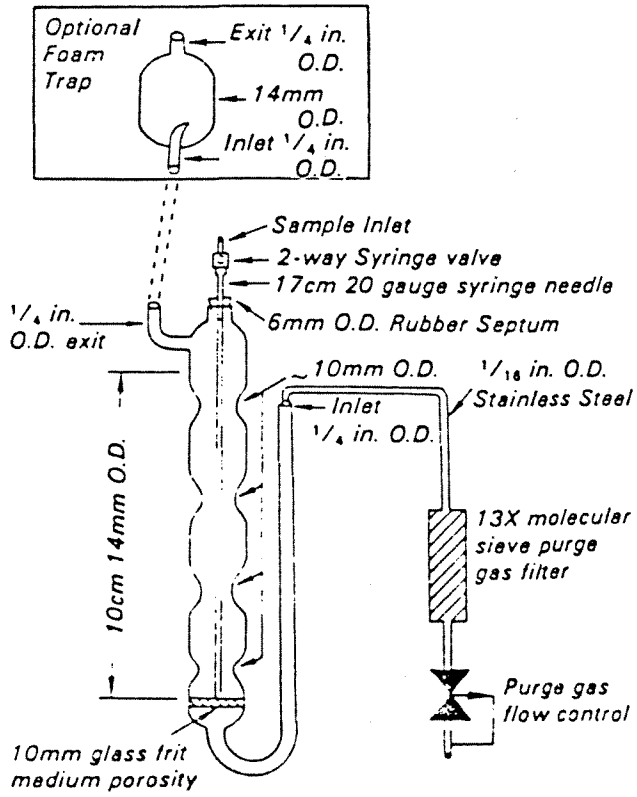

Figure 1. Purging device

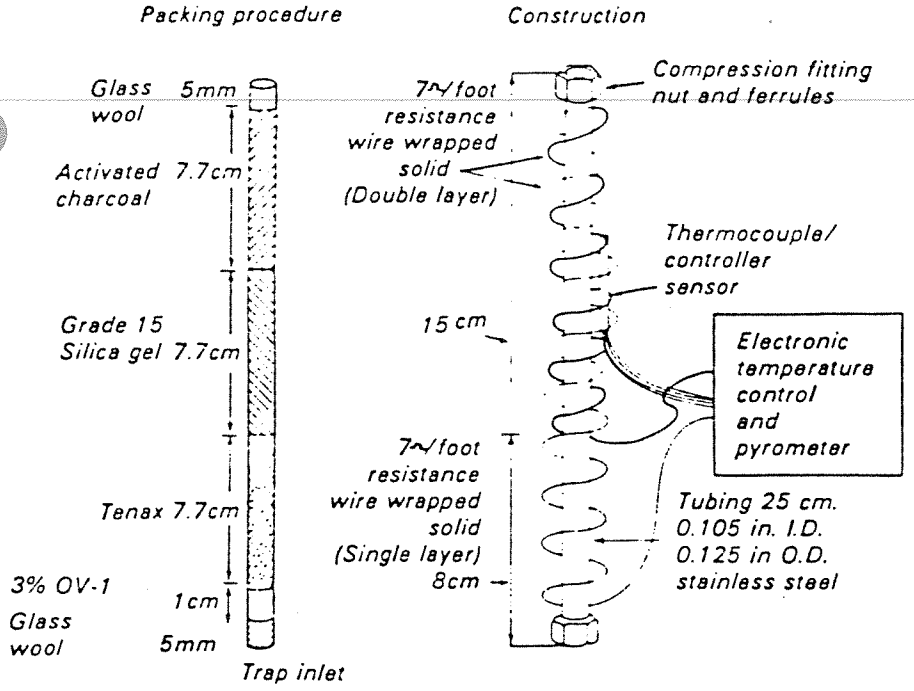

Figure 2. *Trap packings and construction to include desorb capability*

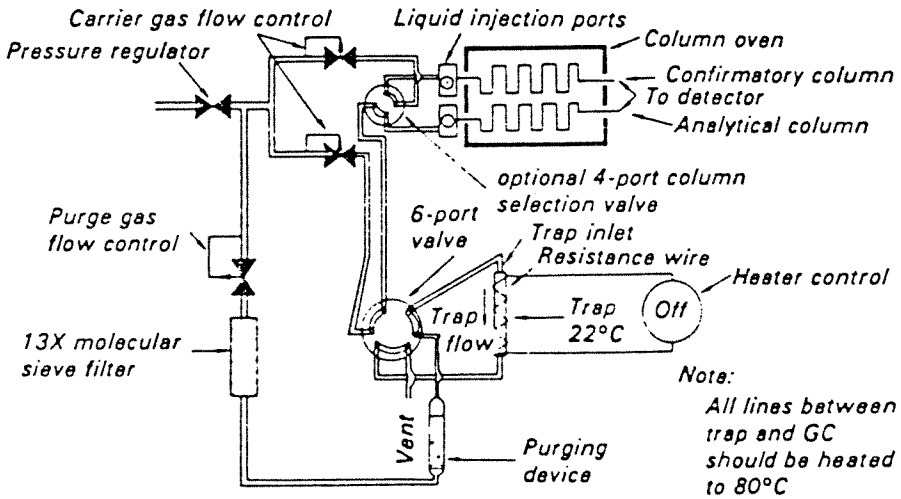

Figure 3. *Schematic of purge and trap device — purge mode*

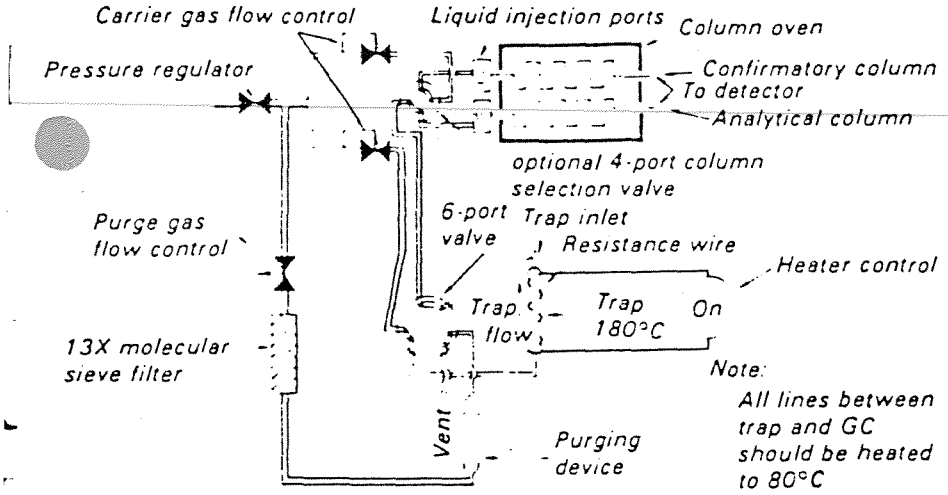

Figure 4. *Schematic of purge and trap device — desorb mode*

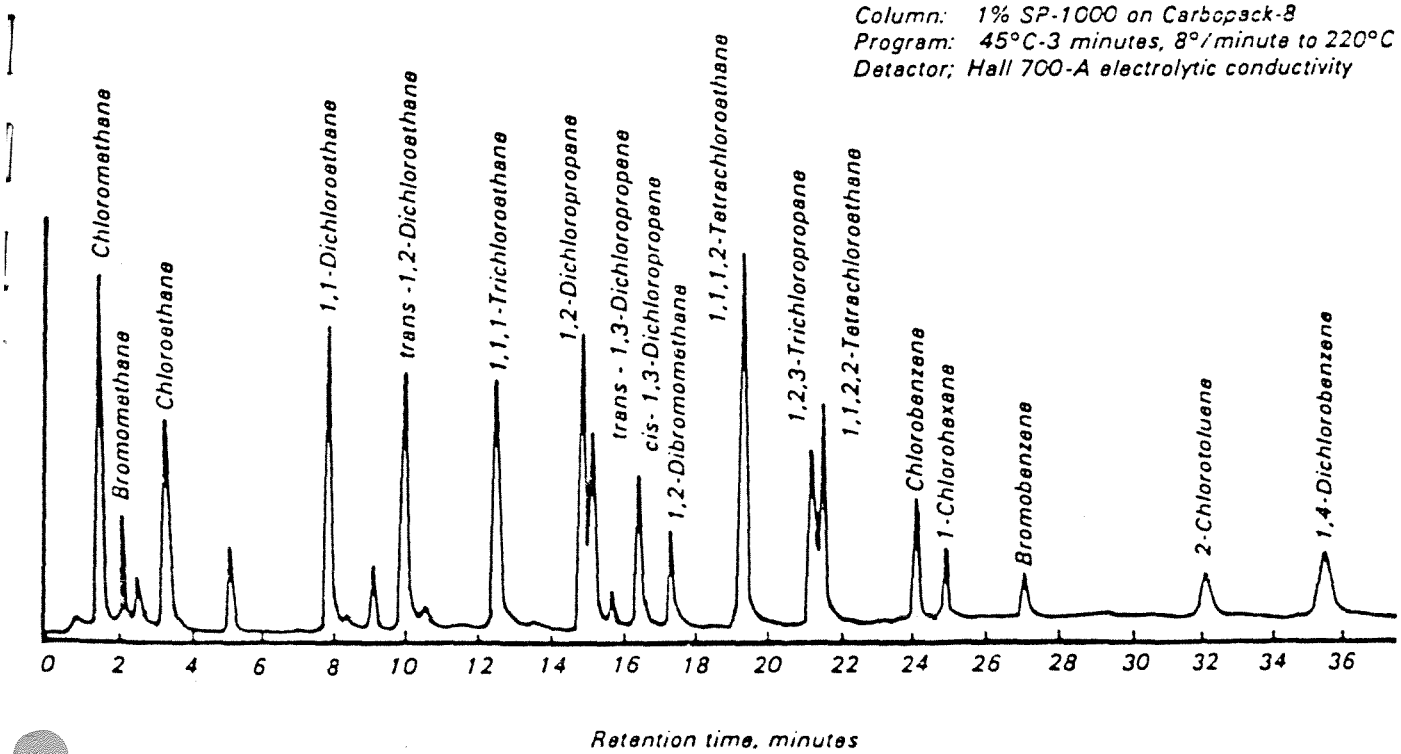

Figure 5. *Gas chromatogram of purgeable halocarbons*

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United States
Environmental Protection
Agency

Environmental Monitoring and
Support Laboratory
Cincinnati OH 45268

Research and Development

EPA

Test Method

Purgeables — Method 624

1. Scope and Application

1.1 This method covers the determination of a number of purgeable organics. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Benzene	34030	71-43-2
Bromodichloromethane	32101	75-27-4
Bromoform	32104	75-25-2
Bromomethane	34413	74-83-9
Carbon tetrachloride	32102	56-23-5
Chlorobenzene	34301	108-90-7
Chloroethane	34311	75-00-3
2-Chloroethylvinyl ether	34576	110-75-8
Chloroform	32106	67-66-3
Chloromethane	34418	74-87-3
Dibromochloromethane	32105	124-48-1
1,2-Dichlorobenzene	34536	95-50-1
1,3-Dichlorobenzene	34566	541-73-1
1,4-Dichlorobenzene	34571	106-46-7
1,1-Dichloroethane	34496	75-34-3
1,2-Dichloroethane	34531	107-06-2
1,1-Dichloroethene	34501	75-35-4
trans-1,2-Dichloroethene	34546	156-60-5
1,2-Dichloropropane	34541	78-87-5
cis-1,3-Dichloropropene	34704	10061-01-5
trans-1,3-Dichloropropene	34699	10061-02-6
Ethyl benzene	34371	100-41-4
Methylene chloride	34423	75-09-2
1,1,2,2-Tetrachloroethane	34516	79-34-5
Tetrachloroethene	34475	127-18-4
Toluene	34010	108-88-3
1,1,1-Trichloroethane	34506	71-55-6
1,1,2-Trichloroethane	34511	79-00-5
Trichloroethene	39180	79-01-6
Trichlorofluoromethane	34488	75-69-4
Vinyl chloride	39175	75-01-4

1.2 The method may be extended to screen samples for acrolein (STORET No. 34210, CAS No. 107-02-8) and acrylonitrile (STORET 34215, CAS No. 107-13-1), however, the preferred method for these two compounds is method 603.

1.3 This is a purge and trap gas chromatographic/mass spectrometer

(GC/MS) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1.

1.4 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater differ from those listed, depending upon the nature of interferences in the sample matrix.

1.5 Until the U.S. Environmental Protection Agency establishes performance criteria based upon the results of interlaboratory testing, any alternative GC/MS method which meets the performance criteria described in Section 8.2 will be permitted. Performance must be verified for such modification by analyzing wastewater as described in Section 8.2.2. In addition, the laboratory must successfully participate in the applicable performance evaluation studies.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of purge and trap systems and gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL sample contained in a specially-designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer[2,3].

3. Interferences

3.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.5. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105 °C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[5-7] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with hole in center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for one hour before use.

5.2 Purge and trap device—The purge and trap device consists of three separate pieces of equipment: the sample purger, trap, and the desorber. Several complete devices are now commercially available.

5.2.1 The sample purger must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15-mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated packing (Section 6.3.2), 15 cm of 2,6-diphenylene oxide polymer (Section 6.3.1), and 8 cm of silica gel, (Section 6.3.3). The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber should be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 220 °C. The desorber design, illustrated in Figure 2, meets these criteria.

5.2.4 The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 GC/MS system.

5.3.1 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, and gases.

5.3.2 Column—6 ft long × 0.1 in ID stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 11.1.

5.3.3 Mass spectrometer—Capable of scanning from 20 to 260 amu every seven seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria in Table 2 when 50 ng of 4-bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

5.3.4 GC/MS interface—Any gas chromatograph to mass spectrometer interface that gives acceptable calibration points at 50 ng or less per injection for each of the parameters of interest and achieves all acceptable performance criteria (see Section 10) may be used. Gas chromatograph to mass spectrometer interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichloro-dimethylsilane.

5.3.5 Data system—A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-mL, 0.006 inch ID needle.

5.6 Syringe valve—two-way, with Luer ends (three each), if applicable to the purging device.

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for one hour. While still hot, transfer the water to a narrow-mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap materials

6.3.1 2,6-Diphenylene oxide polymer—Tenax (60/80 mesh), chromatographic grade or equivalent.

6.3.2 Methyl silicone packing—3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.

6.3.3 Silica gel, Davison Chemical, (35/60 mesh), grade-15 or equivalent.

6.4 Methanol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.5.1 Place about 9.8 mL of methanol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.5.2 Add the assayed reference material as described below:

6.5.2.1 Liquids—Using a 100-μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the four halocarbons that boil below 30 °C (bromomethane, chloroethane, chloromethane, and vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas rapidly dissolves in the methanol.

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at −10° to −20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the four gases and 2-chloroethyl-vinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicate a problem.

6.6 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 7.3.1 or 7.4.1 will bracket the

working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Quality control check standards that can used to determine the accuracy of calibration standards, will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.7 Surrogate standard spiking solution—Select a minimum of three surrogate compounds from Table 3. Prepare stock standard solutions for each surrogate standard in methanol as described in Section 6.5. Prepare a surrogate standard spiking solution from these stock standards at a concentration of 150 µg/10 mL in water. Store the spiking solution at 4 °C in Teflon sealed glass containers with a minimum of headspace. The solutions should checked frequently for stability. They should be replaced after six months. The addition of 10 µL of this solution to 5 mL of sample or standard is equivalent to a concentration of 30 µg/L of each surrogate standard. Surrogate standard spiking solutions, appropriate for use with this method, will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.8 BFB Standard—Prepare a 25 µg/µL solution of BFB in methanol.

7. Calibration

7.1 Assemble a purge and trap device that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by back flushing with an inert gas flow of at least 20 mL/min. Prior to use, daily condition traps 10 minutes while backflushing at 180 °C.

7.2 Connect the purge and trap device to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate parameters equivalent to those in Table 1. Calibrate the purge and trap-GC/MS system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 µL of one or more secondary dilution standards to 50, 250, or 500 mL of reagent water. A 25-µL syringe with a 0.006 inch ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL (See Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system. Aqueous standards may be stored up to 24 hours, if held in sealed vials with zero headspace as described in Section 9.2. If not so stored, they must be discarded after one hour.

7.3.2 Analyze each calibration standard according to Section 11, and tabulate the area response of the primary characteristic ion (See Table 4) against the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3.3 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve or calibration factor must be prepared for that parameter.

7.4 Internal standard calibration procedure. To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. Due to their generally unique retention times, bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane have been used successfully as internal standards.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration of 15 µg/mL of each internal standard compound. The addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 µg/L.

7.4.3 Analyze each calibration standard, according to Section 11, adding 10 µL of internal standard spiking solution directly to the syringe (Section 11.4). Tabulate the area response of the characteristic ions against concentration for each compound and internal standard and calculate response factors (RF) for each compound using equation 1.

$$\text{Eq. 1 } RF = (A_s C_{is})/(A_{is} C_s)$$

where:

A_s = Area of the characteristic ion for the parameter to be measured.

A_{is} = Area of the characteristic ion for the internal standard.

C_{is} = Concentration of the internal standard.

C_s = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve or response ratios, A_s/A_{is}, vs. RF.

7.4.4 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within accuracy and precision limits expected of the method.

8.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of the rapid advances that are occurring in chromatography, the analyst is permitted to certain options to improve the separations or lower the cost of measurements. Each time such modifications are made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 The laboratory must spike all samples with surrogate standards to monitor continuing laboratory performance. This procedure is described in Section 8.4.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 Select a representative spike concentration for each parameter to be measured. Using stock standards, prepare a quality control check sample concentrate in methanol 500 times more concentrated than the selected concentrations. Quality control check sample concentrates, appropriate for use with this method, will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

8.2.2 Using a syringe, add 10 µL of the check sample concentrate and 10 µL of the surrogate standard dosing solution (Section 6.7) to each of a minimum of four 5-mL aliquots of reagent water. A representative wastewater may be used in place of the reagent water, but one or more additional aliquots must be analyzed to determine background levels, and the spike level must exceed twice the background level for the test to be valid. Analyze the aliquots according to the method beginning in Section 11.

8.2.3 Calculate the average percent recovery, (R), and the standard deviation of the percent recovery (s), for all parameters and surrogate standards. Wastewater background corrections must be made before R and s calculations are performed.

8.2.4 Using Table 5, note the average recovery (X) and standard deviation (p) expected for each method parameter. Compare these to the calculated values for R and s. If $s > p$ or $|X - R| > p$, review potential problem areas and repeat the test.

8.2.5 The U.S. Environmental Protection Agency plans to establish performance criteria for R and s based upon the results of interlaboratory testing. When they become available, these criteria must be met before any samples may be analyzed.

8.3 The analyst must calculate method performance criteria for each of the surrogate standards.

8.3.1 Calculate upper and lower control limits for method performance for each surrogate standard, using the values for R and s calculated in Section 8.2.3:

Upper Control Limit (UCL) = R + 3s
Lower Control Limit (LCL) = R − 3s

The UCL and LCL can be used to construct control charts[8] that are useful in observing trends in performance. The control limits above must be replaced by method performance criteria as they become available from the U.S. Environmental Protection Agency.

8.3.2 For each surrogate standard, the laboratory must develop and maintain separate accuracy statements of laboratory performance for wastewater samples. An accuracy statement for the method is defined as R ± s. The accuracy statement should be developed by the analysis of four aliquots of wastewater as described in Section 8.2.2, followed by the calculation of R and s. Alternately, the analyst may use four wastewater data points gathered through the requirement for continuing quality control in Section 8.4. The accuracy statements should be updated regularly[8].

8.4 The laboratory is required to spike all of their samples with the surrogate standard spiking solution to monitor spike recoveries. If the recovery for any surrogate standard does not fall within the control limits for method performance, the results reported for that sample must be qualified as described in Section 13.3. The laboratory should monitor the frequency of data so qualified to ensure that it remains at or below 5%.

8.5 Each day, the analyst must demonstrate, through the analysis of reagent water, that interferences from the analytical system are under control.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 All samples must be iced or refrigerated from the time of collection until extraction. If the sample contains residual chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottles just prior to shipping to the sampling site. U.S. Environmental Protection Agency methods 330.4 and 330.5 may be used for measurement of residual chlorine[9]. Field test kits are available for this purpose.

9.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for one minute. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions[3]. Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. For this reason, a separate sample should be collected, acidified, and analyzed when these aromatics are to be determined. Collect about 500 mL of sample in a clean container. Adjust the pH of the sample to about 2 by adding HCl (1 + 1) while stirring. Check pH with narrow range (1.4 to 2.8) pH paper. Fill a sample container as described in Section 9.2. If chlorine residual is present, add sodium thiosulfate to another sample container and fill as in Section 9.2 and mix thoroughly.

9.4 All samples must be analyze within 14 days of collection.

10. Daily GC/MS Performance Tests

10.1 At the beginning of each day that analyses are to be performed, the

GC/MS system must be checked to see if acceptable performance criteria are achieved for BFB[10]. The performance test must be passed before any samples, blanks, or standards are analyzed, unless the instrument has met the DFTPP test described in method 625 earlier in the day[11].

10.2 These performance tests require the following instrumental parameters.

Electron Energy:	70 Volts (nominal)
Mass Range:	20 to 260
Scan Time:	to give at least 5 scans per peak but not to exceed 7 seconds per scan.

10.3 At the beginning of each day, inject 2 µL of BFB solution directly on column. Alternately, add 2 µL of BFB solution to 5.0 mL of reagent water or standard solution and analyze according to Section 11. Obtain a background corrected mass spectrum of BFB and check that all the key ion criteria in Table 2 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.

11. Sample Extraction and Gas Chromatography

11.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. This table includes retention times and method detection limits that were achieved under these conditions. An example of the parameter separations achieved by Column 1 is shown in Figure 5. Other packed columns or chromatographic conditions may be used if the requirements of Section 8.2 are met.

11.2 After achieving the key ion abundance criteria in Section 10, calibrate the system daily as described in Section 7.

11.3 Adjust the purge gas (helium) flow rate to 40 ± 3 mL/min. Attach the trap inlet to the purging device, and set the device to purge. Open the syringe valve located on the purging device sample introduction needle.

11.4 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 µL of the surrogate spiking solution (Section 6.7) and, if applicable, 10.0 µL of the internal standard spiking solution (Section 7.4.2) through the valve bore, then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution.

11.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

11.6 Close both valves and purge the sample for 11.0 ± 0.1 minutes at ambient temperature.

11.7 At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature program. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for four minutes. If this rapid heating requirement cannot be met, the gas chromatographic column must be used as a secondary trap by cooling it to 30 °C (or subambient, if problems persist) instead of the recommended initial temperature of 45 °C.

11.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

11.9 After desorbing the sample for four minutes, recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. Trap temperatures up to 230 °C may be employed, however, the higher temperature will shorten the useful life of the trap. After approximately seven minutes turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

11.10 If the response for any ion exceeds the working range of the system, dilute the sample aliquot in the second syringe with reagent water and reanalyze.

12. Qualitative Identification

12.1 Obtain EICPs for the primary ion (Table 4) and at least two secondary ions for each parameter of interest. The following criteria must be met to make a qualititative identification.

12.1.1 The characteristic ions of each parameter of interest must maximize in the same or within one scan of each other.

12.1.2 The retention time must fall within ±30 seconds of the retention time of the authentic compound.

12.1.3 The relative peak heights of the three characteristic ions in the EICPs must fall within ±20% of the relative intensities of these ions in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library.

12.2 Structural isomers that have very similar mass spectra and less than 30 seconds difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13. Calculations

13.1 When a parameter has been identified, the quantitation of that parameter should be based on the integrated abundance from the EICP of the first listed characteristic ion given in Table 4. If the sample produces an interference for the primary ion, use a secondary characteristic ion to quantitate. Quantitation may be performed using the external or internal standard techniques.

13.1.1 If the external standard calibration procedure is used, calculate the concentration of the parameter being measured from the area of the characteristic ion using the calibration curve or calibration factor in Section 7.3.2.

13.1.2 If the internal standard calibration procedure was used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and equation 2.

Eq. 2.

Concentration µg/L = $(A_s C_{is})/(A_{is})(RF)$

where:

A_s = Area of the characteristic ion for the parameter or surrogate standard to be measured.
A_{is} = Area of the characteristic ion for the internal standard.
C_{is} = Concentration of the internal standard.

13.2 Report results in micrograms per liter. The results for cis- and trans-1,3 dichloropropene should be reported as total 1,3-dichloropropene (STORET No. 34561, CAS No. 542-75-6). When duplicate and spiked samples are analyzed, report all data obtained with the sample results.

13.3 If any of the surrogate standard recoveries fall outside the control limits which were established as directed in Section 8.4, data for all parameters determined by this method in that sample must be labeled as suspect.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero[1]. The MDL concentrations listed in Table 1 were obtained using reagent water[12]. Similar results were achieved using representative wastewaters.

14.2 The average recoveries and the average standard deviations of the percent recoveries, presented in Table 5, were the result of a study of the accuracy and precision of this method by several laboratories. The values listed represent the results from 2 to 4 laboratories[13].

14.3 The U.S. Environmental Protection Agency is in the process of conducting an interlaboratory method study to fully define the performance of this method.

References

1. See Appendix A
2. Bellar, T.A., and J.J. Lichtenberg, *Journal American Water Works Association, 66,* p. 739, (1974).
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4. "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants." U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268, March 1977, Revised April 1977. Effluent Guidelines Division, Washington, DC 10460.
5. "Carcinogens—Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, Aug. 1977.
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8. "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," EPA-600/4-79-019, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.
9. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.
10. Budde, W.L. and Eichelberger, J.W., "Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories," EPA-600/4-80-025, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268, p. 16, April 1980.
11. Eichelberg, J.W. Harris, L.E., and Budde, W.L., "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography—Mass Spectrometry Systems.", *Analytical Chemistry, 47,* 995-1000 (1979).
12. "Method Detection Limit for Methods 624 and 625," Olynyk, P., Budde, W.L. Eichelberger, J.W., unpublished report, October 1980.
13. Kleopfer, R.D., "POTW Toxic Study, Analytical Quality Assurance Final Report," U.S. Environmental Protection Agency, Region VII, Kansas City, Kansas 66115, 1981.

Table 1. *Chromatographic Conditions and Method Detection Limits*

Parameter	Retention Time (min.) Column 1	Method Detection Limit (µg/L)
Chloromethane	2.3	nd
Bromomethane	3.1	nd
Vinyl chloride	3.8	nd
Chloroethane	4.6	nd
Methylene chloride	6.4	2.8
Trichlorofluoromethane	8.3	nd
1,1-Dichloroethene	9.0	2.8
1,1-Dichloroethane	10.1	4.7
trans-1,2-Dichloroethene	10.8	1.6
Chloroform	11.4	1.6
1,2-Dichloroethane	12.1	2.8
1,1,1-Trichloroethane	13.4	3.8
Carbon tetrachloride	13.7	2.8
Bromodichloromethane	14.3	2.2
1,2-Dichloropropane	15.7	6.0
trans-1,3-Dichloropropene	15.9	5.0
Trichloroethene	16.5	1.9
Benzene	17.0	4.4
Dibromochloromethane	17.1	3.1
1,1,2-Trichloroethane	17.2	5.0
cis-1,3-Dichloropropene	17.2	nd
2-Chloroethylvinyl ether	18.6	nd
Bromoform	19.8	4.7
1,1,2,2-Tetrachloroethane	22.1	6.9
Tetrachloroethene	22.2	4.1
Toluene	23.5	6.0
Chlorobenzene	24.6	6.0
Ethyl benzene	26.4	7.2
1,3-Dichlorobenzene	33.9	nd
[illegible]-Dichlorobenzene	35.0	nd
[illegible]-Dichlorobenzene	35.4	nd

nd = not determined

Column conditions: Carbopak B (60/80 mesh) coated with 1% SP-1000 packed in a 6 ft by 2 mm ID glass column with helium carrier gas at a flow rate of 30 mL/min. Column temperature is isothermal at 45°C for 3 min, then programmed at 8°C per minute to 220°C and held for 15 min.

