

**WORKPLAN TO  
EVALUATE THE EXTENT OF CHLORINATED VOCs  
IN GROUNDWATER  
AT THE  
TECUMSEH PRODUCTS COMPANY  
GRAFTON, WISCONSIN, FACILITY**

**PREPARED FOR  
TECUMSEH PRODUCTS COMPANY  
NEW HOLSTEIN, WISCONSIN**

**AUGUST 1994**

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AUGUST 1994

*Lisa Drzewiecki*

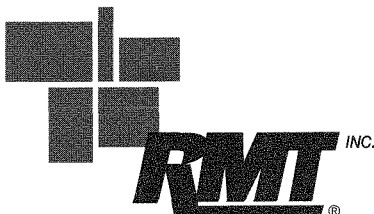
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Photovac® Technical Bulletin #27

## Section 1

### INTRODUCTION

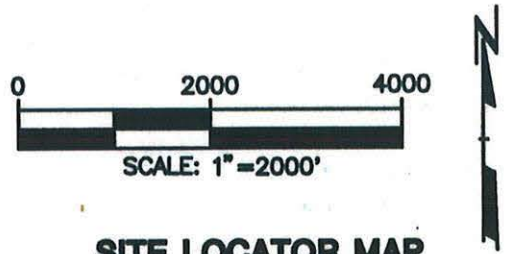
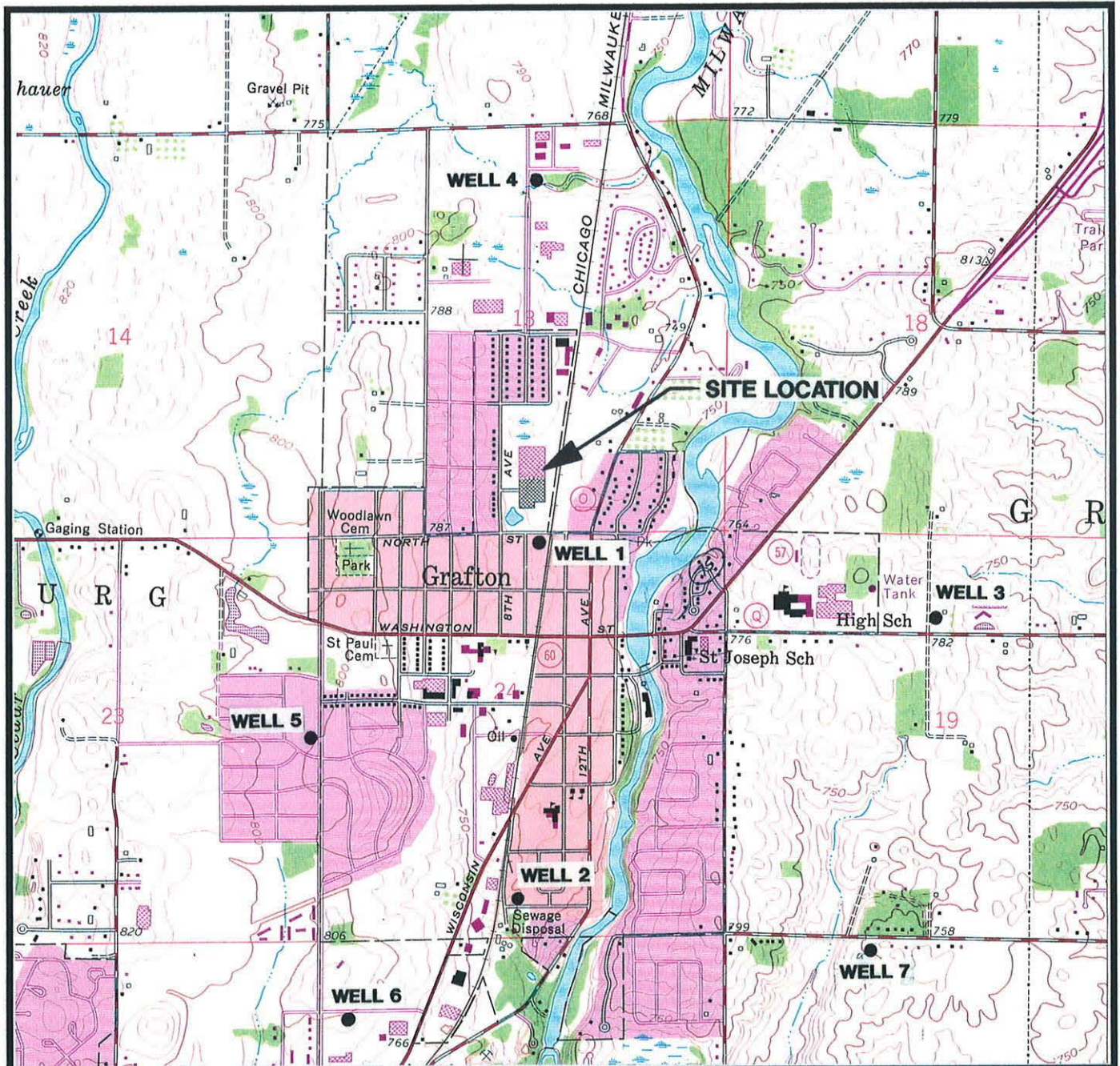
#### 1.1 Background

Tecumseh Products Company operates a manufacturing facility located at 900 North Street in Grafton, Wisconsin (see Figure 1). The primary manufacturing operations at present include machining and partially assembling air conditioner compressor parts, secondary trimming of aluminum diecast parts for small gasoline engines and transaxels, and light engine-related assemblies, such as brake brackets, and ignitions. This facility also houses a service-related training center and an aftermarket parts warehouse for the Engine and Transmission Group. A facility plan is presented on Figure 2.