Table 2. *BFB Key Ion Abundance Criteria*

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but < 101% of mass 174
177	5 to 9% of mass 176

Table 3. Suggested Surrogate and Internal Standards

Compound	Retention Time (min.)[a]	Primary Ion	Secondary Ions
Surrogate Standards			
Benzene d-6	17.0	84	—
4-Bromofluorobenzene	28.3	95	174, 176
1,2-Dichloroethane d-4	12.1	102	—
1,4-Difluorobenzene	19.6	114	63, 88
Ethylbenzene d-5	26.4	111	—
Ethylbenzene d-10	26.4	98	—
Fluorobenzene	18.4	96	70
Pentafluorobenzene	23.5	168	—
Internal Standards			
Bromochloromethane	9.3	128	49, 130, 51
2-Bromo-1-chloropropane	19.2	77	79, 156
1,4-Dichlorobutane	25.8	55	90, 92

[a]For chromatographic conditions, see Table 1.

Table 4. Characteristic Ions for Purgeable Organics

Parameter	Primary Ion	Secondary Ions
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	49, 51, 86
Trichlorofluoromethane	101	103
1,1-Dichloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83, 85, 98, 100
trans-1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	98	62, 64, 100
1,1,1-Trichloroethane	97	99, 117, 119
Carbon tetrachloride	117	119, 121
Bromodichloromethane	127	83, 85, 129
1,2-Dichloropropane	112	63, 65, 114
trans-1,3-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Benzene	78	
Dibromochloromethane	127	129, 208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
cis-1,3-Dichrloropropene	75	77
2-Chloroethylvinyl ether	106	63, 65
Bromoform	173	171, 175, 250, 252, 254, 256
1,1,2,2-Tetrachloroethane	168	83, 85, 131, 133, 166
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
Chlorobenzene	112	114
Ethyl benzene	106	91
1,3-Dichlorobenzene	146	148, 113
1,2-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113

Table 5. Accuracy and Precision for Purgeable Organics

Parameter	Reagent Water Average Percent Recovery	Reagent Water Standard Deviation (%)	Wastewater Average Percent Recovery	Wastewater Standard Deviation (%)
Benzene	99	9	98	10
Bromodichloromethane	102	12	103	10
Bromoform	104	14	105	16
Bromomethane	100	20	88	23
Carbon tetrachloride	102	16	104	15
Chlorobenzene	100	7	102	9
Chloroethane	97	22	103	31
2-Chloroethylvinyl ether	101	13	95	17
Chloroform	101	10	101	12
Chloromethane	99	19	99	24
Dibromochloromethane	103	11	104	14
1,1-Dichloroethane	101	10	104	15
1,2-Dichloroethane	100	8	102	10
1,1-Dichloroethene	102	17	99	15
trans-1,2-Dichloroethene	99	12	101	10
1,2-Dichloropropane	102	8	103	12
cis-1,3-Dichloropropene	105	15	102	19
trans-1,3-Dichloropropene	104	11	100	18
Ethyl benzene	100	8	103	10
Methylene chloride	96	16	89	28
1,1,2,2-Tetrachloroethane	102	9	104	14
Tetrachloroethene	101	9	100	11
Toluene	101	9	98	14
1,1,1-Trichloroethane	101	11	102	16
1,1,2-Trichloroethane	101	10	104	15
Trichloroethene	101	9	100	12
Trichlorofluoromethane	103	11	107	19
Vinyl chloride	100	13	98	25

Samples were spiked between 10 and 1000 µg/L.

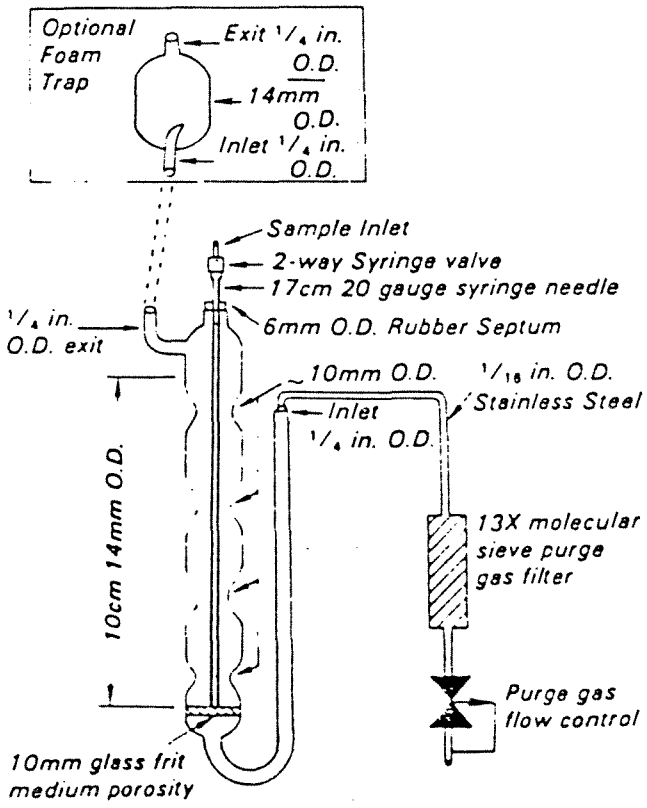


Figure 1. Purging device

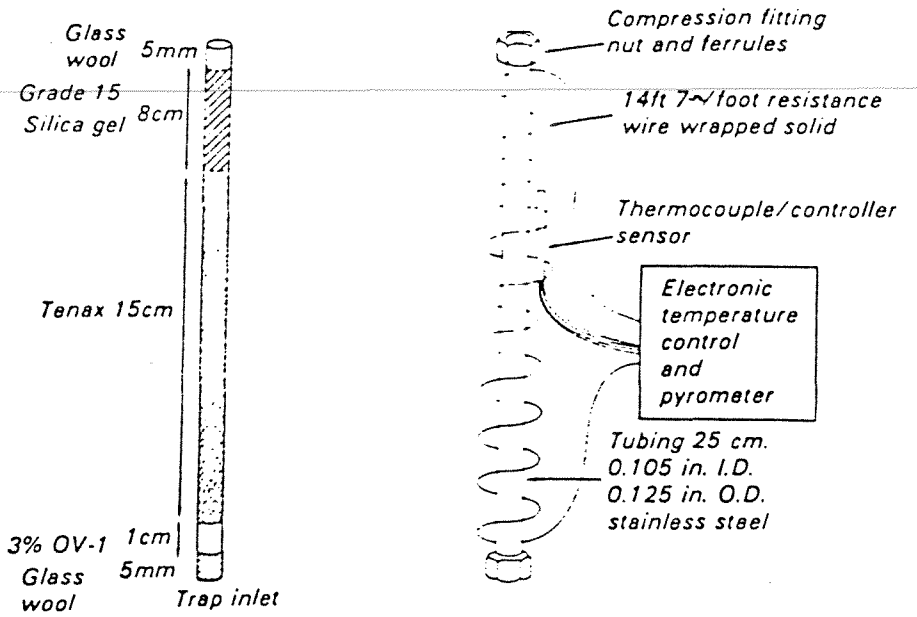


Figure 2. *Trap packings and construction to include desorb capability*

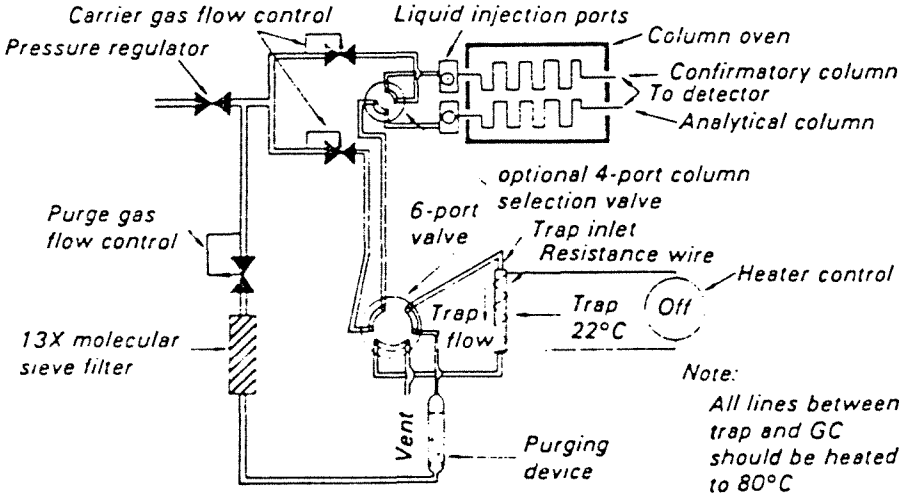


Figure 3. *Schematic of purge and trap device — purge mode*

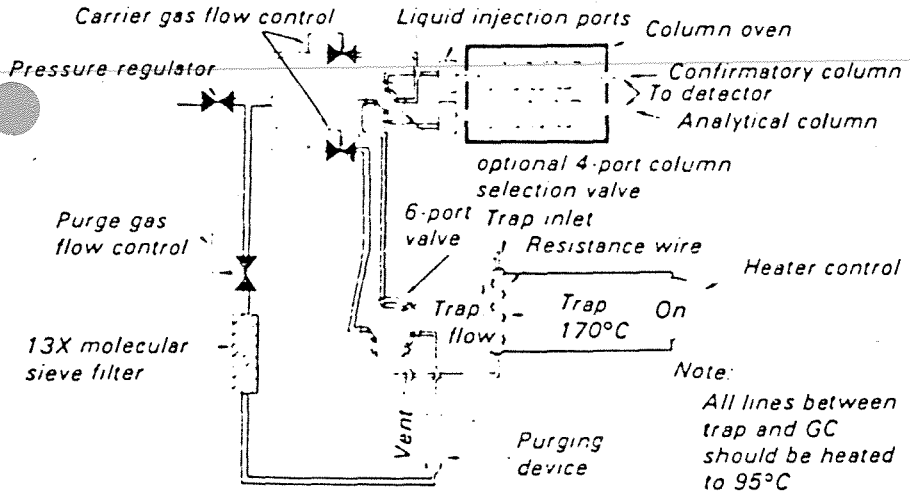


Figure 4. *Schematic of purge and trap device — desorb mode*

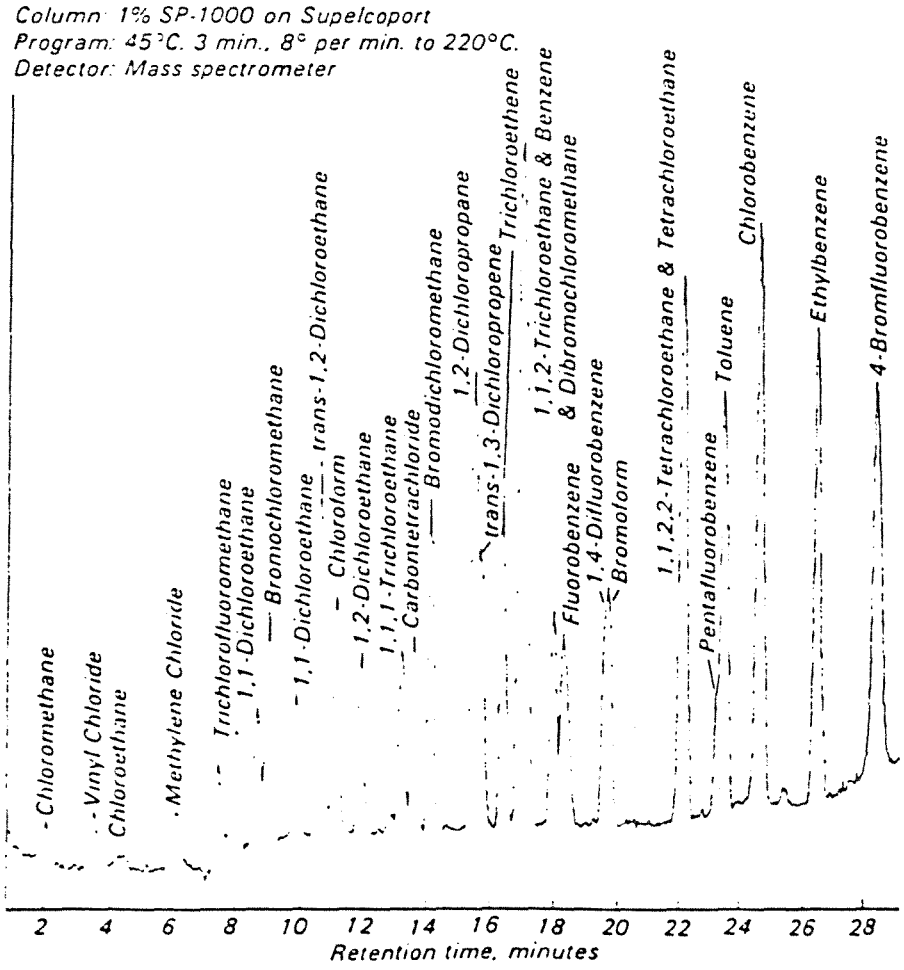


Figure 5. *Gas chromatogram of volatile organics by purge and trap.*

APPENDIX B

Geologic Logs and Construction Diagrams of the AVX Monitoring Wells

Till - clayey, silty, with medium gravel and some rock fragments and small pebbles. Gray.	24 - 49
Sand, silty, clayey sand, very fine to fine, trace of medium gravel. Gray. Wet.	49 - 54
Sand, fine to coarse, fine to medium gravel, pebbles and rock fragments. Brown.	54 - 59
Sand, very fine to medium, trace of silt. Brown. Wet.	59 - 64
Till - silt, very fine to fine sand, fine to medium gravel, and clay. Brown.	64 - 74
Sand, very fine to fine, some silt. Brown.	74 - 75.5
Sand, mostly medium, some silt. Brown.	75.5 - 80

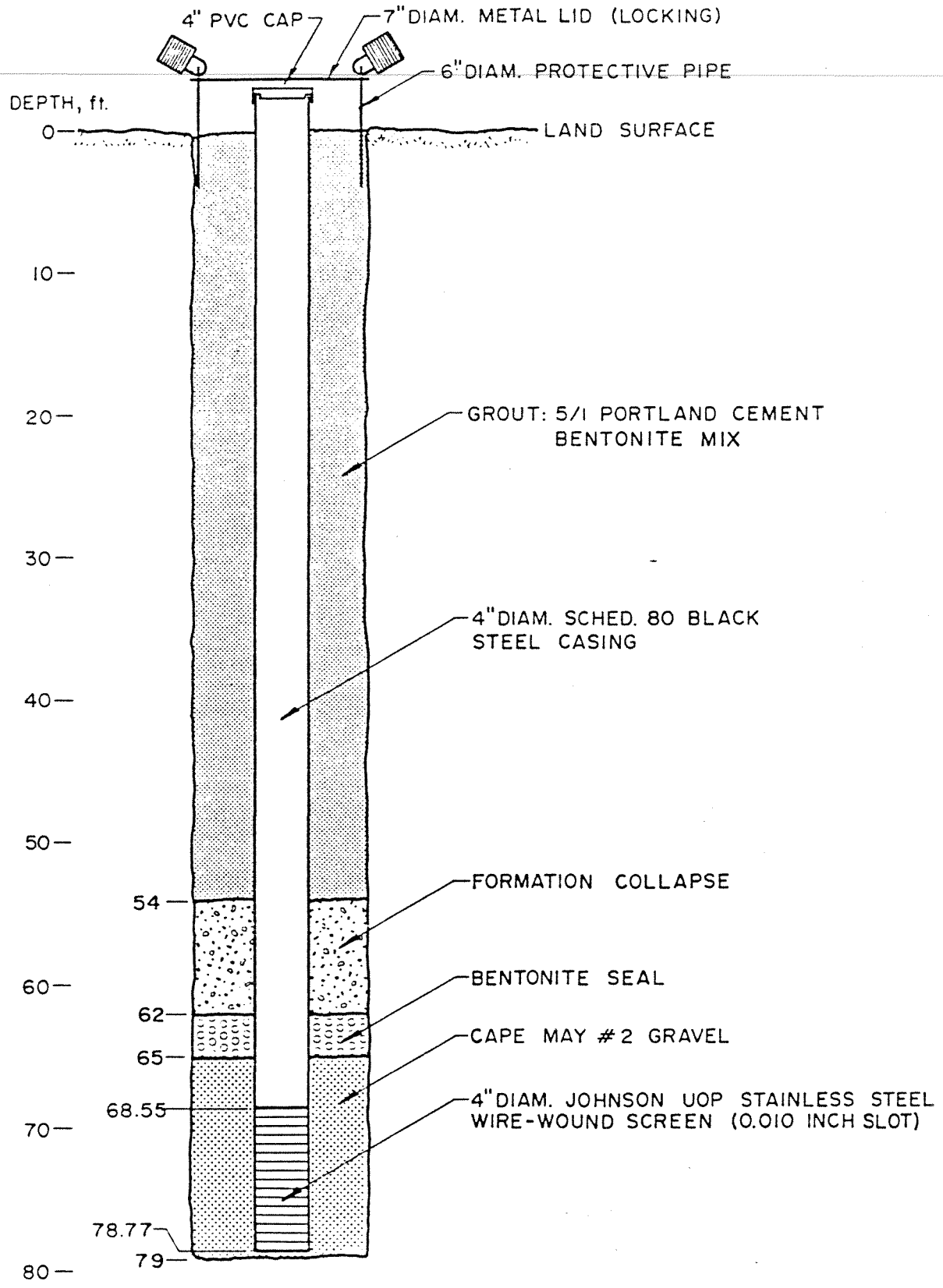


FIGURE B-1

CONSTRUCTION DIAGRAM OF MONITORING WELL AVX-1

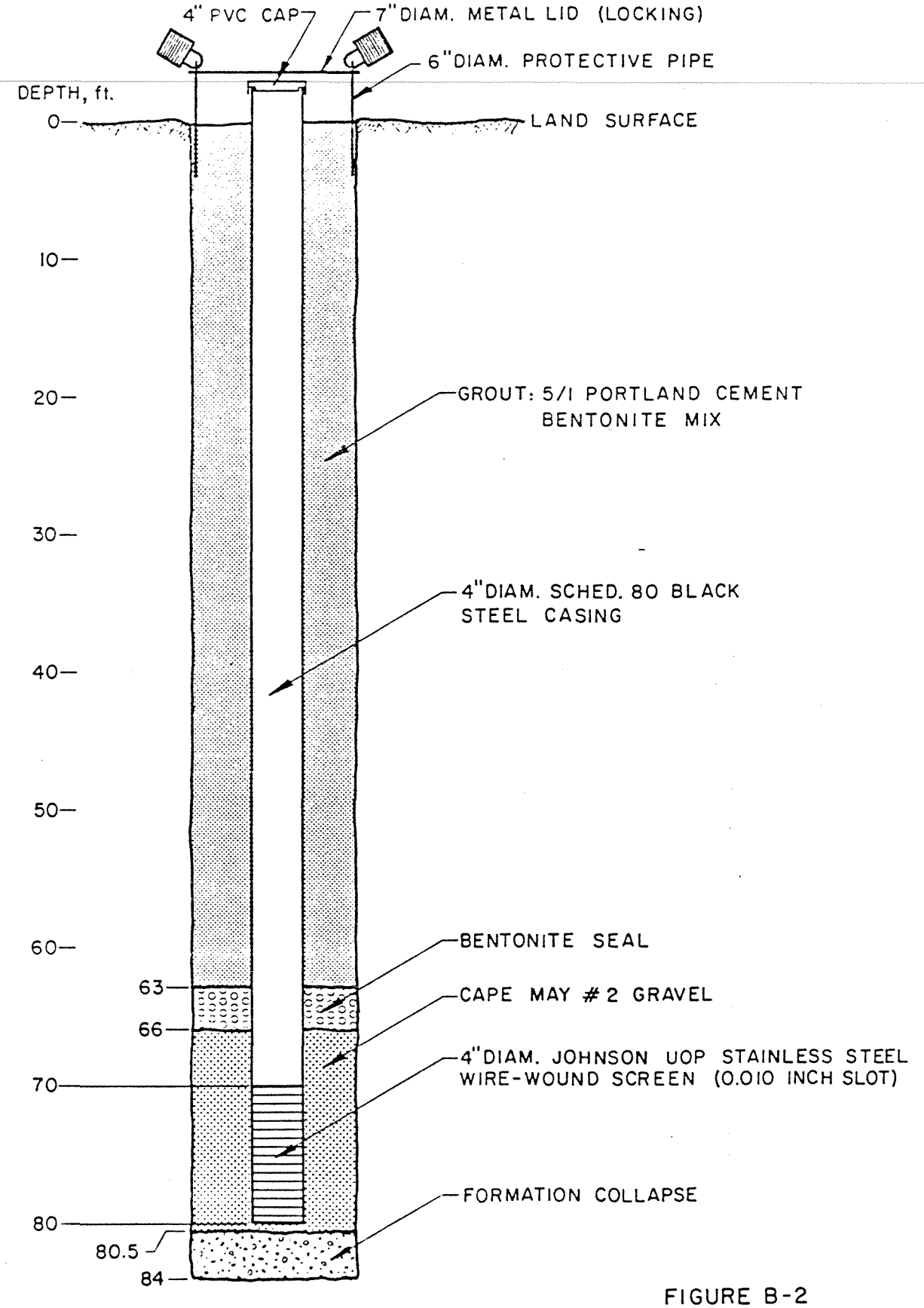
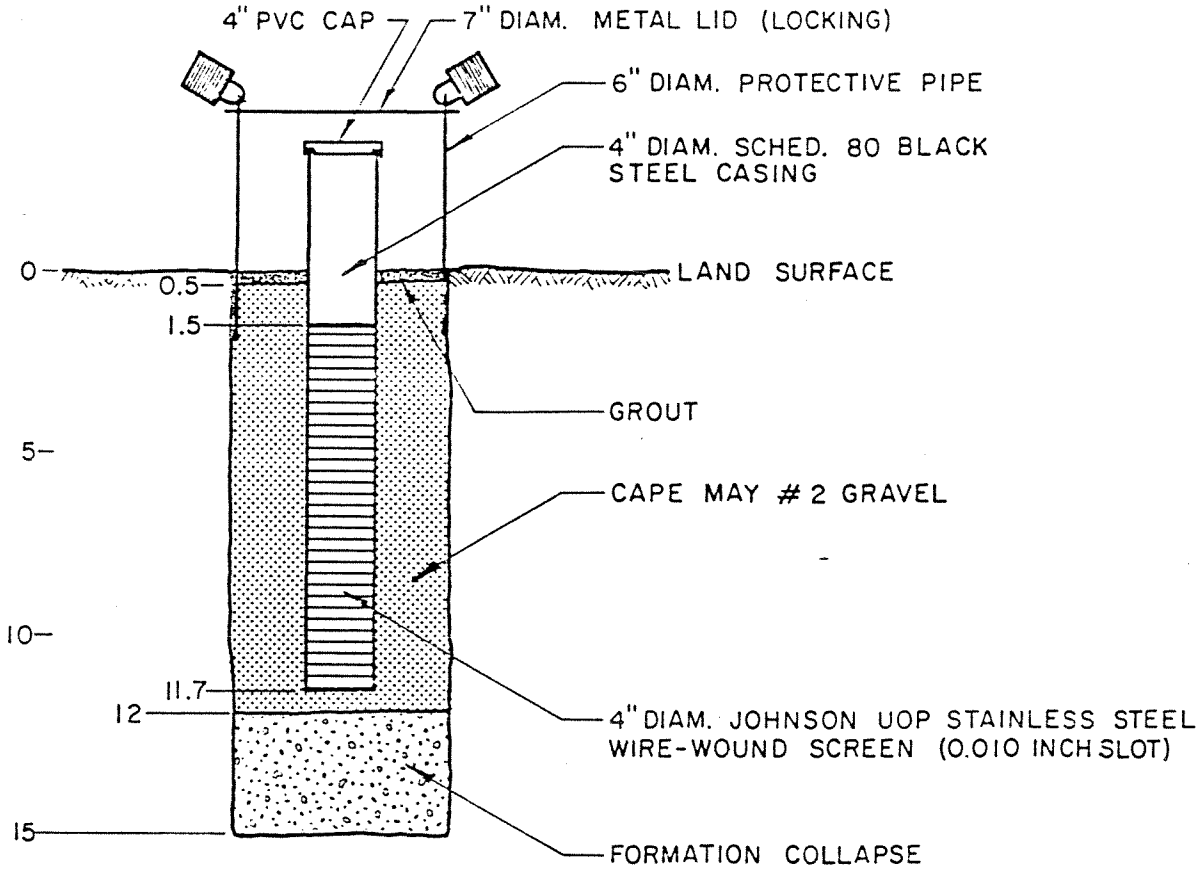


FIGURE B-2

CONSTRUCTION DIAGRAM OF MONITORING WELL AVX-2



NOTE: A BENTONITE SEAL WAS NOT EMPLACED DUE TO PROXIMITY OF GRAVEL PACK TO THE LAND SURFACE. THE CEMENT USED TO FIX THE PROTECTIVE PIPE IN PLACE SERVES TO SEAL THE GRAVEL PACK FROM LAND SURFACE.

FIGURE B-3

CONSTRUCTION DIAGRAM OF MONITORING WELL AVX-3

APPENDIX C

Cambridge Analytical Associates Laboratory Report

Cambridge Analytical Associates
1106 Commonwealth Avenue / Boston, Massachusetts 02215 / (617) 232-2207

FINAL REPORT

Geraghty & Miller, Inc.
7 Atlantic Street
Hackensack, NJ 07601
Attn: Mr. Vince Uhl

PROJECT NUMBER: J8310L2 (AVX)

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

REPORT NUMBER: 84-1295

PREPARED BY: Edward A. Lawler

DATE PREPARED: December 12, 1984

TABLE OF CONTENTS

1. INTRODUCTION

This report summarizes results of chemical analyses performed on samples received by CAA on October 30, 1984. Analytical methods employed for these analyses are described in Section 2 and results are presented in Section 3. The last section contains quality control data and certifications supporting the analytical results.

2. ANALYTICAL METHODS

Analytical methods utilized for sample analysis are summarized in Table 1.

3. RESULTS

Results of analyses are presented in Tables 2, 3 & 4.

Table 5-1. Summary of Inorganic Analytical Methods for Water

Constituent	Method Reference	Method Description
Iron (Fe)	200.7 (1)	ICP
Potassium (K)	200.7 (1)	ICP
Alkalinity	310.1 (1)	Titrimetric, pH 4.5
Chloride	325.3 (1)	Titrimetric, mercuric nitrate
Sulfate	375.4 (1)	Turbidimetric
Ammonia	350.2 (1)	Colorimetric, distillation
pH	150.1 (1)	Electrometric
Conductivity	120.1 (1)	Specific conductance

(1)U.S. EPA. 1979. Methods for Chemical Analysis of Water and Wastes. EPA 600/4-79-020 (Revised, March 1983). EPA/EMSL, Cincinnati, Ohio.

ICP - Inductively coupled argon plasma emission spectroscopy

Table 18. Summary of Analytical Methods

Constituent	Method Reference	Method Description
Volatile Organic Compounds	Method 624 (1)	Purge and trap, gas chromatography/ mass spectrometry
Volatile Organic Compounds - Halogenated	Method 601 (1)	Purge and trap, gas chromatography/ halogen specific detection (Hall)

(1)U.S. EPA. 1982. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. EPA 600/4-82-057. EPA/EMSL, Cincinnati, Ohio.