During the 1960s, 1970s, and 1980s, the Grafton facility machined and assembled two-cycle gasoline engines. The processes and process equipment associated with the engine assembly operations included underground gasoline storage tanks for engine testing, chlorinated solvent vapor degreasers for parts/engine cleaning, and painting of assembled engines.

During the period of December 1988 through June 1992, eight underground storage tanks (USTs) were removed at the site. The tanks contained gasoline (leaded and unleaded), kerosene, Stoddard solvent, and motor oil. Evidence of releases was noted during five of the eight tank abandonments. During the course of investigating the extent of contamination which may have been associated with the Stoddard solvent tank, chlorinated volatile organic compounds (VOCs) were detected in the groundwater and soil. The source of the chlorinated VOCs has not been determined.

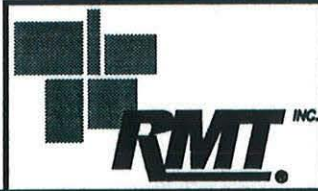
The surface topography of the site slopes gently from west to east. The surface soil consists of 10 to 12 feet of silty clay overlying a saturated sand layer. Beneath the building, the clay is overlain by 4 to 5 feet of sandy, gravelly, rocky fill. Although not encountered in any of the previous investigations, the well log from the Grafton Municipal Well #1, which is located directly south of the facility—just across North Street (see Figure 1)—indicates that bedrock is present at approximately 40 feet below ground surface.



**SITE LOCATOR MAP  
TECUMSEH PRODUCTS COMPANY  
GRAFTON, WISCONSIN**

\$\$\$DWG\$\$\$  
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\$\$\$SCALE\$\$\$

SOURCE: BASE MAP FROM CEDARBURG  
WI. 7.5 MINUTE USGS QUADRANGLE.



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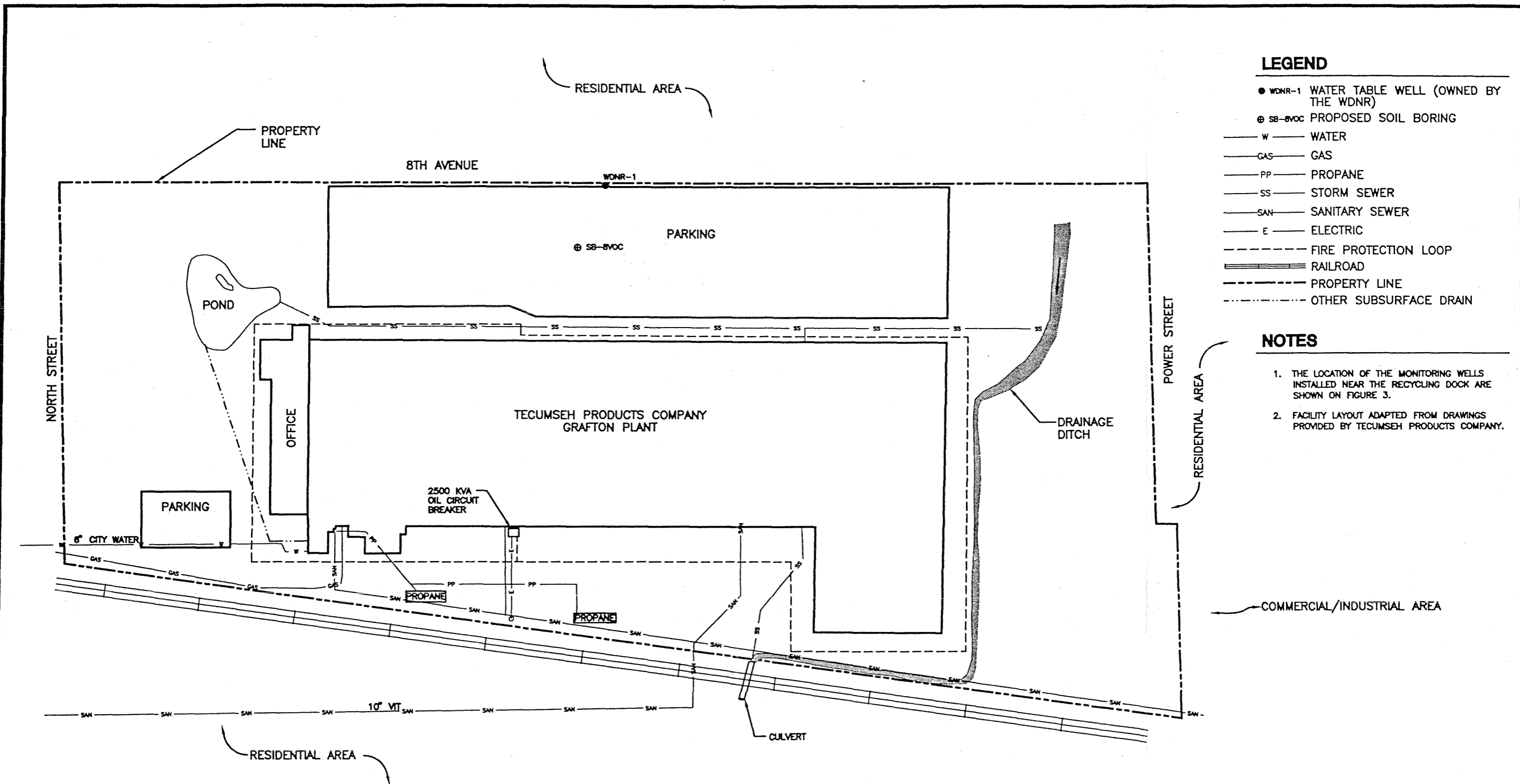
**FIGURE 1**