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

Table 1. Concentrations of Volatile Organic Compounds (Method 601)

Client: Geraghty & Miller, Inc.
CAA Project No.: 84-1129

Date Samples Received: October 30, 1984
Date Analysis Completed: November 14, 1984

		Concentration - ug/kg (ppb)[1]				
		MW-1 - A Series				
	Sample ID:	0-19	19-33	34-36	49-61	64-76
Compound	CAA ID:	8407177	8407178	8407179	8407180	8407181
(1)	chloromethane					
(2)	vinyl chloride					
(3)	chloroethane					
(4)	methylene chloride					
(5)	1,1-dichloroethylene					
(6)	1,1-dichloroethane	5.				
(7)	trans-1,2-dichloroethylene	25				
(8)	chloroform					
(9)	1,2-dichloroethane					
(10)	1,1,1-trichloroethane	3.				
(11)	carbon tetrachloride					
(12)	bromodichloromethane					
(13)	1,2-dichloropropane					
(14)	trans-1,3-dichloropropene					
(15)	trichloroethylene	20.		1.	1.	
(16)	dibromochloromethane					
(17)	1,1,2-trichloroethane					
(18)	cis-1,3-dichloropropene					
(19)	2-chloroethylvinyl ether					
(20)	bromoform					
(21)	1,1,2,2-tetrachloroethane					
(22)	tetrachloroethylene	300				
(23)	chlorobenzene					
	Detection Limit	0.5	0.7	0.6	0.4	0.6

[1] Concentrations less than the detection limit are left blank.

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

[illegible] Concentrations of Volatile Organic Compounds (Method 501)

Client: [illegible]
CAA Project No.: 84-[illegible]

Date Samples Received: October 30, 1984
Date Analysis Completed: November 14, 1984

		Concentration - ug/kg (ppb)[1]				
		MW-2 - A Series				
	Sample ID:	0-15	19-31	39-46	49-61	64-75
Compound	CAA ID:	8407182	8407183	8407184	8407185	8407186
(1) chloromethane						
(2) vinyl chloride						
(3) chloroethane						
(4) methylene chloride						
(5) 1,1-dichloroethylene						
(6) 1,1-dichloroethane						
(7) trans-1,2-dichloroethylene						
(8) chloroform						
(9) 1,2-dichloroethane						
(10) 1,1,1-trichloroethane						
(11) carbon tetrachloride						
(12) bromodichloromethane						
(13) 1,2-dichloropropane						
(14) trans-1,3-dichloropropene						
(15) trichloroethylene					1.	24
(16) dibromochloromethane						
(17) 1,1,2-trichloroethane						
(18) cis-1,3-dichloropropene						
(19) 2-chloroethylvinyl ether						
(20) bromoform						
(21) 1,1,2,2-tetrachloroethane						
(22) tetrachloroethylene						
(23) chlorobenzene						
Detection Limit		0.5	0.6	0.6	0.8	0.8

[1] Concentrations less than the detection limit are left blank.

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

Table 1. Quantitative Concentrations of Volatile Organic Compounds (Method 601)

Client: Geraghty & Miller, Inc.
CAA Project No.: 84-1104

Date Samples Received: October 30, 1984
Date Analysis Completed: November 14, 1984

Compound	Sample ID: / CAA ID:	C-101 / 8407187	C-102 / 8407188	C-103 / 8407189	C-104 / 8407190	C-105 / 8407191
		Concentration - ug/kg (ppb)[1]				
(1)	chloromethane					
(2)	vinyl chloride					
(3)	chloroethane					
(4)	methylene chloride					
(5)	1,1-dichloroethylene					
(6)	1,1-dichloroethane					2.
(7)	trans-1,2-dichloroethylene					1.
(8)	chloroform					
(9)	1,2-dichloroethane					
	1,1,1-trichloroethane					
(11)	carbon tetrachloride					
(12)	bromodichloromethane					
(13)	1,2-dichloropropane					
(14)	trans-1,3-dichloropropene					
(15)	trichloroethylene		2.			
(16)	dibromochloromethane					
(17)	1,1,2-trichloroethane					
(18)	cis-1,3-dichloropropene					
(19)	2-chloroethylvinyl ether					
(20)	bromoform					
(21)	1,1,2,2-tetrachloroethane					
(22)	tetrachloroethylene		10.			3.
(23)	chlorobenzene					
	Detection Limit	0.6	0.5	0.3	0.4	0.3

[1] Concentrations less than the detection limit are left blank.

PUMPING TEST FORM

PROJECT ____________ WELL ________ LOCATION ____________________ PAGE ____ OF __

SCREEN ____________ M.P. ____________ HT. ABOVE G.S. ________ W.L. MEAS. W/ ____________

r ________ PUMPING WELL ________ Q ________ ORIFICE ________ WEATHER ____________

__ DRAWDOWN __ RECOVERY __ LOCATION SKETCH

TEST START ____________________
END ____________________

DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	

APPENDIX F. DOCUMENT AND DATA MANAGEMENT

This Quality Assurance Program component is designed to assure the reliability and safety of documents and data collected for compliance and/or litigation purposes. This program includes a document numbering system, inventory procedure and a filing system under the supervision of the Project Officer.

Documents included in the program include original field notes and logbooks, field data records, correspondence, sample numbering and descriptions, laboratory data, and chain-of-custody records.

All documents must be dated. Logbook entries made by individuals other than to whom the logbook was assigned must be signed by the person making the entry.

All Field notes and logbooks must be turned over to the project manager at the end of a field assignment.

Chain-of-Custody

All Samples shipped to a laboratory will be accompanied by a Chain-of-Custody Record which identifies the contents. The original Record will accompany the shipment and a copy will be retained by the Project Manager.

If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, an airbill or Bill of Lading will be used. Receipts from post offices and bills of lading will be retained as part of the project documentation.

Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis with a separate Record prepared for the laboratory. Shipping containers will be sealed. The "Courier to the Airport" space on the Chain-of-Custody Record shall be dated and signed.

Field Data Records

Where ordered by the project manager or officer, serialized field data records in the form of individual sheets or bound logbooks will be maintained for each sampling station or location. All in-situ measurements and field observations will be recorded along with all pertinent information necessary to explain and reconstruct sampling operations. Each page of the field data record will be dated and initialed by all individuals making entries on that page or engaged in field activities related to the entry.

All working documents used in evaluating data should contain information sufficient to recall and describe each step of the analysis performed should the analyst be required to testify in subsequent enforcement proceedings. Sufficient information must be

provided to enable others to reconstruct the analysis if the analyst not be available to do so. If the analyst believes it necessary to deviate from a particular analytical method, the deviation shall be justified and documented.

Document Correction and Changes

No serialized documents are to be destroyed even if they are illegible or contain inaccuracies. When an error is made in a project logbook the individual may make corrections by crossing out the error and entering the correct information. Changes made in this manner must be dated and initialed. If an error is discovered on a chain-of-custody record or field document, the person who made the error should correct it whenever possible. Corrections or insertions are made by writing "corrected", the date, and the person's initials beside the correction.

Consistency of Documentation

Before releasing any field data, laboratory data, or data evaluation, the project staff shall assemble and cross-check information on corresponding sample tags, custody records, laboratory reports, analyst logbooks, and field logbooks to ensure that data is consistent throughout the project record.

Project Files

After a particular investigation is completed all documents generated as part of the project are to be assembled in a central file. Staff may retain clean (no handwritten comments) copies of documents for their personal files but only after personally verifying that the original or similar copy is in the project file. The project manager is responsible for assuring the collection, assembly, and inventory of all project documents at the time the project is completed. The file then becomes accountable and any records leaving the file must be signed out.

All documents in an accountable file are to be labeled with a serialized number. The following documents are to be included:

Project Logbooks
Field Data Records
Sample Identification Documents
Chain-of-Custody Records
Analytical logbooks, Lab Data, Calculations
Correspondence
Report Notes and Drafts (including all changes requested by a client)
References
Miscellaneous - photos, maps, drawings, etc.

Draft reports are numbered and accountable. They are stamped FOR REVIEW ONLY, DO NOT DUPLICATE on the cover. The author is responsible for disseminating draft reports for peer review.

APPENDIX G - Cambridge Analytical Associates

QA/QC Procedures

United States
Environmental Protection
Agency

Environmental Monitoring and Support
Laboratory
Cincinnati OH 45268

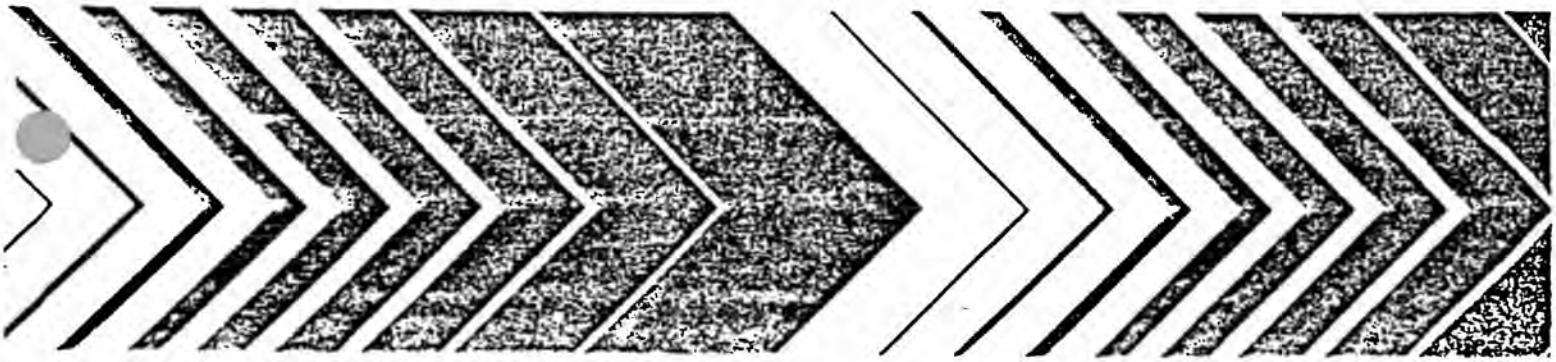
EPA-600/4-79-020
Revised March 1983

100111

Research and Development

EPA

Methods for Chemical Analysis of Water and Wastes



SAMPLE PRESERVATION

Complete and unequivocal preservation of samples, either domestic sewage, industrial wastes, or natural waters, is a practical impossibility. Regardless of the nature of the sample, complete stability for every constituent can never be achieved. At best, preservation techniques can only retard the chemical and biological changes that inevitably continue after the sample is removed from the parent source. The changes that take place in a sample are either chemical or biological. In the former case, certain changes occur in the chemical structure of the constituents that are a function of physical conditions. Metal cations may precipitate as hydroxides or form complexes with other constituents; cations or anions may change valence states under certain reducing or oxidizing conditions; other constituents may dissolve or volatilize with the passage of time. Metal cations may also adsorb onto surfaces (glass, plastic, quartz, etc.), such as, iron and lead. Biological changes taking place in a sample may change the valence of an element or a radical to a different valence. Soluble constituents may be converted to organically bound materials in cell structures, or cell lysis may result in release of cellular material into solution. The well known nitrogen and phosphorus cycles are examples of biological influence on sample composition. Therefore, as a general rule, it is best to analyze the samples as soon as possible after collection. This is especially true when the analyte concentration is expected to be in the low *ug*/l range.

Methods of preservation are relatively limited and are intended generally to (1) retard biological action, (2) retard hydrolysis of chemical compounds and complexes, (3) reduce volatility of constituents, and (4) reduce absorption effects. Preservation methods are generally limited to pH control, chemical addition, refrigeration, and freezing.

The recommended preservative for various constituents is given in Table 1. These choices are based on the accompanying references and on information supplied by various Quality Assurance Coordinators. As more data become available, these recommended holding times will be adjusted to reflect new information. Other information provided in the table is an estimation of the volume of sample required for the analysis, the suggested type of container, and the maximum recommended holding times for samples properly preserved.

TABLE 1

RECOMMENDATION FOR SAMPLING AND PRESERVATION OF SAMPLES ACCORDING TO MEASUREMENT[1]

Measurement	Vol. Req. (ml)	Container[2]	Preservative[3,4]	Holding Time[5]
100 Physical Properties				
Color	50	P,G	Cool, 4°C	48 Hrs.
Conductance	100	P,G	Cool, 4°C	28 Days
Hardness	100	P,G	HNO_3 to pH < 2	6 Mos.
Odor	200	G only	Cool, 4°C	24 Hrs.
pH	25	P,G	None Req.	Analyze Immediately
Residue				
Filterable	100	P,G	Cool, 4°C	7 Days
Non-Filterable	100	P,G	Cool, 4°C	7 Days
Total	100	P,G	Cool, 4°C	7 Days
Volatile	100	P,G	Cool, 4°C	7 Days
Settleable Matter	1000	P,G	Cool, 4°C	48 Hrs.
Temperature	1000	P,G	None Req.	Analyze Immediately
Turbidity	100	P,G	Cool, 4°C	48 Hrs.
200 Metals				
Dissolved	200	P,G	Filter on site HNO_3 to pH < 2	6 Mos.
Suspended	200		Filter on site	6 Mos.[8]
Total	100	P,G	HNO_3 to pH < 2	6 Mos.

TABLE 1 (CONT)

Measurement	Vol. Req. (ml)	Container[2]	Preservative[3,4]	Holding Time[5]
Chromium+6[6]	200	P,G	Cool, 4°C	24 Hrs.
Mercury				
Dissolved	100	P,G	Filter HNO_3 to pH < 2	28 Days
Total	100	P,G	HNO_3 to pH < 2	28 Days
300 Inorganics, Non-Metallics				
Acidity	100	P,G	Cool, 4°C	14 Days
Alkalinity	100	P,G	Cool, 4°C	14 Days
Bromide	100	P,G	None Req.	28 Days
Chloride	50	P,G	None Req.	28 Days
Chlorine	200	P,G	None Req.	Analyze Immediately
Cyanides	500	P,G	Cool, 4°C NaOH to pH >12 0.6g ascorbic acid[8]	14 Days[7]
Fluoride	300	P,G	None Req.	28 Days
Iodide	100	P,G	Cool, 4°C	24 Hrs.
Nitrogen				
Ammonia	400	P,G	Cool,4°C H_2SO_4 to pH < 2	28 Days
Kjeldahl, Total	500	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 Days
Nitrate plus Nitrite	100	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 Days
Nitrate[9]	100	P,G	Cool, 4°C	48 Hrs.
Nitrite	50	P,G	Cool, 4°C	48 Hrs.

TABLE 1 (CONT)

Measurement	Vol. Req. (ml)	Container[2]	Preservative[3,4]	Holding Time[5]
Dissolved Oxygen				
Probe	300	G bottle and top	None Req.	Analyze Immediately
Winkler	300	G bottle and top	Fix on site and store in dark	8 Hours
Phosphorus				
Ortho-phosphate, Dissolved	50	P,G	Filter on site Cool, 4°C	48 Hrs.
Hydrolyzable	50	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 Days
Total	50	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 Days
Total, Dissolved	50	P,G	Filter on site Cool, 4°C H_2SO_4 to pH < 2	24 Hrs.
Silica	50	P only	Cool, 4°C	28 Days
Sulfate	50	P,G	Cool, 4°C	28 Days
Sulfide	500	P,G	Cool, 4°C add 2 ml zinc acetate plus NaOH to pH >9	7 Days
Sulfite	50	P,G	None Req.	Analyze Immediately
400 Organics				
BOD	1000	P,G	Cool, 4°C	48 Hrs.
COD	50	P,G	Cool, 4°C H_2SO_4 to pH < 2	28 Days
Oil & Grease	1000	G only	Cool, 4°C H_2SO_4 to pH < 2	28 Days
Organic carbon	25	P,G	Cool, 4°C H_2SO_4 or HCl to pH < 2	28 Days
Phenolics	500	G only	Cool, 4°C H_2SO_4 to pH <2	28 Days

TABLE 1 (CONT)

Measurement	Vol. Req. (ml)	Container[2]	Preservative[3,4]	Holding Time[5]
MBAS	250	P,G	Cool, 4°C	48 Hrs.
NTA	50	P,G	Cool, 4°C	24 Hrs.

1. More specific instructions for preservation and sampling are found with each procedure as detailed in this manual. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, p. 72–82 (1976) Method D-3370.

2. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.

3. Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

4. When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

5. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of sample under study are stable for the longer time, and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.

6. Should only be used in the presence of residual chlorine.

7. Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before the pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

8. Samples should be filtered immediately on-site before adding preservative for dissolved metals.

9. For samples from non-chlorinated drinking water supplies conc. H_2SO_4 should be added to lower sample pH to less than 2. The sample should be analyzed before 14 days.

Environmental Protection Agency
Support Laboratory
Cincinnati OH 45268

Research and Development

100118

EPA

Test Method

Inductively Coupled Plasma—Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes—Method 200.7

1. Scope and Application

1.1 This method may be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, domestic and industrial wastewaters.

1.2 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interference are taken into account. This is especially true when dissolved solids exceed 1500 mg/L. (See 5.)

1.3 Total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples, appropriate steps *must* be taken to correct for potential interference effects. (See 5.)

1.4 Table 1 lists elements for which this method applies along with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample dependent and as the sample matrix varies, these concentrations may also vary. In time, other elements may be added as more information becomes available and as required.

1.5 Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst is referred to the instructions provided by the manufacturer of the particular instrument.

2. Summary of Method

2.1 The method describes a technique for the simultaneous or sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the

determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 5.1 (and tests for their presence as described in 5.2) should also be recognized and appropriate corrections made.

3. Definitions

3.1 Dissolved — Those elements which will pass through a 0.45 μm membrane filter.

3.2 Suspended — Those elements which are retained by a 0.45 μm membrane filter.

3.3 Total — The concentration determined on an unfiltered sample following vigorous digestion (9.3), or the sum of the dissolved plus suspended concentrations (9.1 plus 9.2).

3.4 Total recoverable — The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid (9.4).

3.5 Instrumental detection limit — The concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength.

3.6 Sensitivity — The slope of the analytical curve, i.e. functional relationship between emission intensity and concentration.

3.7 Instrument check standard — A multielement standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. (See 7.6.1)

3.8 Interference check sample — A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors. (See 7.6.2)

3.9 Quality control sample — A solution obtained from an outside source having known, concentration values to be used to verify the calibration standards. (See 7.6.3)

3.10 Calibration standards — a series of know standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). (See 7.4)

3.11 Linear dynamic range — The concentration range over which the analytical curve remains linear.

3.12 Reagent blank — A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme. (See 7.5.2)

3.13 Calibration blank — A volume of deionized, distilled water acidified with HNO_3 and HCl. (See 7.5.1)

3.14 Method of standard addition — The standard addition technique involves the use of the unknown and the unknown plus a known amount of standard. (See 10.6.1)

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined, however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified (14.7, 14.8 and 14.9) for the information of the analyst.

5. Interferences

5.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

5.1.1 Spectral interferences can be categorized as 1) overlap of a spectral line from another element, 2) unresolved overlap of molecular band spectra, 3) background contribution from continuous or recombination phenomena, and 4) background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer correction of the raw data, requiring the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multielement instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array. Listed in Table 2 are some interference effects for the recommended wavelengths given in Table 1. The data in Table 2 are intended for use only as a rudimentary guide for the indication of potential spectral interferences. For this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

The interference information, which was collected at the Ames Laboratory,[1] is expressed at analyte concentration eqivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interferent element. The suggested use of this information is as follows. Assume that arsenic (at 193.696 nm) is to be determined in a sample containing approximately 10 mg/L of aluminum. According to Table 2, 100 mg/L of aluminum would yield a false signal for arsenic equivalent to approximately 1.3 mg/L. Therefore, 10 mg/L of aluminum would result in a false signal for arsenic equivalent to approximately 0.13 mg/L. The reader is cautioned that other analytical systems may exhibit somewhat different levels of interference than those shown in Table 2, and that the interference effects must be evaluated for each individual system.

Only those interferents listed were investigated and the blank spaces in Table 2 indicate that measurable interferences were not observed for the interferent concentrations listed in Table 3. Generally interferences were discernible if they produced peaks or background shifts corresponding to 2.5% of the peaks generated by the

[1] Ames Laboratory, USDOE, Iowa State University, Ames, Iowa 50011.

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analyte concentrations also listed in Table 3

At present, information on the listed silver and potassium wavelengths are not available but it has been reported that second order energy from the magnesium 383 231 nm wavelength interferes with the listed potassium line at 766 491 nm.

5.1.2 Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aersol flow-rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

5.1.3 Chemical Interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

5.2 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in 5.2.1 through 5.2.4, will ensure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.

5.2.1 Serial dilution—If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrumental detection limit after dilution), an analysis of a dilution should agree within 5 % of the original determination (or within some acceptable control limit (14.3) that has been established for that matrix). If not, a chemical or physical interference effect should be suspected.

5.2.2 Spike addition—The recovery of a spike addition added at a minimum level of 10X the instrumental detection limit (maximum 100X) to the original determination should be recovered to within 90 to 110 percent or within the established control limit for that matrix. If not, a matrix effect should be suspected. The use of a standard addition analysis procedure can usually compensate for this effect. *Caution:* The standard addition technique does not detect coincident spectral overlap. If suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended. (See 5.2.3)

5.2.3 Comparison with alternate method of analysis—When investigating a new sample matrix, comparison tests may be performed with other analytical techniques such as atomic absorption spectrometry, or other approved methodology.

5.2.4 Wavelength scanning of analyte line region—If the appropriate equipment is available, wavelength scanning can be performed to detect potential spectral interferences.

6. Apparatus

6.1 Inductively Coupled Plasma-Atomic Emission Spectrometer.

6.1.1 Computer controlled atomic emission spectrometer with background correction.

6.1.2 Radiofrequency generator.

6.1.3 Argon gas supply, welding grade or better.

6.2 Operating conditions — Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. It is the analyst's responsibility to ensure that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

7. Reagents and standards

7.1 Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. Redistilled acids are acceptable.

7.1.1 Acetic acid, conc (sp gr 1.06).

7.1.2 Hydrochloric acid, conc (sp gr 1.19).

7.1.3 Hydrochloric acid, (1+1). Add 500 mL conc. HCl (sp gr 1.19) to 400 mL deionized, distilled water and dilute to 1 liter.

7.1.4 Nitric acid, conc (sp gr 1.41).

7.1.5 Nitric acid, (1+1). Add 500 mL conc. HNO_3 (sp. gr 1.41) to 400 mL deionized, distilled water and dilute to 1 liter.

7.2 *Dionized, distilled water*: Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized, distilled water for the preparation of all reagents, calibration standards and as dilution water. The purity of this water must be equivalent to ASTM Type II reagent water of Specification D 1193 (14.6).

7.3 *Standard stock solutions* may be purchased or prepared from ultra high purity grade chemicals or metals. All salts must be dried for 1 h at 105°C unless otherwise specified. (CAUTION: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.) Typical stock solution preparation procedures follow.

7.3.1 Aluminum solution, stock, 1 mL = 100 μg Al. Dissolve 0.100 g of aluminum metal in an acid mixture of 4 mL of (1+1) HCl and 1 mL of conc HNO_3 in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask, add an additional 10 mL of (1+1) HCl and dilute to 1 000 mL with deionized, distilled water.

7.3.2 Antimony solution stock, 1 mL = 100 μg Sb. Dissolve 0.2669 g $K(SbO)C_4H_4O_6$ in deionized distilled water, add 10 mL (1+1) HCl and dilute to 1000 mL with deionized, distilled water.

7.3.3 Arsenic solution, stock, 1 mL = 100 µg As Dissolve 0 1320 g of As_2O_3 in 100 mL of deionized, distilled water containing 0 4 g NaOH Acidify the solution with 2 mL conc HNO_3 and dilute to 1,000 mL with deionized, distilled water

7.3.4 Barium solution, stock, 1 mL = 100 µg Ba Dissolve 0 1516 g $BaCl_2$ (dried at 250°C for 2 hrs) in 10 mL deionized, distilled water with 1 mL (1+1) HCl Add 10 0 mL (1+1) HCl and dilute to 1,000 mL with deionized, distilled water.

7.3.5 Beryllium solution, stock, 1 mL = 100 µg Be *Do not dry.* Dissolve 1 966 g $BeSO_4 \cdot 4\ 4H_2O$ in deionized, distilled water add 10.0 mL conc HNO_3 and dilute to 1,000 mL with deionized, distilled water

7.3.6 Boron solution, stock, 1 mL = 100 µg B *Do not dry* Dissolve 0 5716 g anhydrous H_3BO_3 in deionized distilled water dilute to 1,000 mL. Use a reagent meeting ACS specifications, keep the bottle tightly stoppered and store in a desiccator to prevent the entrance of atmospheric moisture.

7.3.7 Cadmium solution, stock, 1 mL = 100 µg Cd Dissolve 0.1142 g CdO in a minimum amount of (1+1) HNO_3 Heat to increase rate of dissolution Add 10 0 mL conc. HNO_3 . and dilute to 1,000 mL with deionized, distilled water.

7.3.8 Calcium solution, stock, 1 mL = 100 µg Ca Suspend 0.2498 g $CaCO_3$ dried at 180°C for 1 h before weighing in deionized, distilled water and dissolve cautiously with a minimum amount of (1+1) HNO_3 Add 10 0 mL conc HNO_3 and dilute to 1,000 mL with deionized, distilled water

7.3.9 Chromium solution, stock, 1 mL = 100 µg Cr Dissolve 0 1923 g of CrO_3 in deionized, distilled water. When solution is complete, acidify with 10 mL conc HNO_3 and dilute to 1,000 mL with deionized, distilled water

7.3.10 Cobalt solution, stock, 1 mL = 100 µg Co Dissolve 0 1000 g of cobalt metal in a minimum amount of (1+1) HNO_3 Add 10 0 mL (1+1) HCl and dilute to 1,000 mL with deionized, distilled water

7.3.11 Copper solution, stock, 1 mL = 100 µg Cu Dissolve 0 1252 g CuO in a minimum amount of (1+1) HNO_3 Add 10 0 mL conc HNO_3 and dilute to 1 000 mL with deionized, distilled water

7.3.12 Iron solution, stock, 1 mL = 100 µg Fe Dissolve 0.1430 g Fe_2O_3 in a warm mixture of 20 mL (1+1) HCl and 2 mL of conc HNO_3 Cool, add an additional 5 mL of conc HNO_3 and dilute to 1000 mL with deionized, distilled water.