**LEGEND**

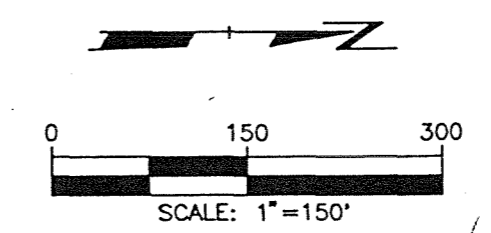
- WDNR-1 WATER TABLE WELL (OWNED BY THE WDNR)
- ⊕ SB-BVOC PROPOSED SOIL BORING
- W — WATER
- GAS — GAS
- PP — PROPANE
- SS — STORM SEWER
- SAN — SANITARY SEWER
- E — ELECTRIC
- - - - - FIRE PROTECTION LOOP
- ==== RAILROAD
- - - - - PROPERTY LINE
- - - - - OTHER SUBSURFACE DRAIN

**NOTES**

1. THE LOCATION OF THE MONITORING WELLS INSTALLED NEAR THE RECYCLING DOCK ARE SHOWN ON FIGURE 3.
2. FACILITY LAYOUT ADAPTED FROM DRAWINGS PROVIDED BY TECUMSEH PRODUCTS COMPANY.



**SITE PLAN  
TECUMSEH PRODUCTS COMPANY**



	DWN BY: DPR
	APPROVED BY: <i>PEH</i>
	DATE: AUGUST 1994
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AUG 04 1994      FIGURE 2

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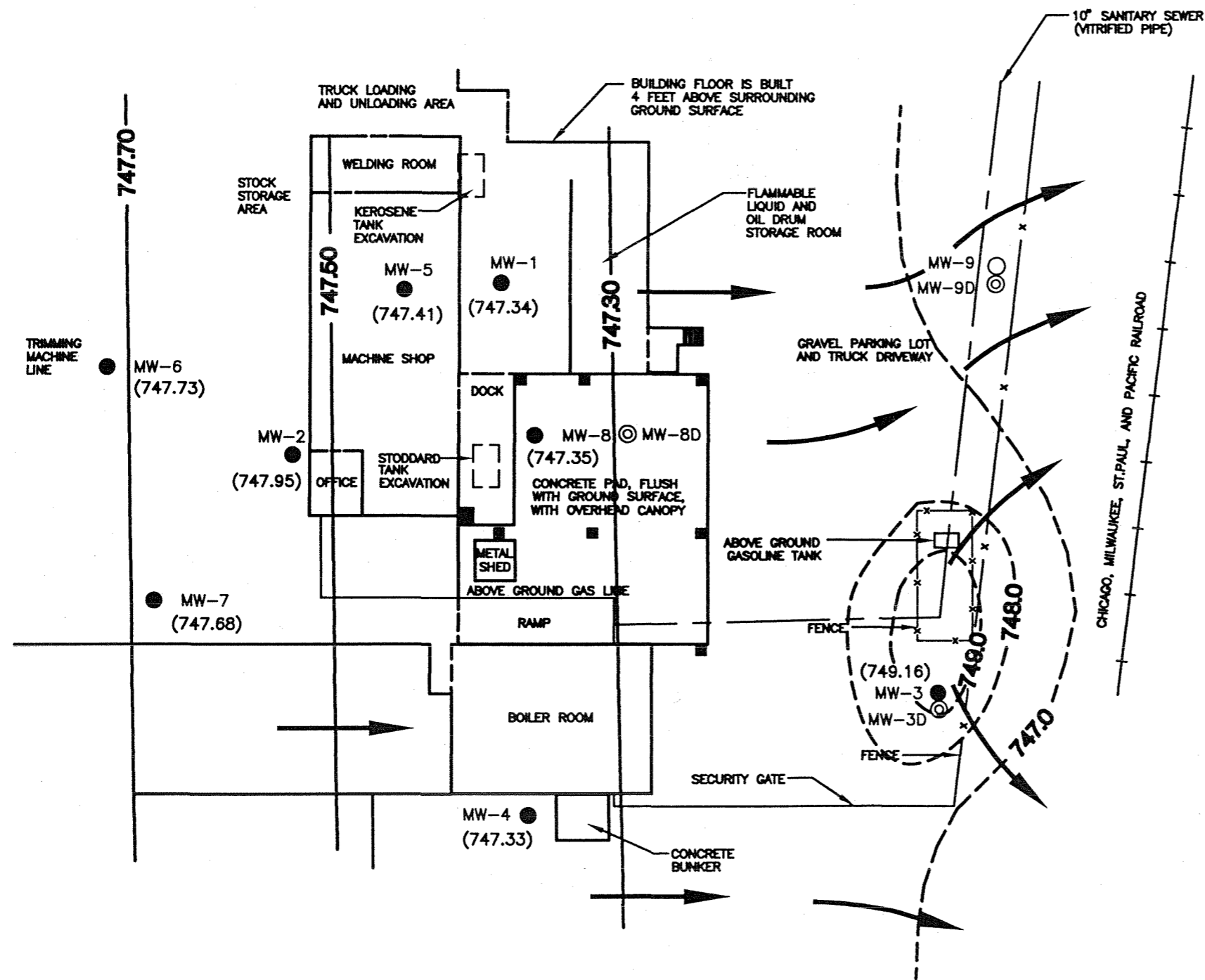
The water table is reported to be present at the silty clay - sand interface. Regionally, groundwater flows from west to east, toward the Milwaukee River, which is about 2,000 feet east of the site. On-site water level measurements generally confirm this flow direction if the data from MW-3 are disregarded, as shown on Figure 3. The water level in well MW-3 has been measured at consistently higher elevations than the water levels in the other on-site monitoring wells. The higher water level in MW-3 is hypothesized to be due to leakage from the vitrified clay sanitary sewer line that is more than 40 years old and that runs adjacent to the well (see Figure 3).

There are a total of eight water table wells at the site (see Figure 3). The following are the VOCs that may have been site-related that have been detected in groundwater during at least one of the four rounds of quarterly sampling completed between May 1993 and February 1994: 1,2-dichloropropane; 1,1-dichloroethane; 1,2-dichloroethane; 1,1-dichloroethene; cis-1,2-dichloroethene; naphthalene; toluene; 1,1,1-trichloroethane; trichloroethene; vinyl chloride; 1,2,4-trimethylbenzene; meta-xylene; and para-xylene. During the February 22, 1994, groundwater sampling round, eight VOCs were detected at concentrations greater than their respective Enforcement Standards (ESs) under Chapter NR 140, Wisconsin Administrative Code (TCE was the most frequently detected VOC in exceedance of its ES). In addition, five other VOCs were detected at concentrations greater than their respective Preventive Action Limits.

The WDNR conducted a hazardous waste inspection of the facility on April 24, 1994. In a letter dated, April 26, 1994, the WDNR provided their review comments on a series of reports prepared by previous consultants regarding the USTs. Among several other issues covered in this letter, the WDNR directed Tecumseh Products to submit a workplan for determining the source and extent (lateral and vertical) of the chlorinated VOCs at the Grafton facility.

## **1.2 Objectives**

RMT plans to conduct an additional subsurface investigation at the Grafton facility to better define hydrogeologic conditions beneath the facility and to evaluate the direction of migration and extent of chlorinated solvents in groundwater. As appropriate, investigations of the hydrogeologic conditions in the bedrock and of vadose zone contamination will be conducted during a subsequent phase(s) of investigation.



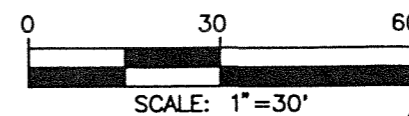
**LEGEND**

- MW-7 EXISTING WATER TABLE WELL
- MW-9 PROPOSED WATER TABLE WELL
- ⊙ MW-3D PROPOSED PIEZOMETER
- UPRIGHT SUPPORTS FOR CANOPY, CEILING
- DOORWAY
- (749.16) WATER TABLE ELEVATION (FEET ABOVE M.S.L.)
- 747.0- WATER TABLE CONTOUR (DASHED WHERE INFERRED)
- ➔ APPROXIMATE DIRECTION OF GROUNDWATER FLOW

**NOTES**

1. THE WATER LEVEL IN MW-2 WAS NOT INCORPORATED INTO THE WATER TABLE MAP DUE TO THE PRESENCE OF 3.1 FEET OF FREE PRODUCT (MACHINE OIL) IN THE WELL.
2. A SANITARY SEWER LEAK MAY BE THE SOURCE OF THE ANOMOLOUS HEAD AT MW-3.
3. THE LOCATION OF WELLS MW-9 AND MW-9D WILL BE BASED UPON THE RESULTS OF IN-FIELD GC ANALYSES FROM BORINGS. SEE TEXT FOR DISCUSSION OF LOCATION PROTOCOLS.

**WATER TABLE MAP  
(JUNE 9, 1994)  
TECUMSEH PRODUCTS COMPANY  
GRAFTON, WISCONSIN**



	DWN BY: DPR
	APPROVED BY: <i>LEH</i>
	DATE: AUGUST 1994
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FIGURE 3

\$\$\$DWG\$\$\$  
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## Section 2 SITE INVESTIGATION

### 2.1 Field Investigative Activities

The objective of the field investigation is to assess the extent and degree of chlorinated VOCs in the groundwater at the Tecumseh Products Company Grafton, Wisconsin, facility. The proposed drilling program is summarized in Table 1. The approximate locations of the planned soil borings are shown on Figures 2 and 3. Field methods for the planned work are included in Appendices A and B.