7.3.13 Lead solution, stock, 1 mL = 100 µg Pb Dissolve 0 1599 g $Pb(NO_3)_2$ in minimum amount of (1+1) HNO_3. Add 10 0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water

7.3.14 Magnesium solution, stock, 1 mL = 100 µg Mg Dissolve 0 1658 g MgO in a minimum amount of (1+1) HNO_3 Add 10 0 mL conc HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.15 Manganese solution, stock, 1 mL = 100 µg Mn Dissolve 0 1000 g of manganese metal in the acid mixture 10 mL conc. HCl and 1 mL conc HNO_3, and dilute to 1,000 mL with deionized, distilled water

7.3.16 Molybdenum solution, stock, 1 mL = 100 µg Mo Dissolve 0 2043 g $(NH_4)_2MoO_4$ in deionized, distilled water and dilute to 1,000 mL

7.3.17 Nickel solution, stock, 1 mL = 100 µg Ni Dissolve 0.1000 g of nickel metal in 10 mL hot conc. HNO_3, cool and dilute to 1,000 mL with deionized, distilled water

7.3.18 Potassium solution, stock, 1 mL = 100 µg K Dissolve 0.1907 g KCl, dried at 110°C, in deionized, distilled water dilute to 1,000 mL

7.3.19 Selenium solution, stock, 1 mL = 100 µg Se *Do not dry* Dissolve 0 1727 g H_2SeO_3 (actual assay 94.6%) in deionized, distilled water and dilute to 1,000 mL

7.3.20 Silica solution, stock, 1 mL = 100 µg SiO_2 *Do not dry* Dissolve 0 4730 g $Na_2SiO_3 \cdot 9H_2O$ in deionized, distilled water Add 10 0 mL conc HNO_3 and dilute to 1,000 mL with deionized, distilled water

7.3.21 Silver solution, stock, 1 mL = 100 µg Ag Dissolve 0 1575 g $AgNO_3$ in 100 mL of deionized, distilled water and 10 mL conc HNO_3 Dilute to 1,000 mL with deionized, distilled water

7.3.22 Sodium solution, stock, 1 mL = 100 µg Na Dissolve 0 2542 g NaCl in deionized distilled water Add 10 0 mL conc HNO_3 and dilute to 1 000 mL with deionized, distilled water

7.3.23 Thallium solution, stock, 1 mL = 100 µg Tl. Dissolve 0 1303 g $TlNO_3$ in deionized, distilled water Add 10 0 mL conc HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.24 Vanadium solution, stock, 1 mL = 100 µg V Dissolve 0 2297 NH_4VO_3 in a minimum amount of conc. HNO_3 Heat to increase rate of dissolution Add 10 0 mL conc HNO_3 and dilute to 1,000 mL with deionized, distilled water

7.3.25 Zinc solution, stock, 1 mL = 100 µg Zn Dissolve 0 1245 g ZnO in a minimum amount of dilute HNO_3 Add 10 0 mL conc HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.4 Mixed calibration standard solutions—Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks (See 7.4.1 thru 7.4.5) Add 2 mL of (1+1) HCl and dilute to 100 mL with deionized, distilled water (See Notes 1 and 6.) Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards that the elements are compatible and stable Transfer the mixed standard solutions to a FEP fluorocarbon or unused polyethylene bottle for storage Fresh mixed standards should be prepared as needed with the realization that concentration can change on aging Calibration standards must be initially verified using a quality control sample and monitored weekly for stability (See 7 6 3) Although not specifically required, some typical calibration standard combinations follow when using those specific wavelengths listed in Table 1

7.4.1 Mixed standard solution I—Manganese, beryllium, cadmium, lead, and zinc

7.4.2 Mixed standard solution II—Barium, copper, iron, vanadium, and cobalt

7.4.3 Mixed standard solution III Molybdenum, silica, arsenic, and selenium

7.4.4 Mixed standard solution IV - Calcium, sodium, potassium aluminum, chromium and nickel

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7.4.5 Mixed standard solution V—Antimony, boron, magnesium, silver, and thallium

NOTE 1 If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of deionized distilled water and warm the flask until the solution clears Cool and dilute to 100 mL with deionized distilled water For this acid combination the silver concentration should be limited to 2 mg L Silver under these conditions is stable in a tap water matrix for 30 days Higher concentrations of silver require additional HCl

7.5 Two types of blanks are required for the analysis The calibration blank (3 13) is used in establishing the analytical curve while the reagent blank (3.12) is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing

7.5.1 The calibration blank is prepared by diluting 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to 100 mL with deionized, distilled water (See Note 6) Prepare a sufficient quantity to be used to flush the system between standards and samples

7.5.2 The reagent blank must concontain all the reagents and in the same volumes as used in the processing of the samples The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

7.6 In addition to the calibration standards, an instrument check standard (3.7), an interference check sample (3 8) and a quality control sample (3 9) are also required for the analyses

7.6.1 The *instrument check standard* is prepared by the analyst by combining compatible elements at a concentration equivalent to the midpoint of their respective calibration curves (See 12 1.1)

7.6.2 The *interference check sample* is prepared by the analyst in the following manner Select a representative sample which contains minimal concentrations of the analytes of interest by known concentration of interfering elements that will provide an adequate test of the correction factors Spike the sample with the elements of interest at the approximate concentration of either 100 µg/L or 5 times the estimated detection limits given in Table 1. (For effluent samples of expected high concentrations, spike at an appropriate level) If the type of samples analyzed are varied, a synthetically prepared sample may be used if the above criteria and intent are met A limited supply of a synthetic interference check sample will be available from the Quality Assurance Branch of EMSL-Cincinnati (See 12 1 2)

7.6.3 The *quality control sample* should be prepared in the same acid matrix as the calibration standards at a concentration near 1 mg/L and in accordance with the instructions provided by the supplier The Quality Assurance Branch of EMSL-Cincinnati will either supply a quality control sample or information where one of equal quality can be procured (See 12 1 3)

8. Sample handling an preservation

8.1 For the determination of trace elements, contamination and loss are of prime concern Dust in the laboratory environment, impurities in reagents and impurities on laboratory apparatus which the sample contacts are all sources of potential contamination. Sample containers can introduce either positive or negative errors in the measurement of trace elements by (a) contributing contaminants through leaching or surface desorption and (b) by depleting concentrations through adsorption. Thus the collection and treatment of the sample prior to analysis requires particular attention. Laboratory glassware including the sample bottle (whether polyethylene, polypropylene or FEP-fluorocarbon) should be thoroughly washed with detergent and tap water; rinsed with (1+1) nitric acid, tap water, (1+1) hydrochloric acid, tap and finally deionized, distilled water in that order (See Notes 2 and 3)

NOTE 2: Chromic acid may be useful to remove organic deposits from glassware, however, the analyst should be be cautioned that the glassware must be thoroughly rinsed with water to remove the last traces of chromium. This is especially important if chromium is to be included in the analytical scheme A commercial product, NOCHROMIX, available from Godax Laboratories, 6 Varick St., New York, NY 10013, may be used in place of chromic acid Chomic acid should not be used with plastic bottles.

NOTE 3 If it can be documented through an active analytical quality control program using spiked samples and reagent blanks, that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure

8.2 Before collection of the sample a decision must be made as to the type of data desired, that is dissolved, suspended or total, so that the appropriate preservation and pretreatment steps may be accomplished Filtration, acid preservation, etc , are to be performed at the time the sample is collected or as soon as possible thereafter.

8.2.1 For the determination of dissolved elements the sample must be filtered through a 0 45-µm membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus are recommended to avoid possible contamination) Use the first 50-100 mL to rinse the filter flask Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) HNO_3 to a pH of 2 or less. Normally, 3 mL of (1+1) acid per liter should be sufficient to preserve the sample.

8.2.2 For the determination of suspended elements a measured volume of unpreserved sample must be filtered through a 0 45-µm membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservative is required.

8.2.3 For the determination of total or total recoverable elements, the sample is acidified with (1+1) HNO_3 to pH 2 or less as soon as possible, preferable at the time of collection. The sample is not filtered before processing.

9. Sample Preparation

9.1 For the determinations of dissolved elements, the filtered, preserved sample may often be analyzed as received. The acid matrix and concentration of the samples and calibration standards must be the same. (See Note 6) If a precipitate formed upon acidification of the sample or during transit or storage, it must be redissolved before the analysis by adding additional acid and/or by heat as described in 9.3

9.2 For the determination of suspended elements, transfer the membrane filter containing the insoluble material to a 150-mL Griffin beaker and add 4 mL conc HNO_3 Cover the

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beaker with a watch glass and heat gently. The warm acid will soon dissolve the membrane.

Increase the temperature of the hot plate and digest the material. When the acid has nearly evaporated, cool the beaker and watch glass and add another 3 mL of conc HNO_3. Cover and continue heating until the digestion is complete, generally indicated by a light colored digestate. Evaporate to near dryness (2 mL), cool, add 10 mL HCl (1+1) and 15 mL deionized, distilled water per 100 mL dilution and warm the beaker gently for 15 min to dissolve any precipitated or residue material. Allow to cool, wash down the watch glass and beaker walls with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the volume based on the expected concentrations of elements present. This volume will vary depending on the elements to be determined (See Note 6). The sample is now ready for analysis. Concentrations so determined shall be reported as "suspended."

NOTE 4: In place of filtering, the sample after diluting and mixing may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

9.3 For the determination of total elements, choose a measured, volume of the well mixed acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 3 mL of conc. HNO_3. Place the beaker on a hot plate and evaporate to near dryness cautiously, making certain that the sample does not boil and that no area of the bottom of the beaker is allowed to go dry. Cool the beaker and add another 5 mL portion of conc HNO_3. Cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing.) Again, evaporate to near dryness and cool the beaker. Add 10 mL of 1+1 HCl and 15 mL of deionized, distilled water per 100 mL of final solution and warm the beaker gently for 15 min to dissolve any precipitate or residue resulting from evaporation. Allow to cool, wash down the beaker walls and watch glass with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the sample to a predetermined volume based on the expected concentrations of elements present. The sample is now ready for analysis. (See Note 6). Concentrations so determined shall be reported as "total."

NOTE 5: If low determinations of boron are critical, quartz glassware should be use.

NOTE 6: If the sample analysis solution has a different acid concentration from that given in 9.4, but does not introduce a physical interference or affect the analytical result, the same calibration standards may be used.

9.4 For the determination of total recoverable elements, choose a measured volume of a well mixed, acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to the sample and heat on a steam bath or hot plate until the volume has been reduced to near 25 mL making certain the sample does not boil. After this treatment, cool the sample and filter to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the volume to 100 mL and mix. The sample is now ready for analysis. Concentrations so determined shall be reported as "total."

10. Procedure

10.1 Set up instrument with proper operating parameters established in 6.2. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 min of operation prior to calibration.

10.2 Initiate appropriate operating configuration of computer.

10.3 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in 7.4. Flush the system with the calibration blank (7.5.1) between each standard. (See Note 7.) (The use of the average intensity of multiple exposures for both standardization and sample analysis has been found to reduce random error.)

NOTE 7: For boron concentrations greater than 500 µg/L extended flush times of 1 to 2 min may be required.

10.4 Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than ± 5 percent (or the established control limits whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

10.5 Begin the sample run flushing the system with the calibration blank solution (7.5.1) between each sample. (See Note 7.) Analyze the instrument check standard (7.6.1) and the calibration blank (7.5.1) each 10 samples.

10.6 If it has been found that method of standard addition are required, the following procedure is recommended:

10.6.1 The standard addition technique (14.2) involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal thus producing a different slope from that of the calibration standards. It will not correct for additive interference which causes a baseline shift. The simplest version of this technique is the single-addition method. The procedure is as follows. Two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a small volume V_s of a standard analyte solution of concentration c_s. To the second (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration c_x is calculated:

$$c_x = \frac{S_B V_s c_s}{(S_A - S_B) V_x}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and c_s should be chosen so that S_A is roughly twice S_B on the average. It is best if V_s is made much less than V_x, and thus c_s is much greater than c_x, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results from this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear.
2. The chemical form of the analyte added must respond the same as the analyte in the sample.

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3 The interference effect must be constant over the working range of concern
4 The signal must be corrected for any additive interference

11. Calculation

11.1 Reagent blanks (7.5.2) should be subtracted from all samples. This is particularly important for digested samples requiring large quantities of acids to complete the digestion.

11.2 If dilutions were performed, the appropriate factor must be applied to sample values

11.3 Data should be rounded to the thousandth place and all results should be reported in mg/L up to three significant figures

12. Quality Control (Instrumental)

12.1 Check the instrument standardization by analyzing appropriate quality control check standards as follow

12.1.1 Analyze an appropriate instrument check standard (7.6.1) containing the elements of interest at a frequency of 10%. This check standard is used to determine instrument drift. If agreement is not within ±5% of the expected values or within the established control limits, whichever is lower, the analysis is out of control. The analysis should be terminated, the problem corrected, and the instrument recalibrated

Analyze the calibration blank (7.5.1) at a frequency of 10%. The result should be within the established control limits of two standard deviations of the mean value. If not, repeat the analysis two more times and average the three results. If the average is not within the control limit, terminate the analysis, correct the problem and recalibrate the instrument

12.1.2 To verify interelement and background correction factors analyze the interference check sample (7.6.2) at the beginning, end, and at periodic intervals throughout the sample run. Results should fall within the established control limits of 1.5 times the standard deviation of the mean value. If not, terminate the analysis, correct the problem and recalibrate the instrument

12.1.3 A quality control sample (7.6.3) obtained from an outside source must first be used for the initial verification of the calibration standards. A fresh dilution of this sample shall be anlayzed every week thereafter to monitor their stability. If the results are not within ±5% of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

Precision and Accuracy

13.1 In an EPA round robin phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been dosed with various metal concentrates. Table 4 lists the true value, the mean reported value and the mean % relative standard deviation.

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Table 1. Recommended Wavelengths [1] and Estimated Instrumental Detection Limits

Element	Wavelength, nm	Estimated detection limit, µg/L [2]
Aluminum	308.215	45
Arsenic	193.696	53
Antimony	206.833	32
Barium	455.403	2
Beryllium	313.042	0.3
Boron	249.773	5
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Potassium	766.491	see [3]
Selenium	196.026	75
Silica (SiO_2)	288.158	58
Silver	328.068	7
Sodium	588.995	29
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2

[1] The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. (See 5.1.1.).

[2] The estimated instrumental detection limits as shown are taken from "Inductively Coupled Plasma-Atomic Emission Spectroscopy-Prominent Lines," EPA-600/4-79-017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[3] Highly dependent on operating conditions and plasma position.

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Table 2 Analyte Concentration Equivalents (mg/L) Arising From Interferents at the 100 mg/L Level

Analyte	Wavelength, nm	Interferent									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308 215	—	—	—	—	—	—	0 21	—	—	1 4
Antimony	206 833	0 47	—	2 9	—	0 08	—	—	—	25	0 45
Arsenic	193 696	1 3	—	0 44	—	—	—	—	—	—	1 1
Barium	455 403	—	—	—	—	—	—	—	—	—	—
Beryllium	313 042	—	—	—	—	—	—	—	—	0 04	0 05
Boron	249 773	0 04	—	—	—	0 32	—	—	—	—	—
Cadmium	226 502	—	—	—	—	0 03	—	—	0 02	—	—
Calcium	317 933	—	—	0 08	—	0.01	0 01	0 04	—	0 03	0 03
Chromium	267 716	—	—	—	—	0 003	—	0 04	—	—	0 04
Cobalt	228 616	—	—	0 03	—	0.005	—	—	0 03	0 15	—
Copper	324 754	—	—	—	—	0 003	—	—	—	0 05	0.02
Iron	259 940	—	—	—	—	—	—	0.12	—	—	—
Lead	220 353	0 17	—	—	—	—	—	—	—	—	—
Magnesium	279 079	—	0.02	0 11	—	0.13	—	0.25	—	0.07	0.12
Manganese	257.610	0 005	—	0.01	—	0.002	0.002	—	—	—	—
Molybdenum	202 030	0 05	—	—	—	0.03	—	—	—	—	—
Nickel	231 604	—	—	—	—	—	—	—	—	—	—
Selenium	196.026	0 23	—	—	—	0.09	—	—	—	—	—
Silicon	288 158	—	—	0.07	—	—	—	—	—	—	0.01
Sodium	588 995	—	—	—	—	—	—	—	—	0 08	—
Thallium	190 864	0 30	—	—	—	—	—	—	—	—	—
Vanadium	292.402	—	—	0 05	—	0 005	—	—	—	0 02	—
Zinc	213.856	—	—	—	0 14	—	—	—	0.29	—	—

Table 3. Interferent and Analyte Elemental Concentrations Used for Interference Measurements in Table 2

Analytes	(mg/L)	Interferents	(mg/L)
Al	10	Al	1000
As	10	Ca	1000
B	10	Cr	200
Ba	1	Cu	200
Be	1	Fe	1000
Ca	1	Mg	1000
Cd	10	Mn	200
Co	1	Ni	200
Cr	1	Ti	200
Cu	1	V	200
Fe	1		
Mg	1		
Mn	1		
Mo	10		
Na	10		
Ni	10		
Pb	10		
Sb	10		
Se	10		
Si	1		
Tl	10		
V	1		
Zn	10		

Table 4. ICP Precision and Accuracy Data

	Sample # 1			Sample #2			Sample #3		
Element	True Value µg/L	Mean Reported Value µg/L	Mean Percent RSD	True Value µg/L	Mean Reported Value µg/L	Mean Percent RSD	True Value µg/L	Mean Reported Value µg/L	Mean Percent RSD
Be	750	733	6.2	20	20	9.8	180	176	5.2
Mn	350	345	2.7	15	15	6.7	100	99	3.3
V	750	749	1.8	70	69	2.9	170	169	1.1
As	200	208	7.5	22	19	23	60	63	17
Cr	150	149	3.8	10	10	18	50	50	3.3
Cu	250	235	5.1	11	11	40	70	67	7.9
Fe	600	594	3.0	20	19	15	180	178	6.0
Al	700	696	5.6	60	62	33	160	161	13
Cd	50	48	12	2.5	2.9	16	14	13	16
Co	500	512	10	20	20	4.1	120	108	21
Ni	250	245	5.8	30	28	11	60	55	14
Pb	250	236	16	24	30	32	80	80	14
Zn	200	201	5.6	16	19	45	80	82	9.4
Se	40	32	21.9	6	8.5	42	10	8.5	8.3

Not all elements were analyzed by all laboratories

CHLORIDE

Method 325.3 (Titrimetric, Mercuric Nitrate)

STORET NO. 00940

1. Scope and Application
   1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   1.2 The method is suitable for all concentration ranges of chloride content; however, in order to avoid large titration volume, a sample aliquot containing not more than 10 to 20 mg Cl per 50 ml is used.
   1.3 Automated titration may be used.
2. Summary of Method
   2.1 An acidified sample is titrated with mercuric nitrate in the presence of mixed diphenylcarbazone-bromophenol blue indicator. The end point of the titration is the formation of the blue-violet mercury diphenylcarbazone complex.
3. Comments
   3.1 Anions and cations at concentrations normally found in surface waters do not interfere.
   3.2 Sulfite interference can be eliminated by oxidizing the 50 ml of sample solution with 0.5 to 1 ml of H_2O_2.
4. Apparatus
   4.1 Standard laboratory titrimetric equipment including a 1 ml or 5 ml microburet with 0.01 ml graduations.
5. Reagents
   5.1 Standard sodium chloride, 0.025 N: Dissolve 1.4613 g ± 0.0002 g sodium chloride (dried at 600 °C for 1 hour) in chloride-free water in a 1 liter volumetric flask and dilute to the mark. 1 ml = 886.5 μg Cl.
   5.2 Nitric acid, HNO_3 solution (3 + 997)
   5.3 Sodium hydroxide solution, NaOH, (10 g/l)
   5.4 Hydrogen peroxide (30%), H_2O_2
   5.5 Hydroquinone solution (10 g/liter): Dissolve 1 g of purified hydroquinone in water in a 100 ml volumetric and dilute to the mark.
   5.6 Mercuric nitrate titrant (0.141 N): Dissolve 25 g $Hg(NO_3)_2 \cdot H_2O$ in 900 ml of distilled water acidified with 5.0 ml conc. HNO_3 in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard sodium chloride solution (5.1) using procedure 6. Adjust to exactly 0.141 N and check. Store in a dark bottle. A 1.00 ml aliquot is equivalent to 5.00 mg of chloride.
   5.7 Mercuric nitrate titrant (0.025 N): Dissolve 4.2830 g $Hg(NO_3)_2 \cdot H_2O$ in 50 ml of distilled water acidified with 0.5 ml conc. HNO_3 (sp. gr. 1.42) in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard

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sodium chloride solution (5.1) using procedure 6. Adjust to exactly 0.025 N and check. Store in a dark bottle.

5.8 Mercuric nitrate titrant (0.0141 N): Dissolve 2.4200 g $Hg(NO_3)_2 \cdot H_2O$ in 25 ml of distilled water acidified with 0.25 ml of conc. HNO_3 (sp. gr. 1.42) in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard sodium chloride solution (5.1) using procedure 6. Adjust to exactly 0.0141 N and check. Store in a dark bottle. A 1 ml aliquot is equivalent to 500 ug of chloride.

5.9 Mixed indicator reagent: Dissolve 0.5 g crystalline diphenylcarbazone and 0.05 g bromophenol blue powder in 75 ml 95% ethanol in a 100 ml volumetric flask and dilute to the mark with 95% ethanol. Store in brown bottle and discard after 6 months.

5.10 Xylene cyanole FF solution: Dissolve 0.005 g of xylene cyanole FF dye in 95% ethanol or isopropanol in a 100 ml volumetric and dilute to the mark with 95% ethanol or isopropanol.

6. Procedure

6.1 Use 50 ml of sample or an aliquot of sample diluted to 50 ml with distilled water, so that the concentration of chloride does not exceed 20 mg/l aliquot. If the sample or aliquot contains more than 2.5 mg of chloride, use 0.025N mercuric nitrate titrant (5.7) in step 6.6. If the sample or aliquot contains less than 2.5 mg of chloride, use 0.0141N mercuric nitrate titrant (5.8) in step 6.6. Determine an indicator blank on 50 ml chloride-free water using step 6.6. If the sample contains less than 0.1 mg/l of chloride concentrate an appropriate volume to 50 ml.

6.2 Add 5 drops of mixed indicator reagent (5.9), shake or swirl solution.

6.3 If a blue-violet or red color appears add HNO_3 solution (5.2) dropwise until the color changes to yellow.

6.4 If a yellow or orange color forms immediately on addition of the mixed indicator, add NaOH solution (5.3) dropwise until the color changes to blue-violet; then add HNO_3 solution (5.2) dropwise until the color changes to yellow.

6.5 Add 1 ml excess HNO_3 solution (5.2).

6.6 Titrate with 0.025 N mercuric nitrate titrant (5.7) until a blue-violet color persists throughout the solution. See 6.1 for choice of titrant normality. Xylene cyanol FF solution (5.10) may be added with the indicator to sharpen the end point. This will change color shades. Practice runs should be made.

6.7 Additional steps to eliminate particular interferences:

6.7.1 If chromate is present and iron is not present the end point may be difficult to detect.

be an olive-purple color.

6.7.2 If chromate is present at > 100 mg/l and iron is not present, add 2 ml of fresh hydroquinone solution (5.5).

6.7.3 If ferric ion is present use volume containing no more than 2.5 mg of ferric ion or ferric ion plus chromate ion. Add 2 ml fresh hydroquinone solution (5.5).

6.7.4 If sulfite ion is present, add 0.5 ml of H_2O_2 solution (5.4) to 50 ml sample and mix for 1 minute.

7. Calculation

$$\text{mg chloride/l} = \frac{(A - B)N \times 35{,}450}{\text{ml of sample}}$$

where:
A = ml titrant for sample
B = ml titrant for blank
N = normality mercuric nitrate titrant

mg NaCl/l = mg chloride/l x 1.65

8. Precision and Accuracy

8.1 Forty two analysts in eighteen laboratories analyzed synthetic water samples containing exact increments of chloride, with the following results:

Increment as Chloride mg/liter	Precision as Standard Deviation mg/liter	Accuracy: as Bias, %	Accuracy: as Bias, mg/liter
17	1.54	+2.16	+0.4
18	1.32	+3.50	+0.6
91	2.92	+0.11	+0.1
97	3.16	–0.51	–0.5
382	11.70	–0.61	–2.3
398	11.80	–1.19	–4.7

(FWPCA Method Study 1, Mineral and Physical Analyses)

8.2 In a single laboratory (EMSL), using surface water samples at an average concentration of 34 mg Cl/l, the standard deviation was ± 1.0.

8.3 A synthetic unknown sample containing 241 mg/l chloride, 108 mg/l Ca, 82 mg/l Mg, 3.1 mg/l K, 19.9 mg/l Na, 1.1 mg/l nitrate N, 0.25 mg/l nitrite N, 259 mg/l sulfate and 42.5 mg/l total alkalinity (contributed by $NaHCO_3$) in distilled water was analyzed in 10 laboratories by the mercurimetric method, with a relative standard deviation of 3.3% and a relative error of 2.9%.

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water", Standard D512-67, Method A, p 270 (1976).

SULFATE

Method 375.4 (Turbidimetric)

STORET NO. Total 00945

1. Scope and Application
   - 1.1 This method is applicable to drinking and surface waters, domestic and industrial wastes.
   - 1.2 The method is suitable for all concentration ranges of sulfate; however, in order to obtain reliable readings. use a sample aliquot containing not more than 40 mg SO_4/1.
   - 1.3 The minimum detectable limit is approximately 1 mg/1 sulfate.
2. Summary of Method
   - 2.1 Sulfate ion is converted to a barium sulfate suspension under controlled conditions. The resulting turbidity is determined by a nephelometer, filter photometer or spectrophotometer and compared to a curve prepared from standard sulfate solutions.
   - 2.2 Suspended matter and color interfere. Correct by running blanks from which the barium chloride has been omitted.
   - 2.3 Silica in concentrations over 500 mg/1 will interfere.
3. Comments
   - 3.1 Proprietary reagents, such as Hach Sulfaver or equivalent, are acceptable.
   - 3.2 Preserve by refrigeration at 4°C.
4. Apparatus
   - 4.1 Magnetic stirrer, variable speed so that it can be held constant just below splashing. Use identical shape and size magnetic stirring bars.
   - 4.2 Photometer: one of the following which are given in order of preference.
     - 4.2.1 Nephelometer
     - 4.2.2 Spectrophotometer for use at 420 nm with light path of 4 to 5 cm.
     - 4.2.3 Filter photometer with a violet filter having a maximum near 420 nm and a light path of 4 to 5 cm.
   - 4.3 Stopwatch, if the magnetic stirrer is not equipped with an accurate timer.
   - 4.4 Measuring spoon, capacity 0.2 to 0.3 ml.
5. Reagents
   - 5.1 Conditioning reagent: Place 30 ml conc. HCl, 300 ml distilled water, 100 ml 95% ethanol or isopropanol and 75 g NaCl in solution in a container. Add 50 ml glycerol and mix.
   - 5.2 Barium chloride, $BaCl_2$, crystals, 20 to 30 mesh.
   - 5.3 Sodium carbonate solution (approximately 0.05N): Dry 3 to 5 g primary standard Na_2CO_3 at 250°C for 4 hours and cool in a desiccator. Weigh 2.5 ±0.2 g (to the nearest mg), transfer to a 1 liter volumetric flask and fill to the mark with distilled water.

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5.4 Standard sulfate solution (1.00 ml = 100 μg SO_4): Prepare by either 5.4.1 or 5.4.2.

5.4.1 Standard sulfate solution from H_2SO_4

5.4.1.1 Standard sulfuric acid, 0.1N: dilute 3.0 ml conc. H_2SO_4 to 1 liter with distilled water. Standardize versus 40.00 ml of 0.05 N Na_2CO_3 solution (5.3) with about 60 ml distilled water by titrating potentiometrically to pH about 5. Lift electrodes and rinse into beaker. Boil gently for 3–5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH inflection point. Calculate normality using

$$N = \frac{A \times B}{53.00 \times C}$$

where:

A = g Na_2CO_3 weighed into 1 liter
B = ml Na_2CO_3 solution
C = ml acid used to inflection point

5.4.1.2 Standard acid, 0.02 N: Dilute appropriate amount of standard acid, 0.1 N (5.4.1.1) to 1 liter (200.00 ml if 0.1000 N). Check by standardization versus 15 ml of 0.05 N Na_2CO_3 solution (5.3).

5.4.1.3 Place 10.41 ml standard sulfuric acid, 0.02 N (5.4.1.2) in a 100 ml volumetric and dilute to the mark.

5.4.2 Standard sulfate solution from Na_2SO_4: Dissolve 147.9 mg anhydrous Na_2SO_4 in distilled water in a 1 liter volumetric flask and dilute to the mark with distilled water.

6. Procedure

6.1 Formation of barium sulfate turbidity

6.1.1 Place 100 ml sample, or a suitable portion diluted to 100 ml, into a 250 Erlenmeyer flask.

6.1.2 Add exactly 5.0 ml conditioning reagent (5.1).

6.1.3 Mix in the stirring apparatus.

6.1.4 While the solution is being stirred, add a measuring spoonful of $BaCl_2$ crystals (5.2) and begin timing immediately.