The work will include the following tasks:

- Install seven soil borings to the bedrock surface, approximately 40 feet below grade, and one soil boring to a depth of approximately 20 feet below grade, as described in Table 1. As the deep borings are advanced, soil samples will be collected with a 2-foot-long split-spoon sampler at 2.5-foot intervals until the water table is encountered. Soil and groundwater samples will be collected at alternating intervals below the water table. Soil samples will be collected at 4-foot intervals until bedrock is reached, and will be described according to the Unified Soil Classification System (USCS). As the deep borings are advanced, groundwater samples will be collected at 4-foot intervals, or where sand is encountered, using a Hydropunch sampler. The shallow soil boring will be located adjacent to a deep boring and therefore will be drilled without sampling soil or groundwater.
- Analyze the headspace concentrations of selected VOCs associated with the unsaturated soil and groundwater samples using a portable gas chromatograph (GC). Results of the field GC analyses will be used to obtain a vertical profile of contaminant distribution in the unconsolidated material. The headspace will be analyzed for TCE, 1,1,1-TCA, toluene, and xylenes.
- Select the soil sample from the MW-8D borehole that has the highest portable GC reading, and laboratory-analyze the sample for VOCs by USEPA Method 8021. In addition, in areas where the bedrock surface is overlain by clay soil, collect up to two soil samples from the clay and laboratory-analyze the samples for VOCs by Method 8021. Groundwater samples cannot be collected in the clay with the Hydropunch to assess the vertical migration of VOCs. The field GC results are not directly comparable to laboratory analytical results due to the complexities of partitioning VOCs between the soil, water, and headspace. The results of the laboratory analysis will provide an indication of whether VOCs are migrating through the clay.

TABLE 1

PROPOSED INVESTIGATION PROGRAM

Proposed Boring	Type of Boring or Monitoring Well	Estimated Depth (feet)	Primary Objective of Sample Point/Well
MW-3D	Piezometer	40	To characterize deep groundwater quality and measure the piezometric head at the southeastern edge of the site.
MW-8D	Piezometer	40	To assess the vertical extent of VOCs beneath the recycling dock and evaluate the vertical hydraulic gradient.
MW-9	Water table well	20	To characterize shallow groundwater quality and measure the water table elevation at the eastern edge of the site.
MW-9D	Piezometer	40	To characterize deep groundwater quality and measure the piezometric head at the eastern edge of the site.
SB-5VOC	Soil boring	40	To characterize groundwater quality along the eastern edge of the site and to delineate the northern extent of the VOC plume.
SB-6VOC <sup>1</sup>	Soil boring	40	To characterize groundwater quality along the eastern edge of the site and to delineate the northern extent of the VOC plume.
SB-7VOC <sup>1</sup>	Soil boring	40	To characterize groundwater quality along the eastern edge of the site and to delineate the northern extent of the VOC plume.
SB-8VOC	Soil boring <sup>2</sup>	40	To characterize upgradient groundwater quality at the western edge of the site.

NOTES:

<sup>1</sup> SB-6VOC and SB-7VOC are contingency borings. The northern extent of chlorinated VOCs in groundwater will be established by first installing SB-5VOC (and vertically profiling groundwater quality with the portable GC) and then, as necessary, moving northward with SB-6VOC and SB-7VOC until the concentrations of the selected VOCs are nondetectable on the portable GC. A water table well will be installed near the northern extent of the contaminant plume.

<sup>2</sup> SB-8VOC may be converted into a water table well if the WDNR well that is located along the western edge of the Tecumseh Products property is unacceptable for use as a monitoring well.

- Complete the shallow boring as a water table well and three of the deep borings as piezometers in general accordance with NR 141, Wisconsin Administrative Code. The depth of the screened intervals for the piezometers will be selected based on the results of the portable GC screening, such that the screened interval will correspond to the highest VOC concentrations. If no VOCs are detected, install the piezometers in a sand layer at or near the bedrock surface. If the bedrock surface is overlain by a clay till, install the piezometers in the deepest sandy layer overlying the clay.
- Develop the monitoring wells by surging and bailing, in accordance with NR 141 regulations.
- Survey the location and elevation of the existing and proposed monitoring well relative to a site datum and to mean sea level, respectively.
- Collect a complete round of groundwater level measurements from the existing and proposed monitoring wells to evaluate horizontal and vertical groundwater flow directions.
- Collect one round of groundwater samples from the new monitoring wells and from MW-3 and MW-8. Analyze the samples for VOCs using USEPA Method 8021. Also analyze a trip blank for quality control purposes. Collect one groundwater sample from MW-3, and analyze the sample for total phosphorus, total Kjeldahl nitrogen, chloride, and sulfate to determine whether the high water level observed at MW-3 is due to leakage from the sanitary sewer.
- Complete up to six single-well response tests to characterize the hydraulic conductivity of representative geologic materials.
- Place well development water, soil cuttings, and other materials derived from drilling operations in 55-gallon drums, and store the drums at an on-site location. Collect a composite sample of the soil cutting for disposal characterization. Analyze the sample for the volatile, semivolatile, and metal fractions of the TCLP; and manage soil disposal consistent with the TCLP findings.

## **2.2 Report Preparation**

Upon completion of the fieldwork and laboratory analyses described above, RMT will prepare a summary report for presentation to the WDNR at a meeting in October 1994. The summary report will include draft figures and data tables, along with the significant findings and conclusions drawn from the data. This document will also include recommendations for additional investigations that may be necessary.

A meeting with Tecumseh Products and the WDNR will be scheduled to discuss the WDNR's comments on the report and to establish cleanup objectives.

**Section 3**

**SCHEDULE**

A schedule for completing this phase of the project is shown on Figure 4. As discussed with WDNR staff at a meeting on July 20, 1994, the scope of the planned field investigation is acceptable and can be considered approved by the WDNR. Tecumseh Products and RMT will not initiate the field program, however, until the WDNR approves the field methods that were submitted on August 2, 1994, and that are reproduced in Appendices A and B. The field program can begin on August 15, 1994, if the WDNR approves the field methods by August 11, 1994.

## Project Schedule Tecumseh Products - Grafton Operations

Activity	July		August					September				October			
	18	25	1	8	15	22	29	5	12	19	26	3	10	17	24
Preparation and submittal of workplan to the WDNR	-----														
WDNR's review and approval of the workplan			-----												
Field program implementation					-----										
Laboratory analysis and data validation						-----									
Evaluation of data and preparation of summary report								-----							
Meeting with the WDNR to discuss findings and to establish the scope of further work												▲			



**APPENDIX A**

**FIELD METHODS**

**(Previously submitted to the WDNR on August 2, 1994)**

**APPENDIX A**  
**FIELD METHODS**

**1. Drilling and Soil Sampling**

Soil borings will be advanced by a truck-mounted drilling rig using 4.25-inch-inner-diameter (minimum) hollow-stemmed augers (HSAs). Samples collected from unconsolidated material will be collected using a 2-inch-inside-diameter, split-barrel sampler (split-spoon) as described in ASTM D1586-84, with the exception that the sampler will be driven 2 feet. Soil samples will be collected at 2.5-foot intervals in the unsaturated zone and at 4-foot intervals below the water table. When the auger is advanced to the top of the desired sampling interval, the decontaminated split-spoon will be lowered into the hollow-stemmed auger on the end of an appropriate drill rod. The split-spoon will be driven 24 inches or to refusal (more than 60 blow counts per 6 inches) using a 140-pound weight. The number of blows required to drive each 6-inch increment will be noted by the driller and also recorded by RMT personnel. Once driven, the split-spoon and rod will be withdrawn from the auger, and the split-spoon will be removed from the rods. The split-spoon will be opened, and the amount of sample recovery will be measured. Samples will be taken from the split-spoon for physical identification and field-screening for VOCs. If a low-permeability clay till is encountered in the bottom 10 feet of the unconsolidated material, samples will also be collected for chemical analysis. The split-spoon will be decontaminated before each sample is collected. (See Subsection 3.2 for decontamination procedures.)

A portion of each sample collected during soil sampling will be retained to develop a log of the borehole. Soil samples will be classified according to the Unified Soil Classification System (USCS), in accordance with ASTM D2488-90. Boring logs will be prepared on WDNR Form 4400-122 in accordance with Chapter NR 141, Wisconsin Administrative Code.

Soil borings that are not converted to groundwater monitoring wells will be abandoned by backfilling the borehole to the ground surface with a bentonite slurry grout using a tremie pipe. Borehole abandonment will be documented on WDNR Form 3300-5B in accordance with NR 141. The area around the borehole will be restored to its original condition.



**2. Groundwater Sampling using a Hydropunch Sampler**

Groundwater samples will be collected at 4-foot intervals in the deep borings using a Hydropunch II sampler. The groundwater samples will be collected at alternating intervals with the split-spoon soil samples. When the auger is advanced to the top of the desired sampling interval, the Hydropunch II will be lowered into the hollow-stemmed augers on the end of an appropriate drill rod. The Hydropunch II will be driven 24 to 30 inches beyond the end of the augers. The tool will then be pulled back approximately 6 inches to expose the intake screen. From 5 to 15 minutes will be required to collect sufficient groundwater for the in-field GC analyses. The Hydropunch II will be withdrawn and opened, and the groundwater will be transferred to a glass vial with a Teflon® septum seal. The vial will be taken to the in-field GC for immediate analysis.

The Hydropunch II will be decontaminated between each sample using the procedures outlined in Subsection 3.2.

**3. Decontamination Methods**

**3.1 Drilling Equipment**

The downhole equipment (both augers and drill rods) will be steam-cleaned before startup and after each boring. Decontamination of the drilling rig and downhole equipment will take place on a temporary decontamination pad constructed at an on-site location specified by the Owner. Decontamination water will be placed in WDOT-approved, 55-gallon drums that will be labeled and stored on-site pending the results of the groundwater analyses.

**3.2 Sampling Equipment**

Precautions will be taken to minimize the potential for contamination between samples. The split-spoon sampler will be cleaned prior to its initial use on-site and between samples. Cleaning procedures will involve scrubbing away soil material with a stiff brush using a trisodium phosphate soap and water solution, and then double-rinsing in clean, potable water.

At the end of drilling at a borehole, the split-spoons will be steam-cleaned, followed by the washing and rinsing procedures. Other reusable sampling equipment (bowls, spatulas, etc.) will be decontaminated by the washing and rinsing procedures. Spent decontamination liquids from the sampling equipment will be handled in the same manner as the water from the drilling equipment decontamination procedures.

**4. Investigation-Derived Wastes**

Soil cuttings generated during the performance of the soil borings will be placed in WDOT-approved, 55-gallon drums, and will be labeled and stored on-site. The soil cuttings will be segregated on the basis of the field GC results. A composite soil sample will be collected from the material that had field GC detections and from the investigation-derived soil that was stored on-site from previous field activities. The composite soil sample will be analyzed for the volatile, semivolatile, and metals fractions of the TCLP to assist in determining the disposal requirements for the investigation-derived waste.

Purge and development water will be placed in WDOT-approved, 55-gallon drums, and will be labeled and stored on-site pending the results of the groundwater analysis.