6.1.5 Stir exactly 1.0 minutes at constant speed.

6.2 Measurement of barium sulfate turbidity

6.2.1 Immediately after the stirring period has ended, pour solution into absorbance cell.

6.2.2 Measure turbidity at 30 second intervals for 4 minutes.

6.2.3 Record the maximum reading obtained in the 4 minute period.

6.3 Preparation of calibration curve.

6.3.1 Prepare calibration curve using standard sulfate solution (5.4).

6.3.2 Space standards at 5 mg/l increments in the 0–40 mg/l sulfate range.

6.3.3 Above 50 mg/l the accuracy decreases and the suspensions lose stability.

6.3.4 Check reliability of calibration curve by running a standard with every 3 or 4 samples.

6.4 Correction for sample color and turbidity.

6.4.1 Run a sample blank using the procedure 6.1 and 6.2 without the addition of barium chloride (6.1.4).

7. Calculations

7.1 Read mg SO_4 from calibration curve

$$\text{mg } SO_4/l = \frac{\text{mg } SO_4 \times 1{,}000}{\text{ml sample}}$$

8. Precision and Accuracy

8.1 Thirty-four analysts in 16 laboratories analyzed six synthetic water samples containing exact increments of inorganic sulfate with the following results:

Increment as Sulfate mg/liter	Precision as Standard Deviation mg/liter	Accuracy as Bias, %	Accuracy as Bias mg/liter
8.6	2.30	−3.72	−0.3
9.2	1.78	−8.26	−0.8
110	7.86	−3.01	−3.3
122	7.50	−3.37	−4.1
188	9.58	+0.04	+0.1
199	11.8	−1.70	−3.4

(FWPCA Method Study 1, Mineral and Physical Analyses).

8.2 A synthetic unknown sample containing 259 mg/l sulfate, 108 mg/l Ca, 82 mg/l Mg, 3.1 mg/l K, 19.9 mg/l Na, 241 mg/l chloride, 0.250 mg/l nitrite N, 1.1 mg/l nitrate N, and 42.5 mg/l total alkalinity (contributed by $NaHCO_3$) was analyzed in 19 laboratories by the turbidimetric method, with a relative standard deviation of 9.1% and a relative error of 1.2%.

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water", Standard D516–68, Method B, p 430 (1976).

2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 496, Method 427C, (1975).

ALKALINITY

Method 310.1 (Titrimetric, pH 4.5)

STORET NO. 00410

1. Scope and Application
   1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   1.2 The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 ml.
   1.3 Automated titrimetric analysis is equivalent.
2. Summary of Method
   2.1 An unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way.
3. Comments
   3.1 The sample should be refrigerated at 4°C and run as soon as practical. Do not open sample bottle before analysis.
   3.2 Substances, such as salts of weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.
   3.3 For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in:
   Annual Book of ASTM Standards, Part 31, "Water", p 115, D-1067, Method D, (1976).
   3.4 Oil and grease, by coating the pH electrode, may also interfere, causing sluggish response.
4. Apparatus
   4.1 pH meter or electrically operated titrator that uses a glass electrode and can be read to 0.05 pH units. Standardize and calibrate according to manufacturer's instructions. If automatic temperature compensation is not provided, make titration at 25 ±2° C.
   4.2 Use an appropriate sized vessel to keep the air space above the solution at a minimum. Use a rubber stopper fitted with holes for the glass electrode, reference electrode (or combination electrode) and buret.
   4.3 Magnetic stirrer, pipets, flasks and other standard laboratory equipment.
   4.4 Burets, Pyrex 50, 25 and 10 ml.
5. Reagents
   5.1 Sodium carbonate solution, approximately 0.05 N: Place 2.5 ±0.2 g (to nearest mg) Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1 liter volumetric flask and dilute to the mark.

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5.2 Standard acid (sulfuric or hydrochloric), 0.1 N: Dilute 3.0 ml conc H_2SO_4 or 8.3 ml conc HCl to 1 liter with distilled water. Standardize versus 40.0 ml of 0.05 N Na_2CO_3 solution with about 60 ml distilled water by titrating potentiometrically to pH of about 5. Lift electrode and rinse into beaker. Boil solution gently for 3–5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH inflection point. Calculate normality using:

$$N = \frac{A \times B}{53.00 \times C}$$

where:
A = g Na_2CO_3 weighed into 1 liter
B = ml Na_2CO_3 solution
C = ml acid used to inflection point

5.3 Standard acid (sulfuric or hydrochloric), 0.02 N: Dilute 200.0 ml of 0.1000 N standard acid to 1 liter with distilled water. Standardize by potentiometric titration of 15.0 ml 0.05 N Na_2CO_3 solution as above.

6. Procedure

6.1 Sample size

6.1.1 Use a sufficiently large volume of titrant (> 20 ml in a 50 ml buret) to obtain good precision while keeping volume low enough to permit sharp end point.

6.1.2 For < 1000 mg $CaCO_3$/l use 0.02 N titrant

6.1.3 For > 1000 mg $CaCO_3$/l use 0.1 N titrant

6.1.4 A preliminary titration is helpful.

6.2 Potentiometric titration

6.2.1 Place sample in flask by pipetting with pipet tip near bottom of flask

6.2.2 Measure pH of sample

6.2.3 Add standard acid (5.2 or 5.3), being careful to stir thoroughly but gently to allow needle to obtain equilibrium.

6.2.4 Titrate to pH 4.5. Record volume of titrant.

6.3 Potentiometric titration of low alkalinity

6.3.1 For alkalinity of < 20 mg/l titrate 100–200 ml as above (6.2) using a 10 ml microburet and 0.02 N acid solution (5.3).

6.3.2 Stop titration at pH in range of 4.3–4.7, record volume and exact pH. Very carefully add titrant to lower pH exactly 0.3 pH units and record volume.

7. Calculations

7.1 Potentiometric titration to pH 4.5

$$\text{Alkalinity, mg/l } CaCO_3 = \frac{A \times N \times 50{,}000}{\text{ml of sample}}$$

where:

A = ml standard acid

N = normality standard acid

7.2 Potentiometric titration of low alkalinity:

$$\text{Total alkalinity, mg/l } CaCO_3 = \frac{(2B - C) \times N \times 50{,}000}{\text{ml of sample}}$$

where:

B = ml titrant to first recorded pH

C = total ml titrant to reach pH 0.3 units lower

N = normality of acid

8. Precision and Accuracy

8.1 Forty analysts in seventeen laboratories analyzed synthetic water samples containing increments of bicarbonate, with the following results:

Increment as Alkalinity mg/liter, $CaCO_3$	Precision as Standard Deviation mg/liter, $CaCO_3$	Accuracy as Bias, %	Accuracy as Bias, mg/l, $CaCO_3$
8	1.27	+10.61	+0.85
9	1.14	+22.29	+2.0
113	5.28	- 8.19	-9.3
119	5.36	- 7.42	-8.8

(FWPCA Method Study 1, Mineral and Physical Analyses)

8.2 In a single laboratory (EMSL) using surface water samples at an average concentration of 122 mg $CaCO_3$/l, the standard deviation was ±3.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 278, Method 403, (1975).
2. Annual Book of ASTM Standards, Part 31, "Water", p 113, D-1067, Method B, (1976).

100137

NITROGEN, AMMONIA

Method 350.2 (Colorimetric; Titrimetric; Potentiometric – Distillation Procedure)

STORET NO. Total 00610
Dissolved 00608

1. Scope and Application
   1.1 This distillation method covers the determination of ammonia-nitrogen exclusive of total Kjeldahl nitrogen, in drinking, surface and saline waters, domestic and industrial wastes. It is the method of choice where economics and sample load do not warrant the use of automated equipment.
   1.2 The method covers the range from about 0.05 to 1.0 mg NH_3–N/l for the colorimetric procedure, from 1.0 to 25 mg/l for the titrimetric procedure, and from 0.05 to 1400 mg/l for the electrode method.
   1.3 This method is described for macro glassware; however, micro distillation equipment may also be used.
2. Summary of Method
   2.1 The sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid. The ammonia in the distillate can be determined colorimetrically by nesslerization, titrimetrically with standard sulfuric acid with the use of a mixed indicator, or potentiometrically by the ammonia electrode. The choice between the first two procedures depends on the concentration of the ammonia.
3. Sample Handling and Preservation
   3.1 Samples may be preserved with 2 ml of conc. H_2SO_4 per liter and stored at 4°C.
4. Interferences
   4.1 A number of aromatic and aliphatic amines, as well as other compounds, both organic and inorganic, will cause turbidity upon the addition of Nessler reagent, so direct nesslerization (i.e., without distillation), has been discarded as an official method.
   4.2 Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out. Volatile alkaline compounds, such as certain ketones, aldehydes, and alcohols, may cause an off-color upon nesslerization in the distillation method. Some of these, such as formaldehyde, may be eliminated by boiling off at a low pH (approximately 2 to 3) prior to distillation and nesslerization.
   4.3 Residual chlorine must also be removed by pretreatment of the sample with sodium thiosulfate before distillation.

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5. Apparatus

5.1 An all-glass distilling apparatus with an 800–1000 ml flask.

5.2 Spectrophotometer or filter photometer for use at 425 nm and providing a light path of 1 cm or more.

5.3 Nessler tubes: Matched Nessler tubes (APHA Standard) about 300 mm long, 17 mm inside diameter, and marked at 225 mm ±1.5 mm inside measurement from bottom.

5.4 Erlenmeyer flasks: The distillate is collected in 500 ml glass-stoppered flasks. These flasks should be marked at the 350 and the 500 ml volumes. With such marking, it is not necessary to transfer the distillate to volumetric flasks.

6. Reagents

6.1 Distilled water should be free of ammonia. Such water is best prepared by passage through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin. Regeneration of the column should be carried out according to the manufacturer's instructions.

NOTE 1: All solutions must be made with ammonia-free water.

6.2 Ammonium chloride, stock solution: 1.0 ml = 1.0 mg NH_3–N. Dissolve 3.819 g NH_4Cl in distilled water and bring to volume in a 1 liter volumetric flask.

6.3 Ammonium chloride, standard solution: 1.0 ml = 0.01 mg. Dilute 10.0 ml of stock solution (6.2) to 1 liter in a volumetric flask.

6.4 Boric acid solution (20 g/1): Dissolve 20 g H_3BO_3 in distilled water and dilute to 1 liter.

6.5 Mixed indicator: Mix 2 volumes of 0.2% methyl red in 95% ethyl alcohol with 1 volume of 0.2% methylene blue in 95% ethyl alcohol. This solution should be prepared fresh every 30 days.

NOTE 2: Specially denatured ethyl alcohol conforming to Formula 3A or 30 of the U.S. Bureau of Internal Revenue may be substituted for 95% ethanol.

6.6 Nessler reagent: Dissolve 100 g of mercuric iodide and 70 g of potassium iodide in a small amount of water. Add this mixture slowly, with stirring, to a cooled solution of 160 g of NaOH in 500 ml of water. Dilute the mixture to 1 liter. If this reagent is stored in a Pyrex bottle out of direct sunlight, it will remain stable for a period of up to 1 year.

NOTE 3: This reagent should give the characteristic color with ammonia within 10 minutes after addition, and should not produce a precipitate with small amounts of ammonia (0.04 mg in a 50 ml volume).

6.7 Borate buffer: Add 88 ml of 0.1 N NaOH solution to 500 ml of 0.025 M sodium tetraborate solution (5.0 g anhydrous $Na_2B_4O_7$ or 9.5 g $Na_2B_4O_7 \cdot 10H_2O$ per liter) and dilute to 1 liter.

6.8 Sulfuric acid, standard solution: (0.02 N, 1 ml = 0.28 mg NH_3–N). Prepare a stock solution of approximately 0.1 N acid by diluting 3 ml of conc. H_2SO_4 (sp. gr. 1.84) to 1 liter with CO_2-free distilled water. Dilute 200 ml of this solution to 1 liter with CO_2-free distilled water.

NOTE 4: An alternate and perhaps preferable method is to standardize the approximately 0.1 N H_2SO_4 solution against a 0.100 N Na_2CO_3 solution. By proper dilution the 0.02 N acid can then be prepared.

6.8.1 Standardize the approximately 0.02 N acid against 0.0200 N Na_2CO_3 solution. This last solution is prepared by dissolving 1.060 g anhydrous Na_2CO_3, oven-dried at 140°C, and diluting to 1000 ml with CO_2-free distilled water.

6.9 Sodium hydroxide, 1 N: Dissolve 40 g NaOH in ammonia-free water and dilute to 1 liter.

6.10 Dechlorinating reagents: A number of dechlorinating reagents may be used to remove residual chlorine prior to distillation. These include:

a. Sodium thiosulfate (1/70 N): Dissolve 3.5 g $Na_2S_2O_3 \cdot 5H_2O$ in distilled water and dilute to 1 liter. One ml of this solution will remove 1 mg/l of residual chlorine in 500 ml of sample.

b. Sodium arsenite (1/70 N): Dissolve 1.0 g $NaAsO_2$ in distilled water and dilute to 1 liter.

7. Procedure

7.1 Preparation of equipment: Add 500 ml of distilled water to an 800 ml Kjeldahl flask. The addition of boiling chips which have been previously treated with dilute NaOH will prevent bumping. Steam out the distillation apparatus until the distillate shows no trace of ammonia with Nessler reagent.

7.2 Sample preparation: Remove the residual chlorine in the sample by adding dechlorinating agent equivalent to the chlorine residual. To 400 ml of sample add 1 N NaOH (6.9), until the pH is 9.5, checking the pH during addition with a pH meter or by use of a short range pH paper.

7.3 Distillation: Transfer the sample, the pH of which has been adjusted to 9.5, to an 800 ml Kjeldahl flask and add 25 ml of the borate buffer (6.7). Distill 300 ml at the rate of 6–10 ml/min. into 50 ml of 2% boric acid (6.4) contained in a 500 ml Erlenmeyer flask.

NOTE 5: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution.

Dilute the distillate to 500 ml with distilled water and nesslerize an aliquot to obtain an approximate value of the ammonia-nitrogen concentration. For concentrations above 1 mg/l the ammonia should be determined titrimetrically. For concentrations below this value it is determined colorimetrically. The electrode method may also be used.

7.4 Determination of ammonia in distillate: Determine the ammonia content of the distillate titrimetrically, colorimetrically or potentiometrically as described below.

7.4.1 Titrimetric determination: Add 3 drops of the mixed indicator to the distillate and titrate the ammonia with the 0.02 N H_2SO_4, matching the end point against a blank containing the same volume of distilled water and H_3BO_3 solution.

7.4.2 Colorimetric determination: Prepare a series of Nessler tube standards as follows:

ml of Standard 1.0 ml = 0.01 mg NH_3–N	mg NH_3–N/50.0 ml
0.0	0.0
0.5	0.005
1.0	0.01
2.0	0.02
3.0	0.03
4.0	0.04
5.0	0.05
8.0	0.08
10.0	0.10

Dilute each tube to 50 ml with distilled water, add 2.0 ml of Nessler reagent (6.6) and mix. After 20 minutes read the absorbance at 425 nm against the blank. From the values obtained plot absorbance vs. mg NH_3–N for the standard curve. Determine the ammonia in the distillate by nesslerizing 50 ml or an aliquot diluted to 50 ml and reading the absorbance at 425 nm as described above for the standards. Ammonia-nitrogen content is read from the standard curve.

7.4.3 Potentiometric determination: Consult the method entitled Nitrogen, Ammonia: Selective Ion Electrode Method (Method 350.3) in this manual.

7.5 It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least two standards (a high and low) be distilled and compared to similar values on the curve to insure that the distillation technique is reliable. If distilled standards do not agree with undistilled standards the operator should find the cause of the apparent error before proceeding.

8. Calculations

8.1 Titrimetric

$$\text{mg/l } NH_3 - N = \frac{A \times 0.28 \times 1{,}000}{S}$$

where:

A = ml 0.02 N H_2SO_4 used.

S = ml sample.

8.2 Spectrophotometric

$$\text{mg/l } NH_3 - N = \frac{A \times 1{,}000}{D} \times \frac{B}{C}$$

where:

A = mg NH_3–N read from standard curve.

B = ml total distillate collected, including boric acid and dilution.

C = ml distillate taken for nesslerization.

D = ml of original sample taken.

8.3 Potentiometric

$$mg/l\ NH_3 - N = \frac{500}{D} \times A$$

where:

A = mg NH_3-N/l from electrode method standard curve.

D = ml of original sample taken.

9. Precision and Accuracy

9.1 Twenty-four analysts in sixteen laboratories analyzed natural water samples containing exact increments of an ammonium salt, with the following results:

Increment as Nitrogen, Ammonia mg N/liter	Precision as Standard Deviation mgN/liter	Accuracy as Bias, %	Accuracy as Bias, mg N/liter
0.21	0.122	-5.54	-0.01
0.26	0.070	-18.12	-0.05
1.71	0.244	+0.46	+0.01
1.92	0.279	-2.01	-0.04

(FWPCA Method Study 2, Nutrient Analyses)

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2. Annual Book of ASTM Standards, Part 31, "Water", Standard D1426–74, Method A, p 237 (1976).

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United States
Environmental Protection
Agency

Environmental Monitoring and
Support Laboratory
Cincinnati OH 45268

Research and Development

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EPA

Test Methods

Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater

James E. Longbottom and James J. Lichtenberg, Editors

100143

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Test Method

Purgeable Halocarbons—Method 601

1. Scope and Application

1.1 This method covers the determination of 29 purgeable halocarbons. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Bromodichloromethane	32101	75-27-4
Bromoform	32104	75-25-2
Bromomethane	34413	74-83-9
Carbon tetrachloride	32102	56-23-5
Chlorobenzene	34301	108-90-7
Chloroethane	34311	75-00-3
2-Chloroethylvinyl ether	34576	100-75-8
Chloroform	32106	67-66-3
Chloromethane	34418	74-87-3
Dibromochloromethane	32105	124-48-1
1,2-Dichlorobenzene	34536	95-50-1
1,3-Dichlorobenzene	34566	541-73-1
1,4-Dichlorobenzene	34571	106-46-7
Dichlorodifluoromethane	34668	75-71-8
1,1-Dichloroethane	34496	75-34-3
1,2-Dichloroethane	34531	107-06-2
1,1-Dichloroethene	34501	75-35-4
trans-1,2-Dichloroethene	34546	156-60-5
1,2-Dichloropropane	34541	78-87-5
cis-1,3-Dichloropropene	34704	10061-01-5
trans-1,3-Dichloropropene	34699	10061-02-6
Methylene chloride	34423	75-09-2
1,1,2,2-Tetrachloroethane	34516	79-34-5
Tetrachloroethene	34475	127-18-4
1,1,1-Trichloroethane	34506	71-55-6
1,1,2-Trichloroethane	34511	79-00-5
Trichloroethene	39180	79-01-6
Trichlorofluoromethane	34488	75-69-4
Vinyl chloride	39175	75-01-4

1.2 This is a purge and trap gas chromatographic method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identification should be supported by at least one additional qualitative

technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph mass spectrometer (GC MS) conditions appropriate for the qualitative and quantitative confirmation of results for most of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The halocarbons are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the halocarbons are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the halocarbons onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the halocarbons which are then detected with a halide-specific detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.5. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105 °C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with hole in center (Pierce #13075 or equivalent). Detergent wash, rinse cap with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for one hour before use.

5.2 Purge and trap device—The purge and trap device consists of three separate pieces of equipment: the sample purger, trap, and the desorber. Several complete devices are now commercially available.

5.2.1 The sample purger must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15-mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated backing (Section 6.3.3), 7.7 cm of 2,6-diphenylene oxide polymer (Section 6.3.2), 7.7 cm of silica gel, 7.7 gm of coconut charcoal (Section 6.3.1). If it is not necessary to analyze for dichlorodifluoromethane, the charcoal can be eliminated, and the polymer section lengthened to 15 cm. The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should

not be heated higher than 180 °C and the remaining sections should not exceed 220 °C. The desorber design, illustrated in Figure 2, meets these criteria.

5.2.4 The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—8 ft long × 0.1 in ID stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—6 ft long × 0.1 in ID stainless steel or glass, packed with chemically bonded n-octane on Porasil-C (100/120) mesh or equivalent.

5.3.3 Detector—Electrolytic conductivity or microcoulometric. These types of detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope. The electrolytic conductivity detector was used to develop the method performance statements and MDL listed in Tables 1 and 2. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25 μL, 0.006 in ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb. of activated carbon (Filtrasorb-300 or equivalent (Calgon Corp.)).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for one hour. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap Materials

6.3.1 Coconut charcoal (6/10 mesh sieved to 26 mesh), (Barnaby Chaney, CA-580-26 lot # M-2649 or equivalent).

6.3.2 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.3 Methyl silicone packing—3% OV-1 on 60/80 mesh Chromosorb-W or equivalent.

6.3.4 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methyl Alcohol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methyl alcohol using assayed liquids or gas cylinders as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.5.1 Place about 9.8 mL of methyl alcohol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.5.2 Add the assayed reference material:

6.5.2.1 Liquids—Using a 100-μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the six halocarbons that boil below 30 °C (bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methyl alcohol meniscus. Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve into the methyl alcohol).

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at −10 to −20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the six gases and 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicate a problem.

6.6 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methyl alcohol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Sections 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Quality control check standards that can be used to determine the accuracy of calibration standards will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, in Cincinnati, Ohio.

7. Calibration

7.1 Assemble a purge and trap device that meets the specifications in Section 6.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL min. Prior to use, daily condition traps 10 minutes while backflushing at 180 °C.

7.2 Connect the purge and trap device to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate parameters equivalent to those in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 μL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-μL syringe with a 0.006 inch ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the method detection limit (See Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards can be stored up to 24 hours, if held in sealed vials with zero headspace as described in Section 9.2. If not so stored, they must be discarded after one hour.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (≤10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3.3 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve or calibration factor must be prepared for that parameter.

7.4 Internal standard calibration procedure. To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compounds recommended for use as surrogate spikes in Section 8.7 have been used successfully as internal standards, because of their generally unique retention times.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration of 15 μg/mL of each internal standard compound. The addition of 10 μL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 μg/L.

7.4.3 Analyze each calibration standard, according to Section 10, adding 10 μL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using equation 1.

$$\text{Eq. 1 } RF = (A_s C_{is})/(A_{is} C_s)$$

where:

A_s = Response for the parameter to be measured.
A_{is} = Response for the internal standard.
C_{is} = Concentration of the internal standard.
C_s = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (≤10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4.4 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within accuracy and precision limits expected of the method.

8.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of the rapid advances that are occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications are made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 The laboratory must spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. This procedure is described in Section 8.4.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 Select a representative spike concentration for each compound to be measured. Using stock standards, prepare a quality control check sample concentrate in methyl alcohol 500 times more concentrated than the selected concentrations. Quality control check sample concentrates, appropriate for use with this method, will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

8.2.2 Using a syringe, add 10 μL of the check sample concentrate to each of a minimum of four 5-mL aliquots of reagent water. A representative waste-

water may be used in place of the reagent water, but one or more additional aliquots must be analyzed to determine background levels, and the spike level must exceed twice the background level for the test to be valid. Analyze the aliquots according to the method beginning in Section 10.

8.2.3 Calculate the average percent recovery, (R), and the standard deviation of the percent recovery (s), for the results. Wastewater background corrections must be made before R and s calculations are performed.

8.2.4 Using Table 2, note the average recovery (X) and standard deviation (p) expected for each method parameter. Compare these to the calculated values for R and s. If $s > 2p$ or $|X - R| > 2p$, review potential problem areas and repeat the test.

8.3 The analyst must calculate method performance criteria and define the performance of the laboratory for each spike concentration and parameter being measured.

8.2.5 The U.S. Environmental Protection Agency plans to establish performance criteria for R and s based upon the results of interlaboratory testing. When they become available, these criteria must be met before any samples may be analyzed.

8.3.1 Calculate upper and lower control limts for method performance:

Upper Control Limit (UCL) = R + 3s
Lower Control Limit (LCL) = R − 3s

where R and s are calculated as in Section 8.2.3. The UCL and LCL can be used to construct control charts[7] that are useful in observing trends in performance. The control limits above must be replaced by method performance criteria as they become available from the U.S. Environmental Protection Agency.

8.3.2 The laboratory must develop and maintain separate accuracy statements of laboratory performance for wastewater samples. An accuracy statement for the method is defined as R ± s. The accuracy statement should be developed by the analysis of four aliquots of wastewater as described in Section 8.2.2, followed by the calculation of R and s. Alternately, the analyst may use four wastewater data points gathered through the requirement for continuing quality control in Section 8.4. The accuracy statements should be updated regularly.[7]

8.4 The laboratory is required to collect a portion of their samples in duplicate to monitor spike recoveries. The frequency of spiked sample analysis must be at least 10% of all samples or one sample per month, whichever is greater. One aliquot of the sample must be spiked and analyzed as described in Section 8.2. If the recovery for a particular parameter does not fall within the control limits for method performance, the results reported for that parameter in all samples processed as part of the same set must be qualified as described in Section 11.3. The laboratory should monitor the frequency of data so qualified to ensure that it remains at or below 5%.

8.5 Each day, the analyst must demonstrate through the analysis of reagent water, that interferences from the analytical system are under control.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should maintain constant surveillance of both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard and blank with surrogate halocarbons. A combination of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane is recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as above, add a volume to give 7500 μg of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix and dilute to volume (15 ng μL). If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2). Add 10 μL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis.

9. Sample Collection, Preservation, and Handling

9.1 All samples must be iced or refrigerated from the time of collection until extraction. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottle just prior to shipping to the sampling site. USEPA methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for one minute. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.

10. Sample Extraction and Gas Chromatography

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this Table are estimated retention times and method detection limits that can be achieved by this method. An example of the separations achieved by Column 1 is shown in Figure 5. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the device to purge. Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the

syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 µL of the surrogate spiking solution (8.7) and 10.0 µL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 11.0 ± .1 minutes at ambient temperature.

10.7 After the 11-minute purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for four minutes. If rapid heating of the trap cannot be achieved, the gas chromatographic column must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if poor peak geometry or random retention time problems persist) instead of the initial program temperature of 45 °C.

10.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for four minutes recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately seven minutes turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool the trap is ready for the next sample.

10.10 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for the peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of material from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure was used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and equation 2.

Eq. 2.

Concentration $\mu g/L = (A_s C_{is})/(A_{is})(RF)$

where:

A_s = Response for the parameter to be measured.

A_{is} = Response for the internal standard.

C_s = Concentration of the internal standard.

11.2 Report results in micrograms per liter. When duplicate and spiked samples are analyzed, report all data obtained with the sample results.

11.3 For samples processed as part of a set where the spiked sample recovery falls outside of the control limits which were established according to Section 8.3, data for the affected parameters must be labeled as suspect.

12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[9] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method is recommended for use in the concentration range from the MDL up to 1000 × MDL. Direct aqueous injection techniques should be used to measure concentration levels above 1000 × MDL.

12.3 In a single laboratory (Monsanto Research), using reagent water and wastewaters spiked at or near background levels, the average recoveries presented in Table 2 were obtained[9]. The standard deviation of the measurement in percent recovery is also included in Table 2[9].

12.4 The U.S. Environmental Protection Agency is in the process of conducting an interlaboratory method study to fully define the performance of this method.

References

1. See Appendix A.
2. Bellar, T.A., and Lichtenberg, J.J. *Journal American Water Works Association, 66,* 739, (1974).
3. Bellar, T.A., and Lichtenberg, J.J. "Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds," Proceedings from Symposium on Measurement of Organic Pollutants in Water and Wastewater, American Society for Testing and Materials, STP 686, C.E. Van Hall, editor, 1978.
4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, Aug. 1977.
5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).
6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.
7. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," EPA-600/4-79-019, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory—Cincinnati, Ohio 45268, March 1979.
8. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, U.S. Environmental Protection Agency.