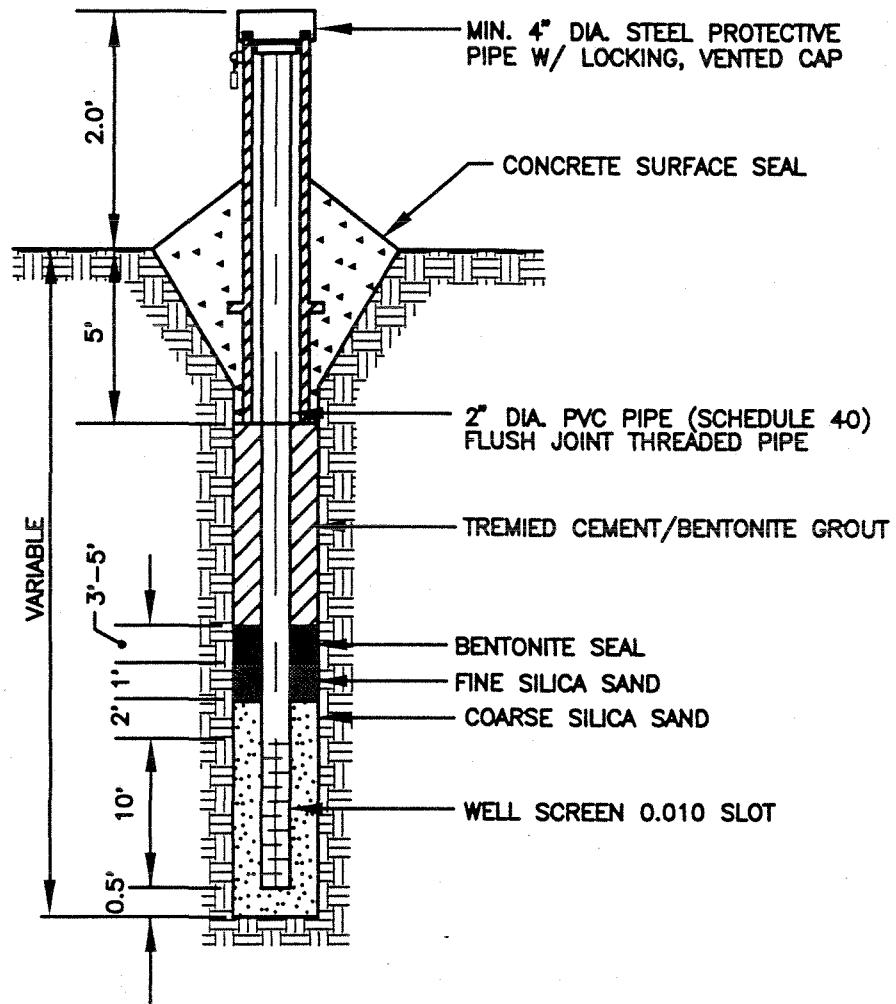
**5. Portable Gas Chromatograph Analyses of Soil Headspace**

Soil and groundwater samples will be field-screened for selected VOCs using a portable gas chromatograph (GC). A Photovac® Model 10S50 GC will be used for the VOC screening. The portable GC analysis will be used to characterize the presence and relative concentration of representative VOCs in the soil headspace on a near real-time basis. The soil headspace will be analyzed for trichloroethene (TCE), 1,1,1-trichloroethane, toluene, and total xylenes.


Standard operating procedures for the portable GC are included in Appendix B.

**6. Groundwater Monitoring Well Installation and Development**

The groundwater monitoring wells will be constructed using 2-inch-diameter Schedule 40 PVC with flush-threaded joints. The water table wells will be constructed as shown on Figure 1 and will have 10-foot-long well screens. The piezometers will have 5-foot-long well screens and will be constructed as shown on Figure 2 with the exception of MW-8D, which will have