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

Table 2. (cont'd) Concentrations of Volatile Organic Compounds (Method 601)

Client: Geraghty & Miller, Inc.
CAA Project No.: 84-1295

Date Samples Received: October 30, 1984
Date Analysis Completed: November 14, 1984

Compound	Sample ID: / CAA ID:	Concentration - ug/kg (ppb)[1] C-106 8407192	C-107 8407193	C-108 8407194	C-109 8407195	C-110 8407196
(1)	chloromethane					
(2)	vinyl chloride					
(3)	chloroethane					
(4)	methylene chloride					
(5)	1,1-dichloroethylene					
(6)	1,1-dichloroethane	4.	32	3.	3.	
(7)	trans-1,2-dichloroethylene		64	1.		
(8)	chloroform					
(9)	1,2-dichloroethane					
(10)	1,1,1-trichloroethane		240			
(11)	carbon tetrachloride					
(12)	bromodichloromethane					
(13)	1,2-dichloropropane					
(14)	trans-1,3-dichloropropene					
(15)	trichloroethylene		8.	1.	1.	
(16)	dibromochloromethane					
(17)	1,1,2-trichloroethane					
(18)	cis-1,3-dichloropropene					
(19)	2-chloroethylvinyl ether					
(20)	bromoform					
(21)	1,1,2,2-tetrachloroethane					
(22)	tetrachloroethylene	4.	27	4.	5.	
(23)	chlorobenzene					
	Detection Limit	0.4	0.4	0.4	0.5	0.4

[1] Concentrations less than the detection limit are left blank.

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

Table 2. (cont'd) Concentrations of Volatile Organic Compounds (Method 601)

Client: Gerrity & Miller, Inc.
CAA Project No.: 84-1295

Date Samples Received: October 30, 1984
Date Analysis Completed: November 14, 1984

Compound	Sample ID: / CAA ID:	C-111 / 8407197	C-112 / 8407198	C-113 / 8407199	C-114 / 8407200	C-115 / 8407201
		Concentration - ug/kg (ppb)[1]				
(1)	chloromethane					
(2)	vinyl chloride					
(3)	chloroethane					
(4)	methylene chloride					
(5)	1,1-dichloroethylene			6.		
(6)	1,1-dichloroethane			2.		
(7)	trans-1,2-dichloroethylene			100.		
(8)	chloroform					
(9)	1,2-dichloroethane					
(10)	1,1,1-trichloroethane			54.		
(11)	carbon tetrachloride					
(12)	bromodichloromethane					
(13)	1,2-dichloropropane					
(14)	trans-1,3-dichloropropene					
(15)	trichloroethylene			42.		
(16)	dibromochloromethane					
(17)	1,1,2-trichloroethane					
(18)	cis-1,3-dichloropropene					
(19)	2-chloroethylvinyl ether					
(20)	bromoform					
(21)	1,1,2,2-tetrachloroethane					
(22)	tetrachloroethylene			6.		4.
(23)	chlorobenzene					
	Detection Limit	0.4	0.5	0.6	0.4	0.5

[1] Concentrations less than the detection limit are left blank.

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

Table 2. (cont'd) Concentrations of Volatile Organic Compounds (Method 601)

Client: Geraghty & Miller, Inc.
CAA Project No.: 84-1295

Date Samples Received: October 30, 1984
Date Analysis Completed: November 14, 1984

		Concentration - ug/kg (ppb)[1]				
	Sample ID:	C-115	C-117	C-118	C119	C-120
Compound	CAA ID:	8407202	8407203	8407204	8407205	8407206
(1)	chloromethane					
(2)	vinyl chloride					
(3)	chloroethane					
(4)	methylene chloride					
(5)	1,1-dichloroethylene		16.			
(6)	1,1-dichloroethane	7.	8.			
(7)	trans-1,2-dichloroethylene	7.	27.			
(8)	chloroform		11.			
	1,2-dichloroethane					
	1,1,1-trichloroethane	23.	220.			
(11)	carbon tetrachloride					
(12)	bromodichloromethane					
(13)	1,2-dichloropropane					
(14)	trans-1,3-dichloropropene					
(15)	trichloroethylene	2.	150.			
(16)	dibromochloromethane					
(17)	1,1,2-trichloroethane					
(18)	cis-1,3-dichloropropene					
(19)	2-chloroethylvinyl ether					
(20)	bromoform					
(21)	1,1,2,2-tetrachloroethane					
(22)	tetrachloroethylene	4.	56.			
(23)	chlorobenzene					
	Detection Limit	0.7	0.4	0.5	0.5	0.6

[1] Concentrations less than the detection limit are left blank.

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

Table 5. Concentrations of Priority Pollutant Volatile Organic Compounds (Method 624)

Client: Geraghty & Miller, Inc.
CAA Project No.: 84-1295

Date Samples Received: October 30, 1984
Date Analysis Completed: November 13, 1984

	Concentration - ug/l (ppb)[1]			
Sample ID:	Well 7	Well 7A	Well 9	Well 9A
Compound CAA ID:	8407207	8407208	8407209	8407210
(1) chloromethane				
(2) bromomethane				
(3) vinyl chloride				
(4) chloroethane				
(5) methylene chloride				
(6) 1,1-dichloroethylene				
(7) 1,1-dichloroethane				
(8) trans-1,2-dichloroethylene		13		
(9) chloroform				
(10) 1,2-dichloroethane				
(11) 1,1,1-trichloroethane		TR(9)		
(12) carbon tetrachloride				
(13) bromodichloromethane				
(14) acrylonitrile				
(15) acrolein				
(16) 1,2-dichloropropane				
(17) trans-1,3-dichloropropene				
(18) trichloroethylene	TR(7)	660	120	530
(19) chlorodibromomethane				
(20) 1,1,2-trichloroethane				
(21) benzene				
(22) cis-1,3-dichloropropene				
(23) 2-chloroethylvinyl ether				
(24) bromoform				
(25) 1,1,2,2-tetrachloroethane				
(26) tetrachloroethylene				
(27) toluene				
(28) chlorobenzene				
(29) ethylbenzene				
(30) total xylenes				
Detection Limit	1.	1.	1.	1.

[1] Concentrations less than the detection limit are left blank. Concentrations between 1 and 10 times the detection limit are listed as trace levels (TR). Detection limits for acrolein and acrylonitrile are 100 and 10 times the nominal detection limit respectively.

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

Table 5. (cont'd) Concentrations of Priority Pollutant Volatile Organic Compounds (Method 624)

Client: Dames & Moore, Inc.
CAA Project No.: 84-1295

Date Samples Received: October 30, 1984
Date Analysis Completed: November 13, 1984

		Concentration - ug/l (ppb)[1]				
	Sample ID:	Well 10	Well 10A	Well 10B	Well Nus-1	Trip Blank
Compound	CAA ID:	8407211	8407212	8407213	8407214	8407215
(1)	chloromethane					
(2)	bromomethane					
(3)	vinyl chloride					
(4)	chloroethane					
(5)	methylene chloride					
(6)	1,1-dichloroethylene					
(7)	1,1-dichloroethane					
(8)	trans-1,2-dichloroethylene		62			
(9)	chloroform					
(10)	1,2-dichloroethane					
(11)	1,1,1-trichloroethane					
(12)	carbon tetrachloride					
(13)	bromodichloromethane					
(14)	acrylonitrile					
(15)	acrolein					
(16)	1,2-dichloropropane					
(17)	trans-1,3-dichloropropene					
(18)	trichloroethylene	210	TR(5)		130	
(19)	chlorodibromomethane					
(20)	1,1,2-trichloroethane					
(21)	benzene					
(22)	cis-1,3-dichloropropene					
(23)	2-chloroethylvinyl ether					
(24)	bromoform					
(25)	1,1,2,2-tetrachloroethane					
(26)	tetrachloroethylene		TR(3)			
(27)	toluene					
(28)	chlorobenzene					
(29)	ethylbenzene					
(30)	total xylenes					
	Detection Limit	1.	1.	1.	1.	1.

[1] Concentrations less than the detection limit are left blank. Concentrations between 1 and 10 times the detection limit are listed as trace levels (TR). Detection limits for acrolein and acrylonitrile are 100 and 10 times the nominal detection limit respectively.

Table 4. Results of Chemical Analyses

Client: [illegible] & Miller, Inc.
Project No.: 84-1295

Constituent	Client ID:	Well 7	Well 7A	Well 9	Well 9A	Well 10	Well 10A	Well 10B	NUS -1
	CAA ID:	8407207	8407208	8407209	8407210	8407211	8407212	8407213	8407214
Fe (mg/l)		<0.025	0.82	4.3	<0.025	<0.025	0.036	1.9	<0.025
K (mg/l)		19	2.1	1.9	<1	<1	2.0	3.1	1.4
Alkalinity (mg/l)		51	74	230	120	110	260	350	29
Chloride (mg/l)		12	22	31	8.5	8.5	31	12	10
Sulfate (mg/l)		58	99	83	38	85	38	220	15
Ammonia (mg/l)		0.76	0.21	0.32	0.24	0.27	0.36	0.21	1.9
pH		7.45	6.75	6.88	7.54	7.46	7.26	7.05	9.82
Conductance (umhos/cm @ 25° c)		220	240	500	270	240	590	590	47

4. QUALITY ASSURANCE DOCUMENTATION

Certification

This work has been checked for accuracy by the following staff personnel:

Director, Organic
Chemistry Laboratory

David L. Fiest

Director, Inorganic
Chemistry Laboratory

Keith A. Hausknecht

Cambridge Analytical Associates
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FINAL REPORT

Geraghty & Miller, Inc.
7 Atlantic Street
Hackensack, NJ 07061
Attn: Mr. Dan Nachman

PROJECT NUMBER: J8310L2

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

REPORT NUMBER: 84-1346

PREPARED BY: Edward A. Lawler

DATE PREPARED: December 17, 1984

TABLE OF CONTENTS

1. INTRODUCTION

This report summarizes results of chemical analyses performed on samples received by CAA on November 9, 1984. Analytical methods employed for these analyses are described in Section 2 and results are presented in Section 3. The last section contains quality control data and certifications supporting the analytical results.

2. ANALYTICAL METHODS

Analytical methods utilized for sample analysis are summarized in Table 1.

3. RESULTS

Results of analyses are presented in Tables 2 & 3.

Table 1. Summary of Analytical Methods

Constituent	Method Reference	Method Description
Iron (Fe)	Method 200.7 (1)	ICP
Potassium (k)	Method 200.7 (1)	ICP
Alkalinity	Method 310.1 (1)	Titrimetric, pH 4.5
Chloride	Method 325.3 (1)	Titrimetric, mercuric nitrate
Sulfate	Method 375.4 (1)	Turbidimetric
Ammonia	Method 350.2 (1)	Colorimetric, distillation
pH	Method 150.1 (1)	Electrometric
Conductivity	Method 120.1 (1)	Specific conductance
Volatile Organic Compounds	Method 624 (2)	Purge and trap, gas chromatography/ mass spectrometry

(1)U.S. EPA. 1979. Methods for Chemical Analysis of Water and Wastes. EPA 600/4-79-020 (Revised, March 1983). EPA/EMSL, Cincinnati, Ohio.

(2)U.S. EPA. 1982. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater. EPA 600/4-82-057. EPA/EMSL, Cincinnati, Ohio.

ICP - Inductively coupled argon plasma emission spectroscopy

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

Table 2. Concentrations of Priority Pollutant Volatile Organic Compounds (Method 624)

Client: Geraghty & Miller, Inc.
CAA Project No.: 84-1346

Date Samples Received: November 9, 1984
Date Analysis Completed: November 18, 1984

		Concentration - ug/l (ppb)[1]			
Compound	Sample ID: / CAA ID:	PW-1 / 8407481	MW-1 / 8407482	MW-2 / 8407483	MW-3 / 8407484
(1)	chloromethane				
(2)	bromomethane				
(3)	vinyl chloride				
(4)	chloroethane				280
(5)	methylene chloride				TR(2)
(6)	1,1-dichloroethylene				14
(7)	1,1-dichloroethane				950
(8)	trans-1,2-dichloroethylene				200
(9)	chloroform				
(10)	1,2-dichloroethane				
(11)	1,1,1-trichloroethane				1,100
(12)	carbon tetrachloride				
(13)	bromodichloromethane				
(14)	acrylonitrile				
(15)	acrolein				
(16)	1,2-dichloropropane				
(17)	trans-1,3-dichloropropene				
(18)	trichloroethylene	51	180		270
(19)	chlorodibromomethane				
(20)	1,1,2-trichloroethane				
(21)	benzene				
(22)	cis-1,3-dichloropropene				
(23)	2-chloroethylvinyl ether				
(24)	bromoform				
(25)	1,1,2,2-tetrachloroethane				
(26)	tetrachloroethylene				210
(27)	toluene				
(28)	chlorobenzene				
(29)	ethylbenzene				
(30)	total xylenes				
	Detection Limit	1.	1.	1.	1.

[1] Concentrations less than the detection limit are left blank. Concentrations between 1 and 10 times the detection limit are listed as trace levels (TR). Detection limits for acrolein and acrylonitrile are 100 and 10 times the nominal detection limit respectively.

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

Table 2 (cont'd) Concentrations of Priority Pollutant Volatile Organic Compounds (Method 624)

Client: Geraghty & Miller, Inc.
CAA Project No.: 84-1346

Date Samples Received: November 9, 1984
Date Analysis Completed: November 18, 1984

Compound	Sample ID: / CAA ID:	Concentration - ug/l (ppb)[1] MW-4 8407485	MW-5 8407486
(1)	chloromethane		
(2)	bromomethane		
(3)	vinyl chloride		
(4)	chloroethane		
(5)	methylene chloride		
(6)	1,1-dichloroethylene		
(7)	1,1-dichloroethane		
(8)	trans-1,2-dichloroethylene		
(9)	chloroform		
(10)	1,2-dichloroethane		
(11)	1,1,1-trichloroethane		
(12)	carbon tetrachloride		
(13)	bromodichloromethane		
(14)	acrylonitrile		
(15)	acrolein		
(16)	1,2-dichloropropane		
(17)	trans-1,3-dichloropropene		
(18)	trichloroethylene	52.	
(19)	chlorodibromomethane		
(20)	1,1,2-trichloroethane		
(21)	benzene		
(22)	cis-1,3-dichloropropene		
(23)	2-chloroethylvinyl ether		
(24)	bromoform		
(25)	1,1,2,2-tetrachloroethane		
(26)	tetrachloroethylene		
(27)	toluene		
(28)	chlorobenzene		
(29)	ethylbenzene		
(30)	total xylenes		
	Detection Limit	1.	1.

[1] Concentrations less than the detection limit are left blank. Concentrations between 1 and 10 times the detection limit are listed as trace levels (TR). Detection limits for acrolein and acrylonitrile are 100 and 10 times the nominal detection limit respectively.

Table 3. Results of Chemical Analyses

Client: Geraghty & Miller, Inc.
Project No.: 84-1346

Constituent	Client ID: / CAA ID:	MW-1 8407482	MW-3 8407484
Fe (mg/l)		0.19	1.3
K (mg/l)		1.1	13
Alkalinity (mg/l)		104	81
Chloride (mg/l as $CaCO_3$)		15	29
Sulfate (mg/l)		22	25
Ammonia-N (mg/l)		<0.05	0.68
pH		7.42	6.83
Conductance (umhos/cm @ 25° c)		230	270

4. QUALITY ASSURANCE DOCUMENTATION

Certification

This work has been checked for accuracy by the following staff personnel:

Director, Organic
Chemistry Laboratory

David L. Fiest

Director, Inorganic
Chemistry Laboratory

Keith A. Hausknecht

APPENDIX D

Recovery and Pumping Test Data

AVX-PW

last p.l. = 36.20

100166

PUMPING TEST FORM

PROJECT J8310-2 WELL AW-PW LOCATION inside plant PAGE 1 OF 2

SCREEN 65-80 M.P. hole in pump house HT. ABOVE G.S. _____ W.L. MEAS. W/ M-scope

r 0 PUMPING WELL AW-PW Q _____ ORIFICE _____ WEATHER 40's 60's - hot showers

___DRAWDOWN X RECOVERY ___LOCATION SKETCH TEST START 11/9/84 9PM

END 11/11 about noon 2PM 8PM

DATE TIME	t	HELD	WET	D.T.W.	36.20 RESIDUAL			MANO-METER	Q	WATER TEMP.	
9PM	0	25	-1.11	30.17	6.03						
	½	30	-1.12	30.34	5.32						
	1	30	-1.29	30.91	5.59						
	1½	30	-1.76	31.76	4.44						
	2	30	-1.25	31.35	4.35						
	2½	30	-1.27	31.40	4.30						
	3	30	-1.24	31.84	4.36						
	3½	30	+1.24	31.84	4.36						
	4	30	+1.82	31.82	4.38						
	4½	30	+1.80	31.80	4.40						
	5	30	+1.81	31.81	4.39						
	6	30	+1.79	31.79	4.41						
	7	30	+1.76	31.76	4.46						
	8	30	-.72	31.72	4.48						
	9	30	-.71	31.71	4.49						
	10	30	+.71	31.71	4.49						
	12	30	+.69	31.69	4.51						
	14	30	-.67	31.67	4.53						
	16	30	+.65	31.65	4.55						
	18	30	+.66	31.66	4.54						
	20	30	+1.65	31.65	4.55						
	22	30	+1.62	31.62	4.58						
	24	30	-.63	31.63	4.57						
	26	30	+1.62	31.62	4.58						
	28	30	+1.60	31.60	4.60						
	30	30	+1.61	31.61	4.59						
	35	30	+1.57	31.57	4.63						
	40	30	+1.56	31.56	4.64						
	45	30	+1.57	31.57	4.63						
	50	30	+1.54	31.54	4.66						
	55	30	+1.54	31.54	4.66						
10PM	60	30	+1.62	31.62	4.58						
	70	30	+1.51	31.51	4.70						
	80	30	+1.49	31.49	4.71						
	90	30	+1.47	31.47	4.73						
	100	30	+1.47	31.47	4.73						
	110	30	+1.47	31.47	4.73						
11PM	120	30	1.47	31.47	4.73						

PUMPING TEST FORM

PROJECT 831062 WELL AYX-PN LOCATION ____ PAGE 2 OF 2

SCREEN ____ M.P. ____ HT. ABOVE G.S. ____ W.L. MEAS. W/ ____

r ____ PUMPING WELL ____ Q ____ ORIFICE ____ WEATHER ____

___ DRAWDOWN X RECOVERY ___ LOCATION SKETCH TEST START ____ END ____

DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
	140	30	+1.46	31.46	4.74						
	160	30	+1.44	31.44	4.76						
MIDNIGHT	180	30	+1.42	31.42	4.78						
	210	30	+1.42	31.42	4.78						
1AM	240	30	+1.40	31.40	4.80						
	270	30	+1.38	31.38	4.82						
2	300	30	+1.37	31.37	4.83						
	330	30	+1.37	31.37	4.83						
3AM	360	30	+1.35	31.35	4.85						
4	420	30	+1.35	31.35	4.85						
5	480	30	+1.34	31.34	4.86						
6	540	30	+1.34	31.34	4.86						
7	600	30	1.34	31.34	4.86						
[illegible]	660	30	+1.32	31.32	4.88						
9AM	720	30	+1.32	31.32	4.88						
11	840	30	+1.32	31.32	4.88						
1PM	960	30	+1.28	31.28	4.92						
3	1080	30	+1.28	31.28	4.92						
5	1200	30	+1.27	31.27	4.93						
7	1320	30	+1.26	31.26	4.94						
9PM	1440	30	+1.23	31.23	4.97						
11	1560	30	+1.19	31.19	5.01						11:41 train went by
1AM	1680	30	+1.17	31.17	5.03						check recovery for blip
[illegible]	1800	30	+1.15	31.15	5.05						2:41 "
5	1920	30	+1.09	31.09	5.11						6:21
7	2040	30	+1.11	31.11	5.09						8:54
9	2160	30	+1.14	31.14	5.06						
11	2280	30	+1.15	31.15	5.05						10:24 - train passes
1PM	2400	30	+1.14	31.14	5.06						
3	2520	30	+1.14	31.14	5.06				city		Q from 12" discharge
5	2640	30	+1.15	31.15	5.05				water		pipe = 20 gpm
7	2760	30	+1.17	31.17	5.03						@ 3:45
9PM	2880	30	+1.16	31.16	5.04						

* barometer [illegible] [illegible] 4PM 11/15 [illegible]

100168

PUMPING TEST FORM

PROJECT J33102 WELL AWK-PW LOCATION ______ PAGE 1 OF __

SCREEN ______ M.P. ______ HT. ABOVE G.S. 2.9' W.L. MEAS. W/ M-scope

r ______ PUMPING WELL AWK-PW Q ______ ORIFICE ______ WEATHER cold - 30's (light rain, turned to light snow at 10:30 PM)

X DRAWDOWN __ RECOVERY __ LOCATION SKETCH

TEST START 11/11/84 9 PM

END ______

DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
9 PM	0	30	+1.16	31.16							
	½	35	+.92	35.92	4.76						
	1	35	+.15	35.15	[illegible]						
	1½	35	+.16	35.16	4.00						
	2	35	+.21	35.21	4.05						
	2½	35	+.26	35.26	[illegible]						
	3	35	+.29	35.29	4.13						
	3½	35	+.32	35.32	4.16						
	4	35	+.34	35.34	4.18						
	4½	35	+.36	35.36	4.20						
	5	35	+.39	[illegible]	4.23						
	6	35	+.41	35.41	4.25						
	7	35	+.42	[illegible]	4.27						
	8	35	+.44	[illegible]	4.28						
	[illegible]	35	+.46	[illegible]	4.30						
	10	35	+.48	35.48	4.32						
	12	35	+.51	35.51	4.35						
	14	35	+.52	35.52	4.36						
	16	35	+.54	35.54	4.38						
	18	35	+.56	35.56	4.40						
	20	35	+.57	35.57	4.41						
	22	35	+.64	35.64	4.48						
	24	35	+.65	35.65	[illegible]						
	26	35	[illegible]	[illegible]	4.51						
	28	35	+.70	[illegible]	4.54						
	30	35	+.70	35.70	4.54						
	35	35	+.71	35.71	4.55						
	40	35	+.73	35.73	4.57						
	45	35	+.73	35.73	4.57						
	50	35	+.74	35.74	4.58						
	55	35	+.75	35.75	4.59						
10 PM	60	35	+.76	35.76	4.60						
	70	35	+.79	35.79	4.63						
	80	35	+.81	35.81	4.65						
	90	35	+.83	35.83	4.67						
	100	35	+.84	35.84	4.68						
	110	35	+.85	35.85	4.69						
11	120	35	+.85	35.85	4.69						

100169

PUMPING TEST FORM

PROJECT TB31923 WELL AWX-PW LOCATION _____ PAGE 2 OF _____

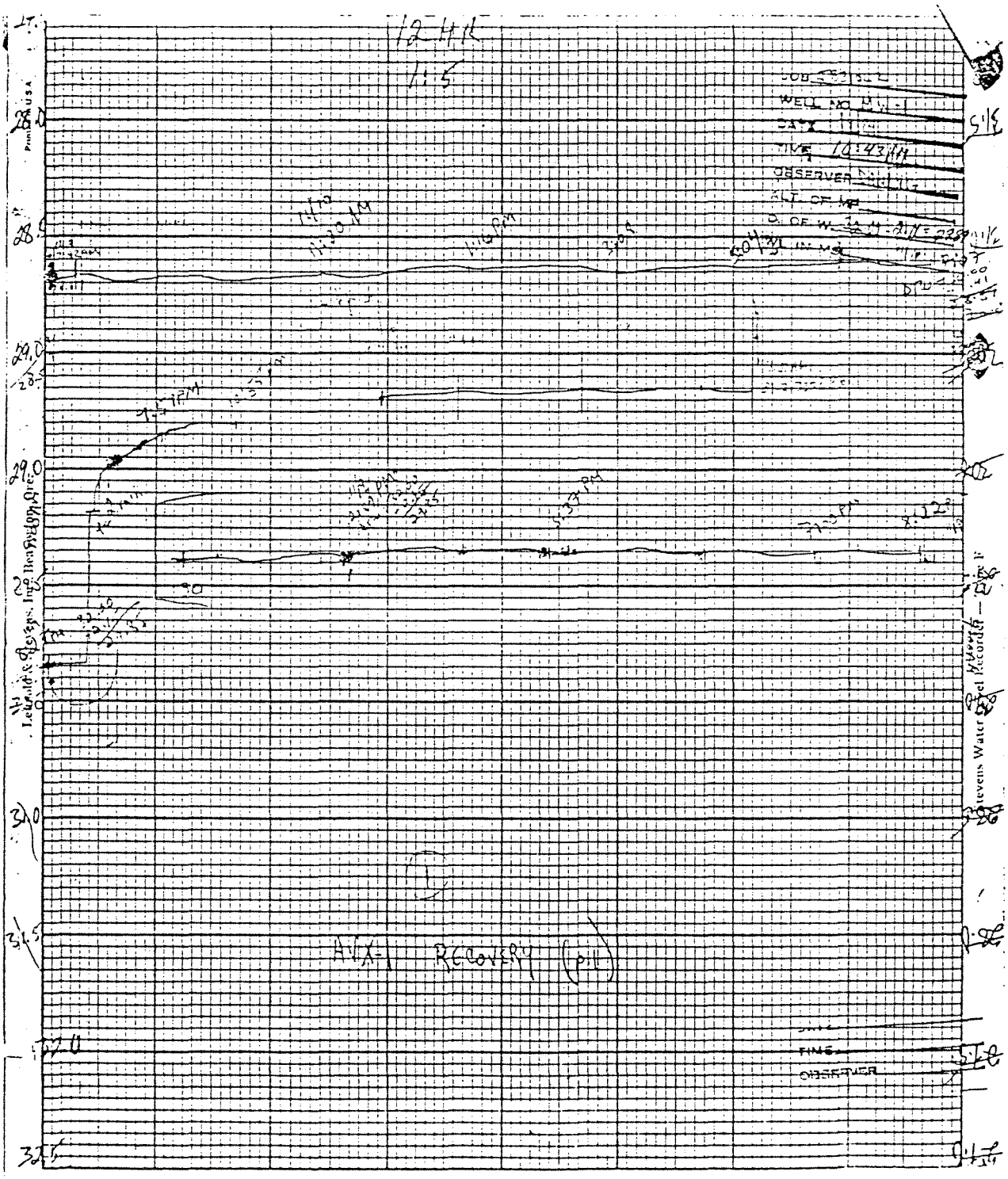
SCREEN _____ M.P. Rim of pump cover HT. ABOVE G.S. _____ W.L. MEAS. W/ _____

r _____ PUMPING WELL AWX-PW Q _____ ORIFICE _____ WEATHER _____

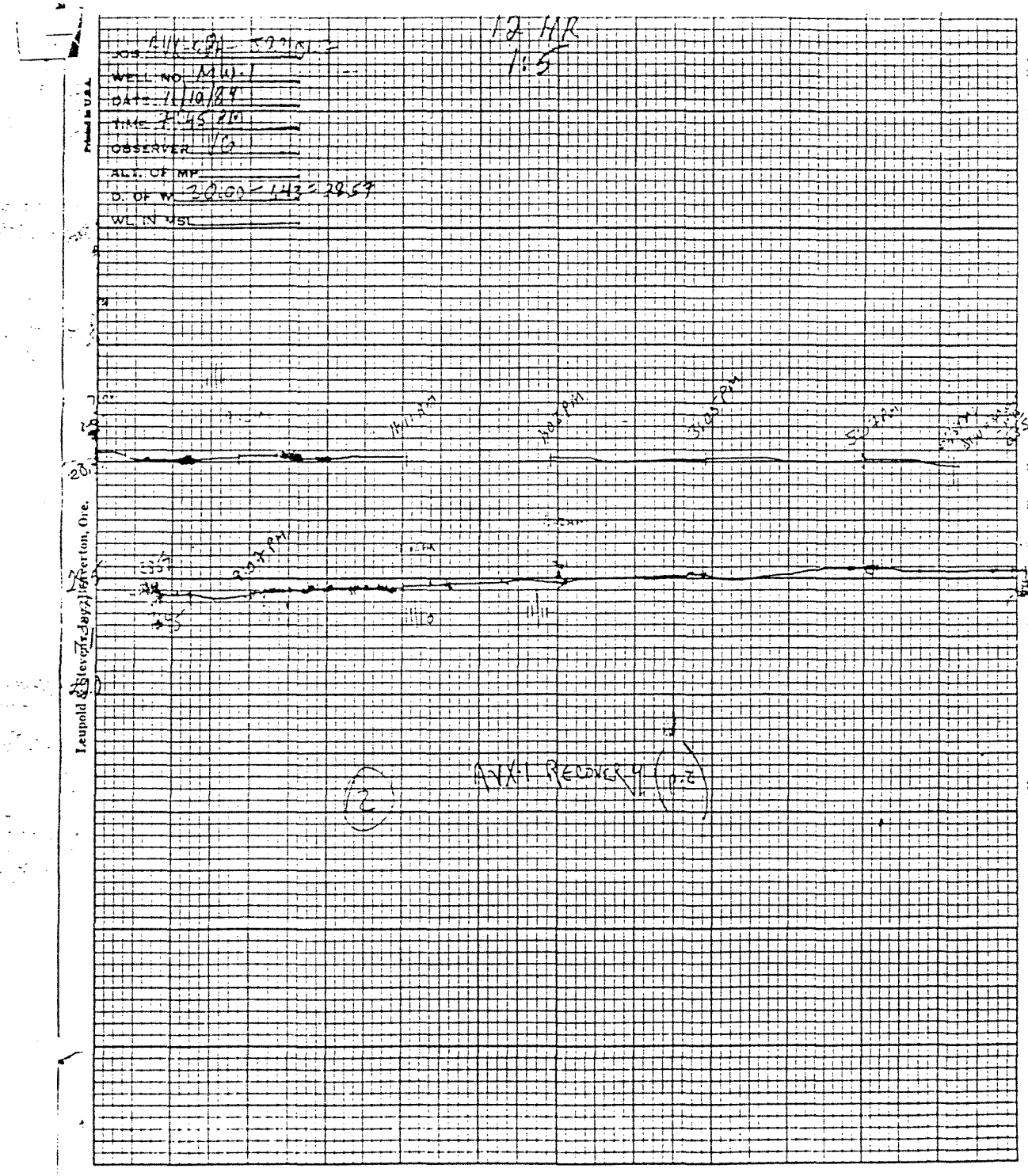
X DRAWDOWN ___ RECOVERY ___ LOCATION SKETCH TEST START _____ END _____

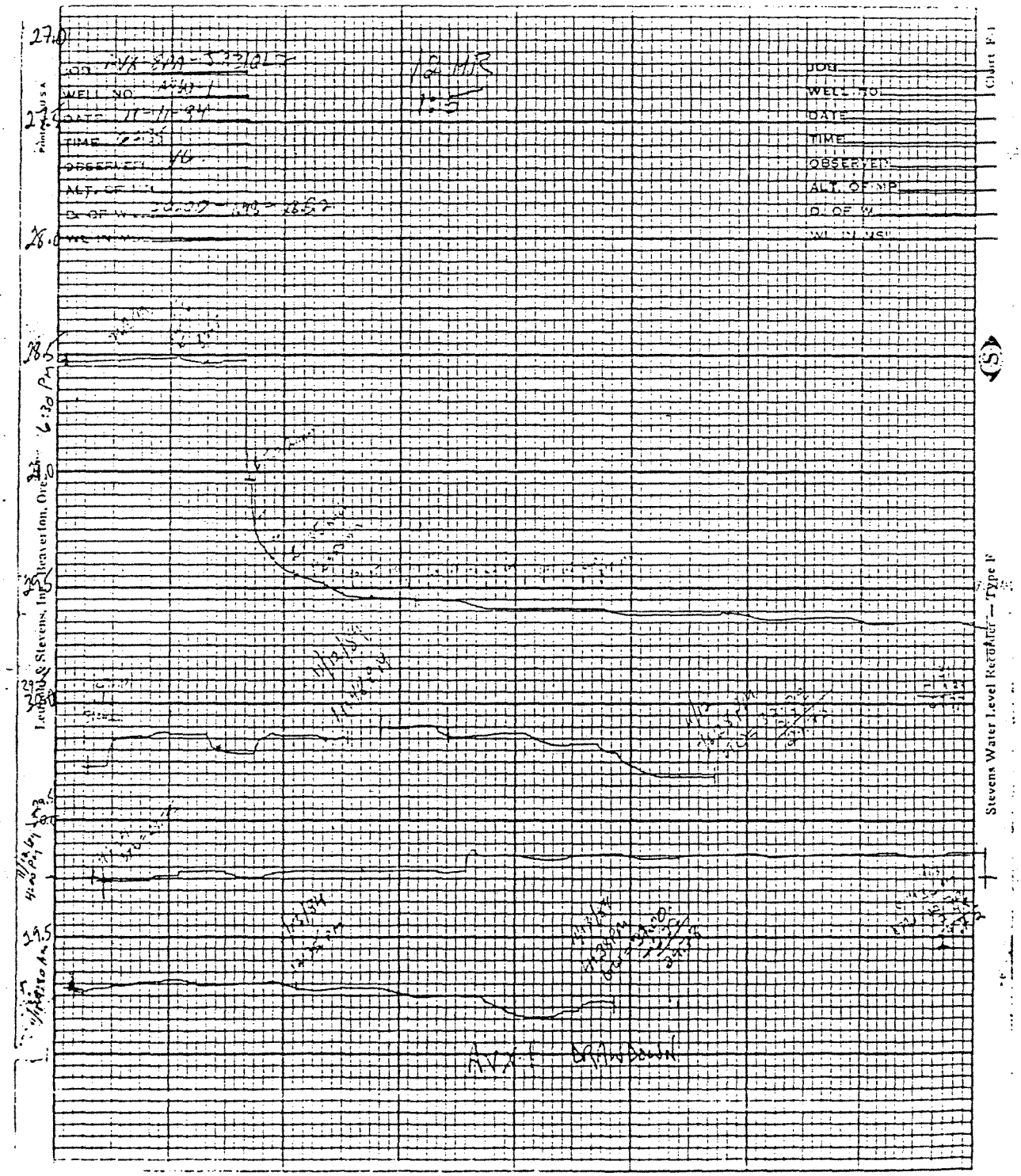
DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
	140	35	+.85	35.85	4.69						
	100	35	+.84	35.84	4.68						
12M	180										
8:33AM	668	35	+1.15	36.15	4.99						
									9AM		
11:43AM		35	+0.25	35.25			?				
										7 gallons in 1.1 minutes =	
										380 gpm	

AVX-1

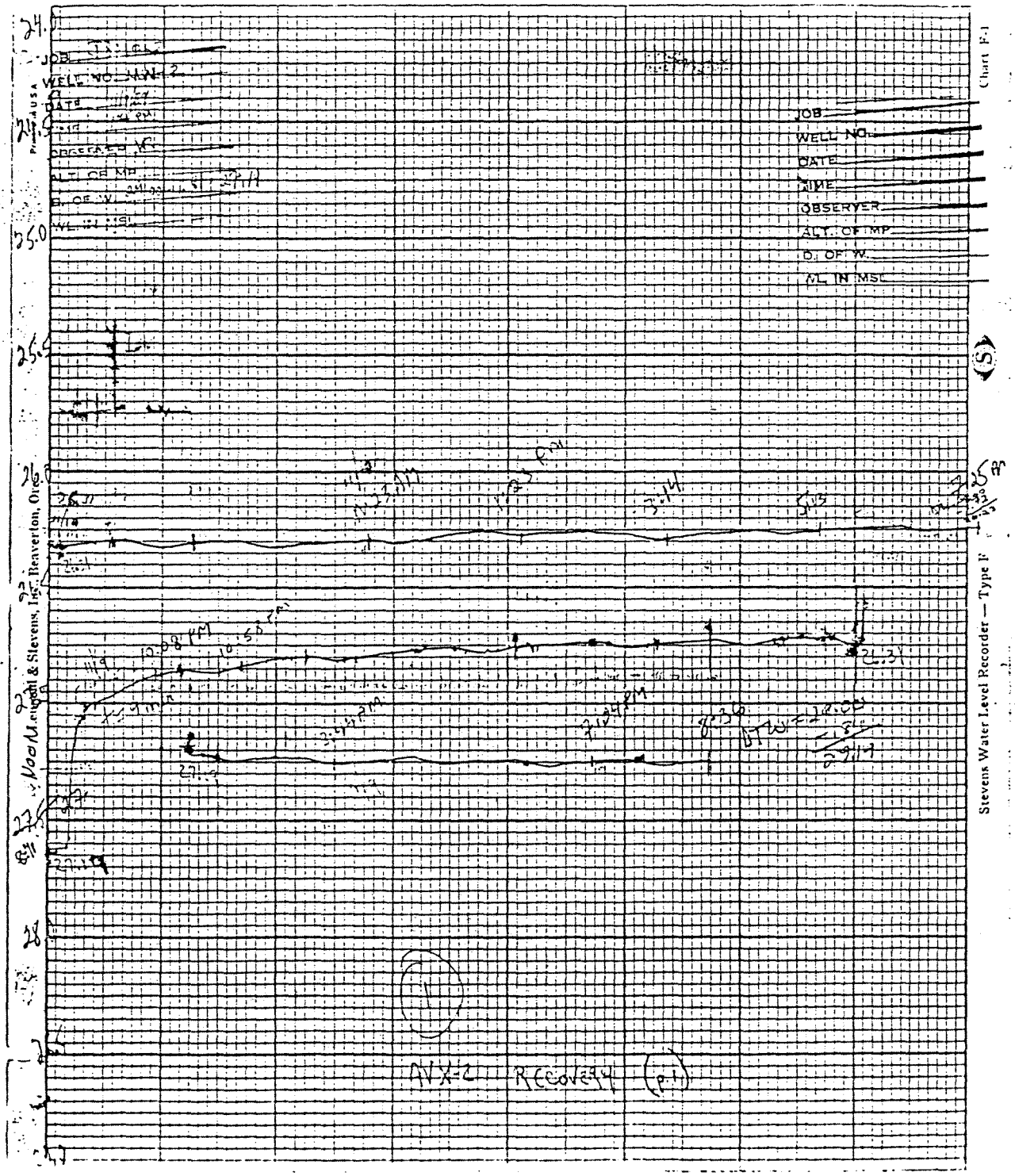

12 HR
1:5
WELL NO. MW-1
DATE
TIME 10:43 AM
OBSERVER
ELT. OF MP
D. OF W.
AIR RECOVERY (pit)
Leeds & Northrup Co. Philadelphia, Pa. U.S.A.
Stevens Water Level Recorder

100172

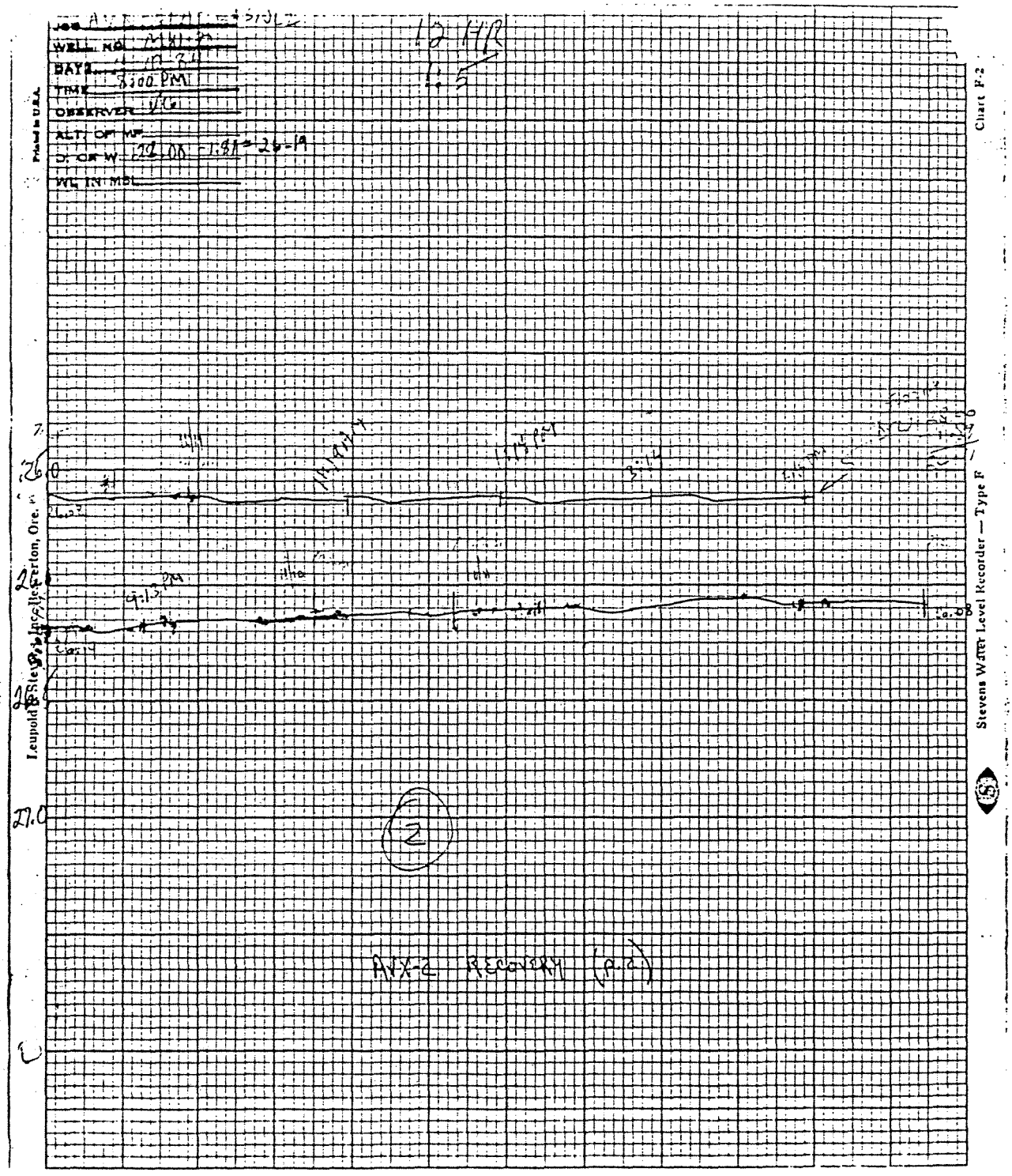

12 HR
1:5
JOB AVX-EPA-5775
WELL NO MW1-1
DATE 11/10/84
TIME 7:45 AM
OBSERVER VG
ALT. OF MP
D. OF W 30.00 - 1.43 = 28.57
WL IN USL
AVX-1 RECOVERY #1 (P-2)
Leupold & Stevens, Inc. Beaverton, Ore.
Stevens Water Level Recorder — Type F
Chart F-2


27.0
28.0
28.5
6:30 PM
Leupold & Stevens, Inc. Beaverton, Oregon
Stevens Water Level Recorder — Type F
Chart F-1
JOB
WELL NO.
DATE
TIME
OBSERVER
ALT. OF MP
D. OF W.
WL IN USE
12 HR
1:5

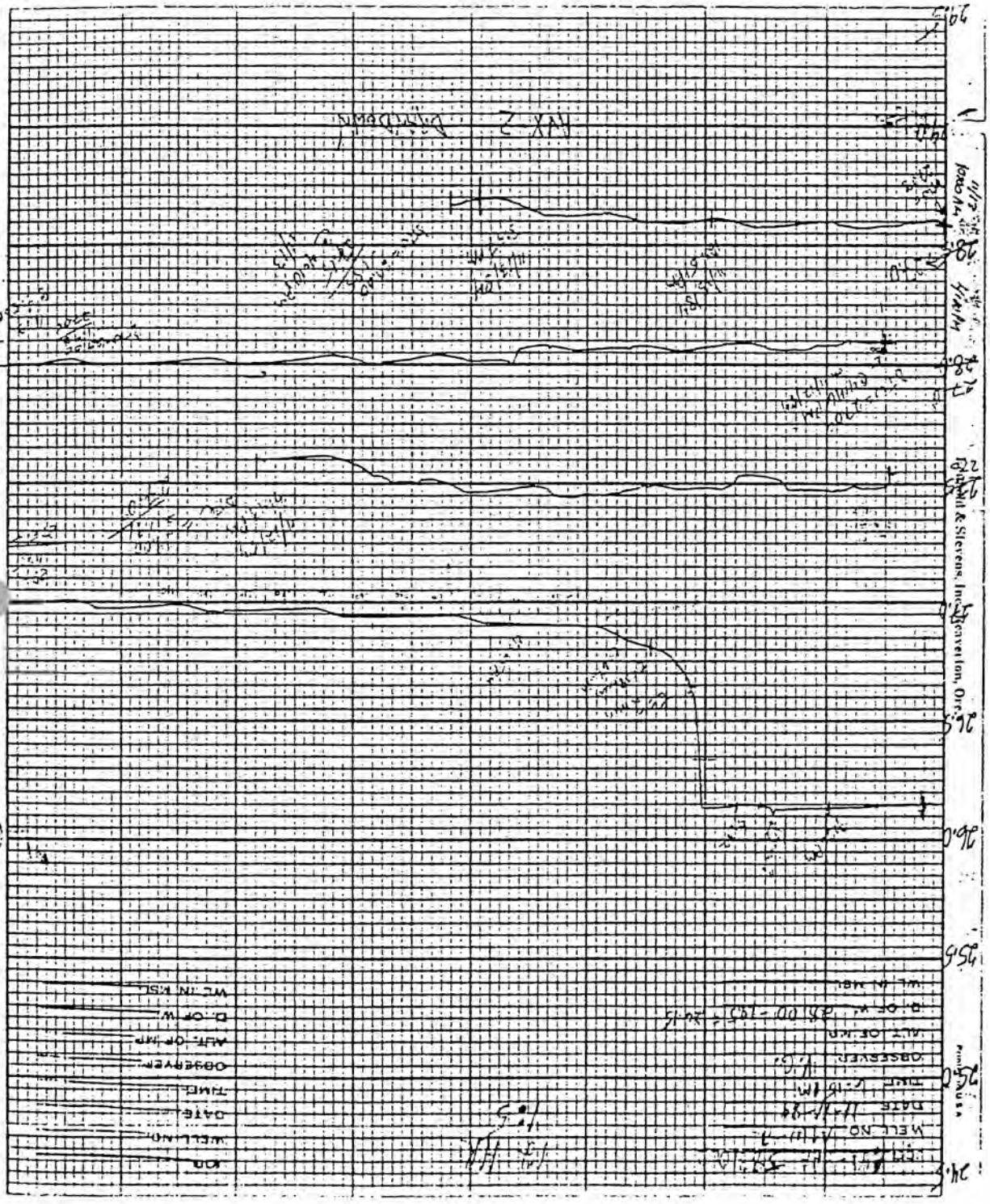
AVX-2


JOB
WELL NO.
DATE
TIME
OBSERVER
ALT. OF MP
D. OF W.
AL. IN MSL
Chart P-1
Stevens Water Level Recorder — Type F
Leupold & Stevens, Inc., Beaverton, Ore.
AVX-2 RECOVERY (p-1)

100176


WELL NO. MW-2
DAY 2
TIME 3:00 PM
OBSERVER VC
ALT. OF MP
D. OF W 12.08 - 1.81 = 26.19
WL IN MSL
12 HR
1.5
Leupold & Stevens Inc., Portland, Ore. U.S.A.
Stevens Water Level Recorder — Type F
Chart F-2
26.0
27.0
MW-2 RECOVERY (P.2)

100177


Stevens Water Level Recorder
Leupold & Stevens, Inc. Beaverton, Oregon
Chart E-1

AVX-3

100179

PUMPING TEST FORM

PROJECT JS31012 WELL MW-3 LOCATION _____ PAGE 1 OF _____

SCREEN _____ M.P. Top of 4" std HT. ABOVE G.S. 2.9' W.L. MEAS. W/ steel tape

r _____ PUMPING WELL AVX-PW Q _____ ORIFICE _____ WEATHER _____

___ DRAWDOWN X RECOVERY ___ LOCATION SKETCH TEST START 11/9/84 9PM

END _____

DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
11/9 8:48	-42	7.00	1.63	5.37							
	6:00	7.00	1.62	5.38							
	21:00	8.00	2.62	5.38							
	35	7.00	1.61	5.39							
	49	7.00	1.61	5.39							
	65	7.00	1.63	5.37							
10:54	114	8.00	2.62	5.38							
11:0?	165	7	1.64	5.36							
11/9 1:0?A	246	7	1.28	5.12							
11:?	3?6	—	1.9?	5.0?							
14?	3??	7	2.3?	4.66							
?:?	33?	7	3.0?	4.0?							
2:41	4??	5	1.2?	3.72							
2:??	5??	5	1.?6	3.??							
7:25	?:?	5	1.??	3.47							
9:?7	?:7	5	1.60	3.4?							← 0.5' below ground surface
11:19	859	5	1.58	3.42							
1:19	979	5	1.52	3.48							
3:12	1092	5	1.48	3.52							
5:08	1207	5	1.42	3.58							
7:17	1327	5	1.37	3.63							
9:10	1440	5	1.34	3.66							
11:09	1560	5	1.31	3.69							
1:??	1?2?	5	1.28	3.72							
11/11 3:??	12:?	5	1.24	3.76	←						
7:??	?:?	5	1.49	3.51							recovery slowing down
9:??	?:??	5	1.52	3.48							
11:17	2297	5	1.50	3.50							
1:19	2416	5	1.48	3.52							
3:12		5	1.40	3.60							
5:??		5	1.?7	3.?3							
7:??		5	1.52	3.48							

MW-3

100180

PUMPING TEST FORM

PROJECT 182022 WELL MW-3 LOCATION _____ PAGE 1 OF _____

SCREEN _____ M.P. _____ HT. ABOVE G.S. _____ W.L. MEAS. W/ _____

r _____ PUMPING WELL AVX-PW Q _____ ORIFICE _____ WEATHER _____

X DRAWDOWN ___ RECOVERY ___ LOCATION SKETCH TEST START 11/11/84 9PM

END _____

DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
11/11 8:31 PM	=29	5.00	1.31	3.69				STATIC			
9:34	34	5.00	1.31	3.69							
9:57	57	5.00	1.30	3.70							
10:24	1:4	5.00	1.31	3.69							
11:08	1:8	5.00	1.30	3.70							
11/12 8:27		5	1.26	3.74							

MW-3

OFF-SITE WELLS

100182

PUMPING TEST FORM

PROJECT J831042 WELL CDM-14 LOCATION _____ PAGE 1 OF _____

SCREEN _____ M.P. top 1½" metal well cap HT. ABOVE G.S. _____ W.L. MEAS. W/ steel tape

r _____ PUMPING WELL AVX-PN Q _____ ORIFICE _____ WEATHER _____

___ DRAWDOWN X RECOVERY ___ LOCATION SKETCH TEST START 11/9/84 9PM

END _____

	DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
11/9	20[5] PM		42	0.89	41.11							
AVX-PN	start 4 9PM											
			4									
	11:11[A]	131	113.00	2.39	40.61	-	.50					
11:10	20[4]A	3e4	42.	2.50	40.50	-	.61					
	10:34	8:14	44.00	3.60	40.40	-	-					
	3:39[P]	1119	43.00	2.63	40.37	-	.74					
11/11	3:34[P]		43.50	2.75	40.25		.86	←	AVX PN Transducer			11/11 9PM
	11:23		43.00	2.21	40.79							
			~~42~~	~~1.22~~	~~40.01~~							
11/12	10[2] AM		43	1.99	41.01		.76					

CDM-14

PUMPING TEST FORM

PROJECT J831062 WELL DM-19A LOCATION ______ PAGE ___ OF ___

SCREEN ______ M.P. top of 2" steel inner casing! HT. ABOVE G.S. ______ W.L. MEAS. W/ ______

r ______ PUMPING WELL AVX-PW Q ______ ORIFICE ______ WEATHER ______

___ DRAWDOWN X RECOVERY ___ LOCATION SKETCH TEST START 11/6/84 9 PM END ______

DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
11/9 2:06 PM		47	1.84	45.16							
AVX-PW off 11/9 PM											
11:18	138	47.00	2.06	44.94		.22					
11/10 [illegible]	319	47	2.20	44.80		.36					
10:40	820	47.00	2.28	44.72		.44					
3:43	1123	47.00	2.33	44.67		.49					
11/11 3:38 P		46.00	1.42	44.58		0.58		←	AVX-PW	turned	on 9PM
10:45		46.00	1.17	44.83							
11/12 10:34		47	1.88	45.12		0.54					

DSM 19A

100184

PUMPING TEST FORM

PROJECT J831062 WELL DH-19 LOCATION _____ PAGE 1 OF _____

SCREEN _____ M.P. top of 2" Dia inner casing HT. ABOVE G.S. _____ W.L. MEAS. W/ steel tape

r _____ PUMPING WELL AUX-PW Q _____ ORIFICE _____ WEATHER _____

___DRAWDOWN X RECOVERY ___LOCATION SKETCH TEST START 11/9/84 9PM

END _____

	DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
11/9	2" PM		48	2.86	45.14							
	AUX-PW on:	11 - 9PM										
	11:24	141	48.00	3.24			.38					
11/10	2:15 PM	315	47	2.34	44.66		.48					
	10:45	823	47.00	2.42	44.58		.56					
	3:48	1128	47.00	2.45	44.55		.59					
11/11	3:40		46.00	1.57	44.43		.71		← AUX PW turned on 9PM			
	10:47		46.00	1.18	44.82							
12	10:32		47	1.92	45.08		0.65					

D + M 19

100185

PUMPING TEST FORM

PROJECT J8310-2 WELL D&M-9 (east) LOCATION _____ PAGE 1 OF _____

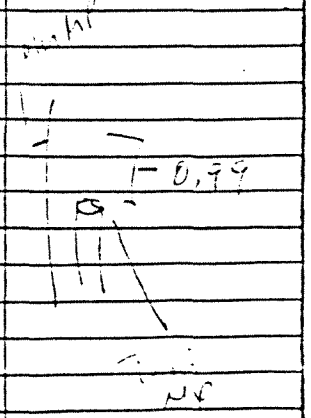
SCREEN _____ M.P. Top of 12" Csg HT. ABOVE G.S. _____ W.L. MEAS. W/ Tape

r _____ PUMPING WELL RW-PW Q _____ ORIFICE _____ WEATHER _____

___DRAWDOWN X RECOVERY ___LOCATION SKETCH TEST START 11/9/84 9PM

END _____

	DATE TIME	t	HELD	WET	D.T.W.	s			MANO— METER	Q	WATER TEMP.	
11/9	3:24 PM		19	1.98	17.02							
RW	PW shut off 9 PM											
	11:38	158	19.00	2.33	16.67		.35					
11/10	3:06	370	19	2.40	16.60		.42					
	11:49	889	19	2.41	16.59		.43					
	3:57	1137	18.00	1.47	16.53		.49					
11/11	3:51		18.00	1.58	16.42		0.60			← RW	Pulled on 9 PM	
	[illegible]		18.00	1.26	16.74							
11/12	10:41		19	2.12	16.88		0.46					



D+M-9 (EAST)

PUMPING TEST FORM

PROJECT J831022 WELL D&M 9A (west) LOCATION ______ PAGE ___ OF ___

SCREEN ______ M.P. top of 2" steel inner [illegible] HT. ABOVE G.S. ______ W.L. MEAS. W/ ______

r ______ PUMPING WELL ______ Q ______ ORIFICE ______ WEATHER ______

___ DRAWDOWN X RECOVERY ___ LOCATION SKETCH TEST START ______ END ______

DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
11/9/84 3²⁷ PM		20	2.06	17.94							
AUX-PW on till 9PM											
11:35	155	20.00	2.42	17.58		.36					
11/10 3¹⁵ AM	373	20	2.49	17.52		.42					
11:11	886	20	2.50	17.50		.44		AUX PW turned on 9PM			
3:55	1135	19.00	1.55	17.45		.49					
11/11 3:48 P		19.00	1.67	17.33		0.61					
10:57		19.00	1.35	17.65							
11/12 10⁴³		20	2.21	17.79		.46					

[illegible]

0.30 [illegible] 0.30

D&M 9A (WEST)

100187

PUMPING TEST FORM

PROJECT J831022 WELL DEM-10 LOCATION _____ PAGE 1 OF _____

SCREEN _____ M.P. top of 12" steel casing HT. ABOVE G.S. _____ W.L. MEAS. W/ Steel Tape

r _____ PUMPING WELL ANX-PW Q _____ ORIFICE _____ WEATHER _____

TEST START 11/9/84 9 PM

___ DRAWDOWN X RECOVERY ___ LOCATION SKETCH END _____

	DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
11/9	3:33 PM		28	2.27	25.73							
	ANX-PW shut-off	9 PM										
	11:55	176	27.00	1.60	25.40		33					
11/10	3:55 A	552	27	1.69	25.31		.42					
	11:57 A	897	27	1.71	25.29		44					
	4:07 P	1147	27.00	1.75	25.25		.48					
11/11	4:00 A		2700	1.87	25.13		0.60		← ANX PW Turned on 9PM			
	4:03 P		2730	1.85	25.15							
11/12	10:55 A		27	1.44	25.56		0.43					

DxM 10

100188

PUMPING TEST FORM

PROJECT T831022 WELL DTM 10A LOCATION _____ PAGE 1 OF _____

top of 12" Steel.

SCREEN _____ M.P. Inner casing HT. ABOVE G.S. _____ W.L. MEAS. W/ Steel Tape

r _____ PUMPING WELL AVX-PW Q _____ ORIFICE _____ WEATHER _____

TEST START 11/9/84 9PM

___ DRAWDOWN X RECOVERY ___ LOCATION SKETCH END _____

	DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
11/9	3:30PM		28	2.11	25.89							
AVX	PW shut	21	9PM									
		"										
	11:45	135	27.00	1.45	25.55		.34					
11/10	3:20PM	280	27	1.52	25.48		.41					
	11:55A	895	28	2.54	25.46		.43					
	4:04P	1144	27.00	1.60	25.40		.49					
11/11	3:58P		28.00	2.71	25.29		.60		← AVX	PW Turned on	9PM	
	11:06		27.00	1.39	25.61							
11/12	1:25PM		28	2.75	25.25		.36					

DTM-15A

100189

PUMPING TEST FORM

PROJECT 83102 WELL D4M-10B LOCATION _____ PAGE ____ OF ____

top of 2" steel

SCREEN _____ M.P. measuring HT. ABOVE G.S. _____ W.L. MEAS. W/ steel tape

r _____ PUMPING WELL AUX-PW Q _____ ORIFICE _____ WEATHER _____

TEST START 11/9/84 9PM

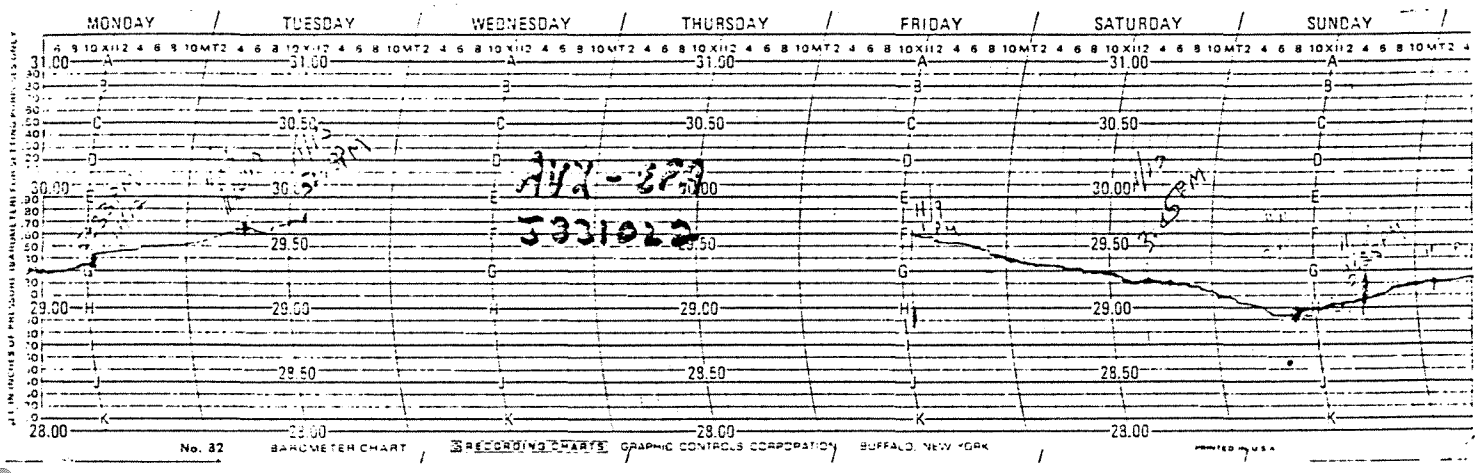
___ DRAWDOWN X RECOVERY ___ LOCATION SKETCH END _____

	DATE TIME	t	HELD	WET	D.T.W.	s			MANO-METER	Q	WATER TEMP.	
11/9	3:36 PM		9	1.62	7.38							
AUX	PW on	11 9 PM										
		9										
	11:53	173	9.00	1.63	7.37		.01					
11/10	3:26	956	9	1.64	7.36		.02					
	12:00 M	900	9	1.62	7.38		—					
	4:09	1149	9.00	1.64	7.36		.02					
11/11	4:03		9.00	1.63	7.37				← AUX PW		turned	on 9 PM
	11:11		9.00	1.63	7.37							
11/12	10:07		10	1.57	7.43							

D4M-10B

RECORDING BAROMETER

100191



RECORDING BAROMETER CHART

100192

U.S. ENVIRONMENTAL PROTECTION AGENCY

POLLUTION REPORT

DATE: April 12, 1985

Region II
Response and Prevention Branch
Edison, NJ 08837

(201) 321-6670 - Commercial
(201) 548-8730 - 24 Hr. Emergency
340-6670 - FTS

TO: C. Daggett, EPA
W. Librizzi, EPA
B. Ogg, EPA
F. Rubel, EPA
J. Marshall, EPA
A. Jenick, EPA
W. Mugdan, EPA
ERD, EPA Washington, D.C.
(Data Gram)
N. Nosenchuck, NYSDEC
C. Halgas, Cattaraugus County
Health Dept.
P. Keller, NYSDEC
J. Anderson, HHS
D. Dana, NYSDEC

POLREP NO.: Seven (7)
INCIDENT NAME: Olean
SITE/SPILL NO.: 16
POLLUTANT: Trichloroethylene (TCE)
CLASSIFICATION: Medium
SOURCE: Unknown
LOCATION: Olean, New York
AMOUNT: Unknown
WATER BODY: Groundwater

1. SITUATION:

A. Same as previous POLREP.

2. ACTION TAKEN:

A. Installation of two water supply wells point of use treatment systems were completed on April 1, 1985 by the ERCS subcontractor. Additional work activities on these two installations took place on April 10th, 1985.

B. On April 9th, water samples to monitor filter effectiveness were taken from carbon units newly installed in two residences.

C. Water samples to check bacterial growth in the two installed carbon units were also collected.

D. A total eight (8) samples for volatile organic compounds were collected. The samples included 1 duplicate and 1 field blank.

E. A total of 2 samples for total organic carbon were collected.

3. STATUS OF FUNDS:

A.	Total Extramural Trust Funds Authorized for Mitigation Contracts	$ 144,000.00
1.a.	Cattaraugus County Health Department	25,000.00
1.b.	ERCS to Date	119,000.00
B.	Expenditures for Mitigation Contracts	
1.a.	Amount Obligated to Cattaraugus County DOH Contract #68-62-0011 Initiated 2/4/82	25,000.00
1.b.	Estimated Expenditures for #68-62-0011	23,174.00
1.c.	Balance Remaining for #68-62-0011	1,826.00
2.a.	Total ERCS Funding Obligated under D2D035, #KCS 405 #KCS 415 and #KCS 438	113,000.00
2.b.	Estimated total expenditures for ERCS contractor under D2D035, #KCS 405 and #KCS 415	78,750.00
2.c.	Total balance remaining for D2D035, #KCS 405 and #KCS 415	8,250.00
3.a.	Amount Obligated to ERCS Contractor, O.H. Materials Order of Services #6893-02-020, DCN #KCS 438	26,000.00
3.b.	Estimated Expenditures for DCN #KCS 438 as of April 12, 1985	11,000.00
3.c.	Balance Remaining For DCN #KCS 438	15,000.00

C. Unobligated Balance Remaining As of April 12, 1985	6,000,00
D. Estimate of Total Expenditures As of April 12, 1985 for All Mitigation Contracts	112,924.00
E. Other Extramural Costs	
1.a. Estimated TAT Salary/Travel Thru April 12, 1984	21,762.00
F. Estimated Intramural Costs Reported by Computer Accounting Thru March 1, 1985	11,303.70
G. TOTAL Estimated Expenditures Thru April 12, 1985 and Percentage of $1,000,000.00	$ 145,989.70 (14.6% 1M)

4. FUTURE PLANS AND RECOMMENDATIONS:

A. Another round of sampling to monitor carbon filter effectiveness will occur 90 days after completion of the installations.

B. The situation will continue to be followed for future actions.

CASE PENDS X CASE CLOSED _____ SUBMITTED BY John Witkowski

(TAT)

John Witkowski,
OSC
Response and Prevention Branch

DATE RELEASED 4/15/85

100195

The Actual Document is Available
for Review in the

Olean Well Field - Start Three

Site File

Site Files are located at the
U.S. Environmental Protection Agency
Region II
Superfund Removal Records Center
Edison, NJ

TAT-02-F-01418

COMMUNITY RELATIONS PLAN

OLEAN WELLFIELDS

OLEAN, NEW YORK

Prepared By:
Anne Tischbein
Weston/SPER Division
Edison, New Jersey 08837

Prepared For:
Robert M. Cobiella, OSC
Emergency and Remedial Response Division
Response and Prevention Branch, U.S. EPA
Site Mitigation Section
Edison, New Jersey 08837

COMMUNITY RELATIONS PLAN
OLEAN WELLFIELDS
OLEAN, NEW YORK

I. PURPOSE:

A request for an EPA removal action in the Town of Olean, New York has been received from the Cattaraugus County Health Department (CCHD). The request was made verbally by Chester R. Halgas, Director of Environmental Health, CCHD, and will be followed up in writing for EPA assistance to mitigate the impact of chemically contaminated groundwater to individual drinking water supply wells in the Town of Olean (see Figures). At this time, the wells in question supply 2 residences. These users depend on the wells as their sole source of potable water and this groundwater contamination poses an immediate and significant risk to human life and health.

The County Department of Health has recommended that the homes in question receive treatment units to protect public health. These 2 wells show contamination by volatile organic compounds (VOC) to levels exceeding New York State Department of Health (NYSDOH) guidelines for potable water which limit concentrations of total volatile organic compounds to less than 100 ppb and a single volatile organic compound to less than 50 ppb. the contaminant is trichloroethylene for which the EPA Suggested no adverse response Level (SNARL) is 200 ppb. This memo discusses a plan for community relations prior to, and during, the immediate removal action.

II. BACKGROUND:

A. Site Setting/Description:

The contaminated wells are located in the Town of Olean, Cattaraugus County, New York. All are located along East State Road to the west of Dugan Road. This area is generally a mixed residential/light industrial area and borders the City of Olean to the west. EPA has conducted 2 previous immediate removal actions in the Town of Olean. In 1982, 16 carbon units were installed to reduce well water contamination. In 1984, 10 double carbon systems were installed. The wells in question for this immediate removal action were not previously addressed during 1982 or 1984 actions.

B. Quantity and Types of Substances Present:

Five hazardous volatile organics have been documented in the well water of the two affected residences to date. These are:

Chloroform
Methylene Chloride
Tetrachloroethylene
1,1,1-Trichloroethane
Trichloroethylene

The cumulative concentration of volatile organics in these 2 wells range from 190 to 200 ppb, with the primary contaminant being trichloroethylene. All of these substances are designated hazardous under CERCLA.

C. This site is on the National Priorities List (NPL).

III. THREAT:

A. Threat of Public Exposure:

This is a case of actual contamination at the tap for 2 families. Contaminants have been documented in these wells in excess of the NYSDOH guidelines (50 ppb for a single volatile organic compound or 100 ppb for any combination of volatile organic compounds) and the New York State Groundwater Standard for Trichloroethene (10 ppb).

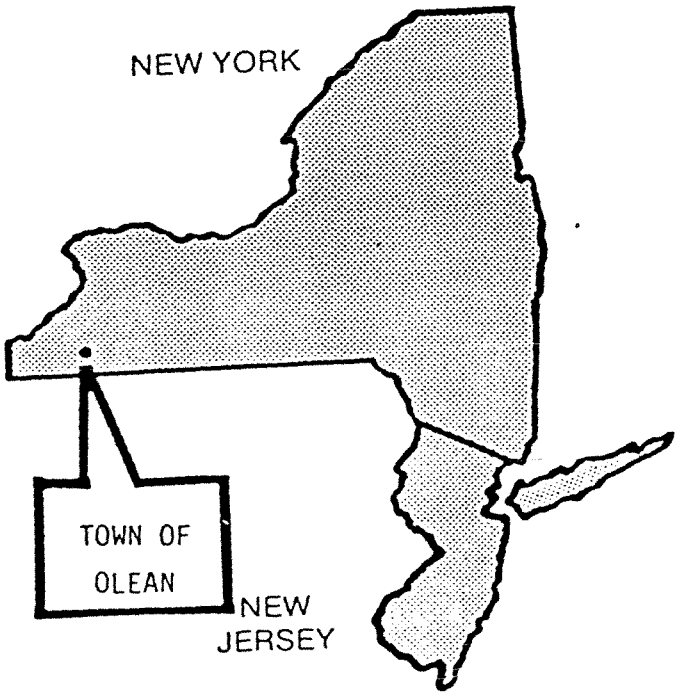
Individual private wells tapping this aquifer provide the potable water supply for the residences. In addition to the potential for exposure through drinking the water or eating food prepared with the water, when water is used for hot showering, the volatilization of the organics elevates the levels of these compounds in air.

B. Evidence of Extent of Releases:

Contamination of local drinking water wells with volatile organic compounds has been in evidence since 1981 when the contamination was discovered and private well owners in the town were notified to boil their water as a precaution. Since this time, EPA has conducted 2 immediate removal actions to mitigate contamination in 26 wells (16 in 1982 and 10 in 1984). In this present situation, sampling revealed concentrations of volatile organics in excess of the NYSDOH guidelines for volatile organics concentration in potable water in 2 individual wells. The samples showed contamination ranging from 190 ppb to 200 ppb.

C. Previous Actions to Abate Threat:

In 1981, 50 owners of private wells were advised to boil their water before use. In 1982, the U.S. EPA funded installation of 16 activated carbon filtration



AREA LOCATION MAP

Figure 1

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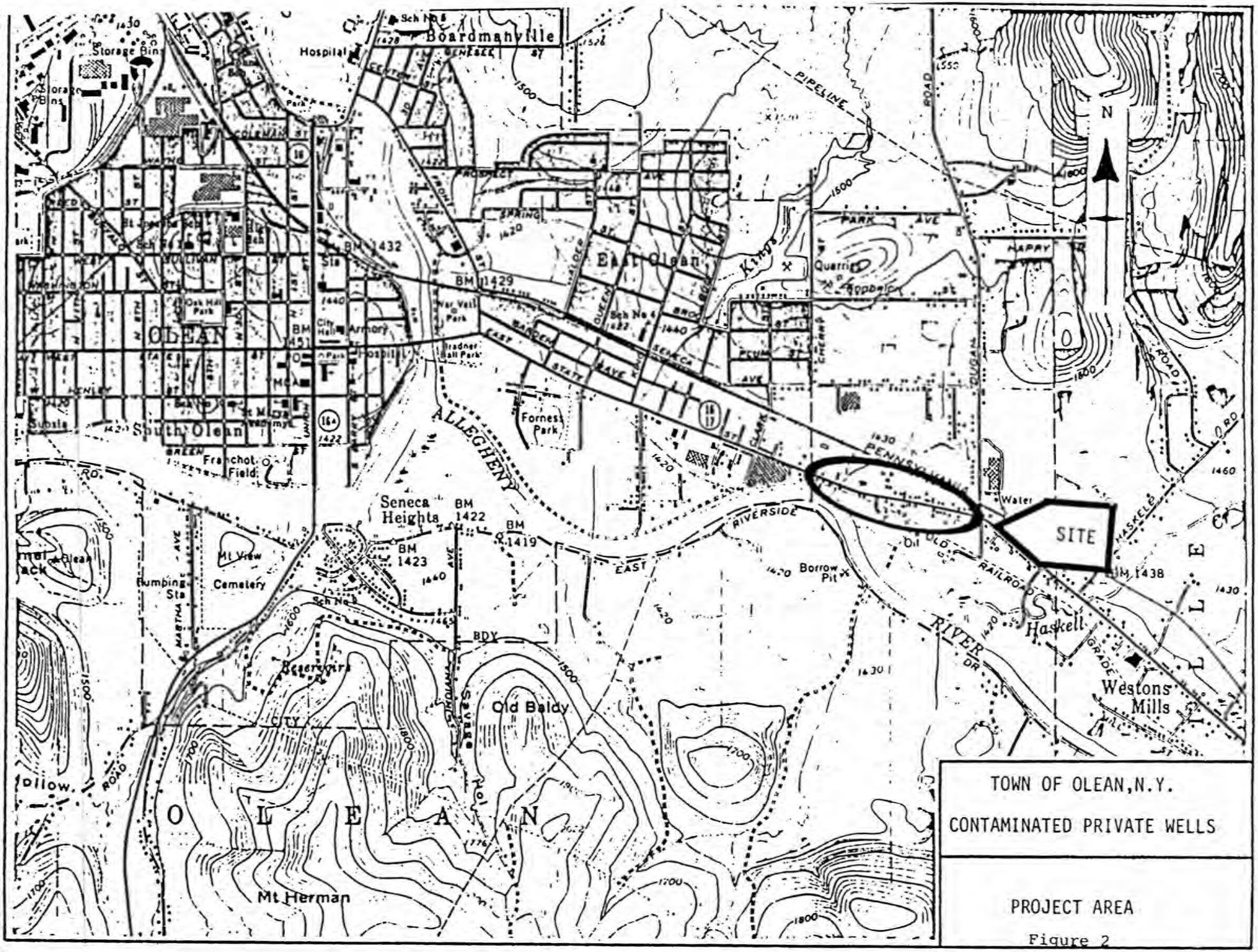
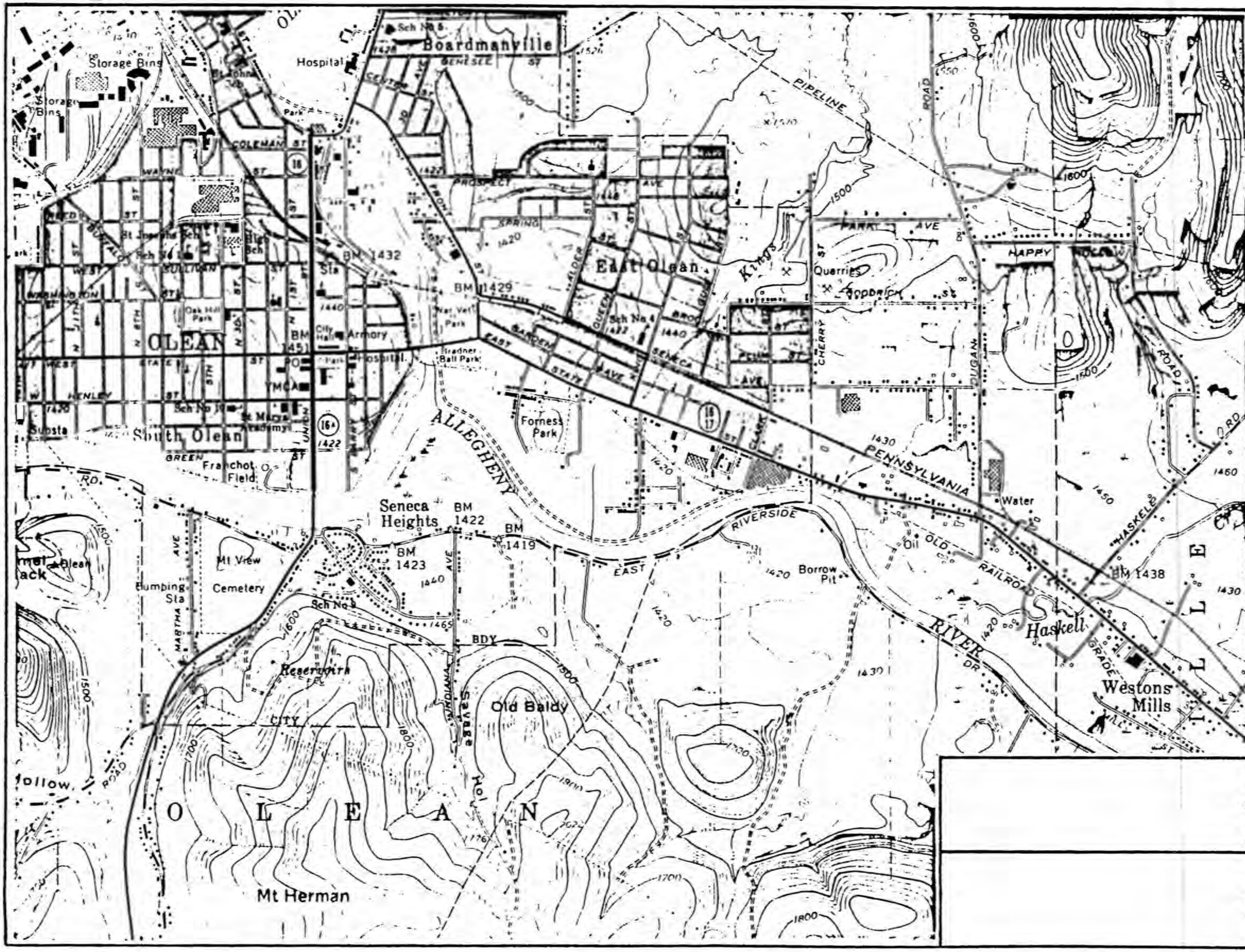


TOWN OF OLEAN, N.Y.

CONTAMINATED PRIVATE WELLS

PROJECT AREA

Figure 2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WAYNE ST
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HAPPY HOLLOW
St Josephs Sch
Sch No 1
High Sch
BUFFALO
WEST
SULLIVAN
WASHINGTON
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Sta
BM 1429
East Olean
Kings
Quarries
GOODRICH ST
Oak Hill Park
OLEAN
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City Hall
Armory
Vet Park
Park
Hospital
Bradner Ball Park
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SENECA
BROOK
GARDEN
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CHERRY
DUGAN
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YMCA
HENLEY
Sch No 3
St Marys Academy
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South Olean
UNION
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Forness Park
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CLARK
PENNSYLVANIA
HASKELL ROAD
Water
GREEN
Franchot Field
RD
Seneca Heights
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BM 1419
BM 1423
RIVERSIDE
OLD RAILROAD
Oil
BM 1438
Mt View Cemetery
Pumbing Sta
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AVE
EAST
Borrow Pit
Sch No 9
BDY
RIVER
DR
Haskell
GRADE
Westons Mills
Reservoirs
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units on individual wells to reduce the contamination. In general, these units appear to be functioning, however, 5 units were recently shown to have allowed breakthrough resulting in finished water concentrations exceeding the 50 ppb NYSDOH guideline. The carbon in these units was replaced by the county health department.

In 1984, the U.S. EPA funded installation of 10 additional activated carbon filtration units on individual wells to reduce contamination.

D. Current Action to Abate Threat:

Such current actions may include the boiling of water used for eating and drinking, limiting the use of water for showering and possibly, the use of bottled water.

IV. PROPOSED PROJECT:

A. Objectives of the Project:

The proposed project calls for the installation of water filtration units designed to remove volatile organic compounds on individual wells. These units are similar to the previous activated carbon units installed in area homes during the 1984 immediate removal action where similar contamination was noted. The units proposed under this project should be effective in removing the volatile organic contaminants in that they include the use of 3 cubic feet of activated carbon in each of 2 tanks. These units are expected to be more effective than the units installed in 1982 in that more carbon is being used, and that the filters will be so connected as to allow the use of filtered water for backwash.

This project includes purchasing, installation and maintenance of the units, and sampling and analysis of the water systems treated over a 6 month period.

B. Objectives of the Community Relations Plan:

The plan is designed to:

1. Provide accurate understandable information to local citizens, elected officials, and the media.

2. Integrate the local government, State and Federal response.

3. Assist public acceptance of the chosen response actions.

4. Enlist the assistance of local officials, as needed.

The officials and groups for whom this plan is designed are: local citizens, citizens groups, school principals, local businesses, elected officials, local, State, and Federal agencies working in association with Region II EPA.

The information will be supplied by EPA's Office of External Programs, New York City with the cognizance of the Office of the Regional Administrator.

C. Community Relations Activities:

Date(s)	Activities	Objective	Staff	Hours
Upon authorization of funding	1. Meeting with State and local officials	To discuss community relations needs	OSC OEP Rep TAT*	24 24 24
Upon authorization of funding as needed	2. Press release	To brief community and press. Provide community with information on progress of the removal action	OSC OEP Rep	24 24
	3. Fact sheet	To provide information for affected/interested public on activity at key decision points	OSC OEP Rep	24 24

*TAT stands for the U.S. EPA authorized contractor, Technical Assistance Team, Roy F. Weston, Inc.

Date(s)	Activities	Objective	Staff	Hours
	4. Briefings	To inform State and local officials about on-going developments at the site	OSC OEP Rep	24 24
	5. Public meetings	To discuss the need for response and review key decision points, explain the clean up method and respond to citizen concerns	OSC OEP Rep	24 24
When system operating	6. Site tours	Local elected officials, local and State government officials	OSC	24

D. List of Key Officials and Contacts:

Federal Agencies	Telephone
EPA Site Mitigation Section	
Robert Cobiella, OSC	(201) 321-6646
EPA Office of External Programs	
Jim Marshall	(212) 264-4913
Rich Cahill	(212) 264-8504
Herman Phillips	(212) 264-1044
Lillian Johnson	(212) 264-2515

Federal Officials	
Senator Alfonse M. D'Amato	(202) 224-6542 (518) 463-2244
Senator Daniel P. Moynihan	(202) 224-4451 (212) 661-5150

Federal Officials	Telephone
Representative Stanley Lundine	(716) 372-1818
New York State Agencies	
New York State Department of Environmental Conservation	
Hazardous Waste Program (Inland Albany Office)	(518) 457-9538
Hazardous Waste Program (Region 9)	(716) 847-4590
New York State Department of Health	(716) 847-4500
New York State Police	(716) 373-2550
New York State Officials	
Senator Jess Present	(716) 372-0345
Assemblyman Daniel Walsh	(716) 372-0345
Cattaraugus County Agencies	
Chester Halgas, P.E., Director Environmental Health Department	(716) 375-4121
Cattaraugus County Officials	
Chairman of County Legislature James Snyder	(716) 372-3511
City of Olean	
Mayor William Smith	(716) 372-2200
Director of Department of Public Works Robert Carr	(716) 372-2200
Fire Department	911
Police	911
Town of Olean	
Supervisor David Torrey	(716) 372-5578
Fire Department	(716) 372-2320

Media Contacts	Telephone
Olean Times Herald Rick Miller	(716) 372-3121
Buffalo Evening News Burton Freed	(716) 372-8375