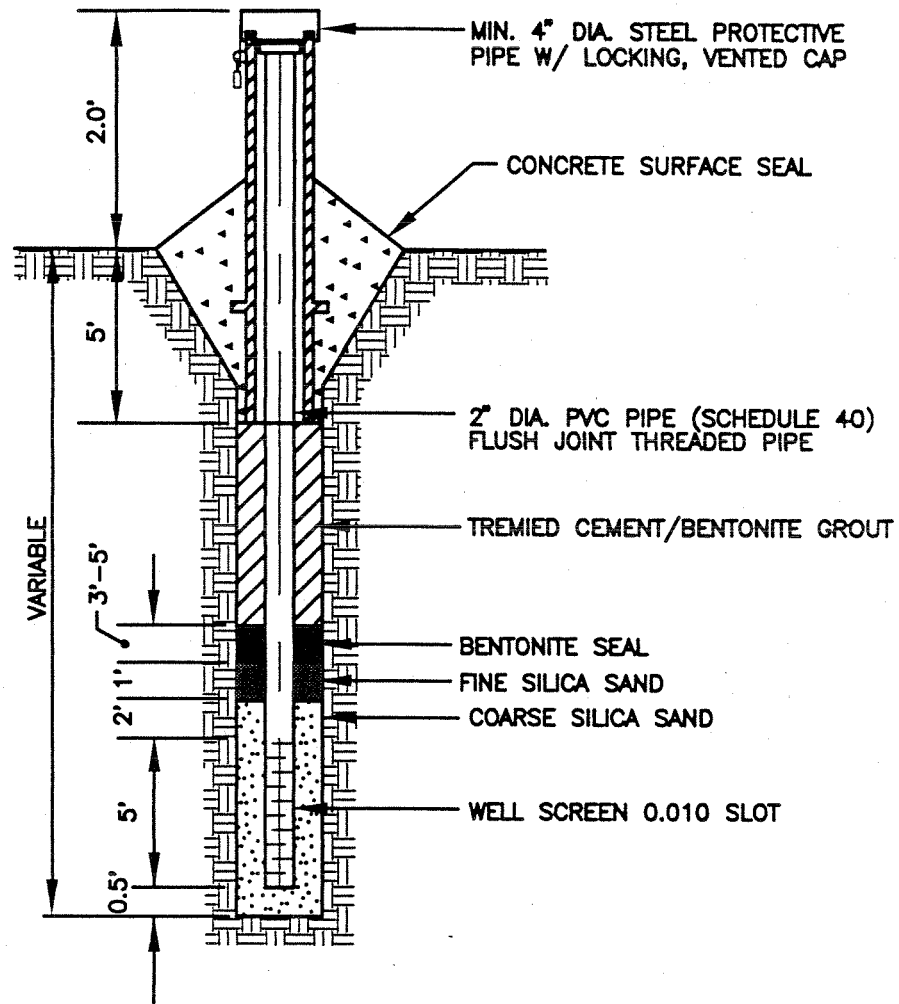


**WATER TABLE WELL  
CONSTRUCTION DETAIL**  
**TECUMSEH PRODUCTS COMPANY  
GRAFTON, WISCONSIN**


	DWN. BY: DPR
	APPROVED BY:
	DATE: JULY 1994
	PROJ. # 3084.01
	FILE # 30840105

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**FIGURE A-1**



**PIEZOMETER CONSTRUCTION DETAIL  
(STICK-UP SURFACE CASING)  
TECUMSEH PRODUCTS COMPANY  
GRAFTON, WISCONSIN**

	DWN. BY: DPR
	APPROVED BY:
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	PROJ. # 3084.01
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**FIGURE A-2**

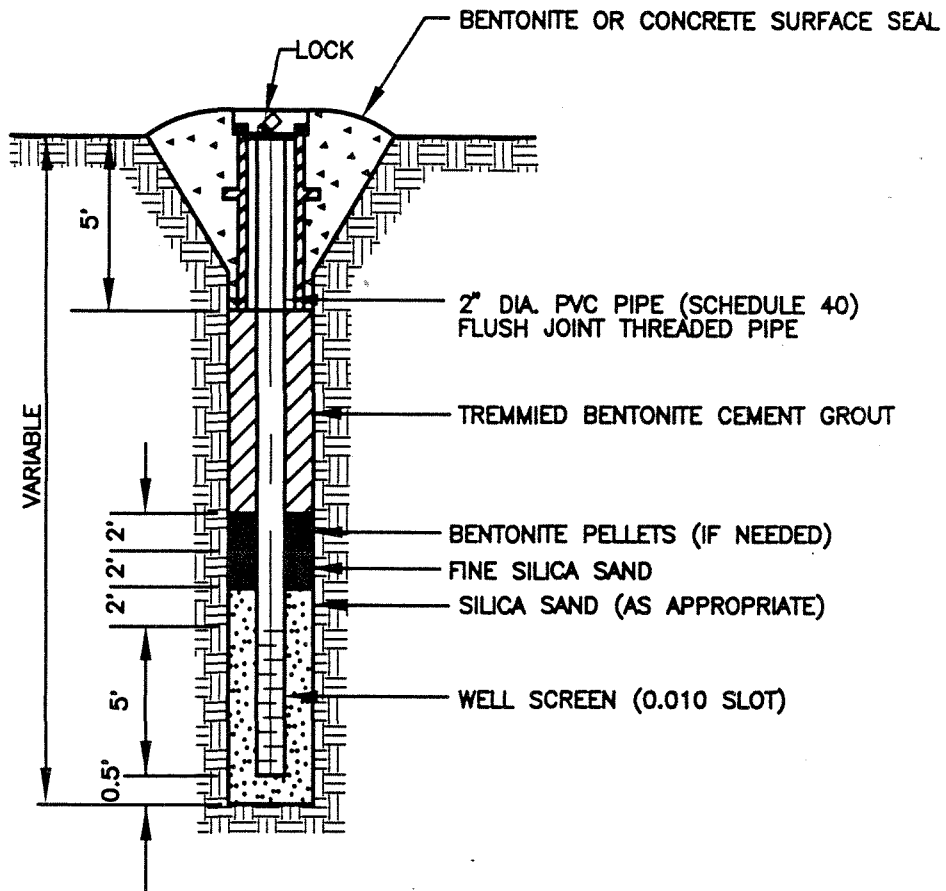
a flush-mount surface cover because of its planned location in the recycling dock area. MW-8D will be constructed as shown on Figure 3. As-built well construction diagrams will be prepared on WDNR Form 4400-113A.

The proposed water table monitoring well (MW-9) will be installed in a soil boring that will extend to approximately 7 feet below the water table. The proposed piezometers will be completed in the zone of highest contamination as determined by the portable GC. If no VOCs are detected with the portable GC, the piezometers will be installed in a sand layer at or near the bedrock surface.


The monitoring wells will be installed through the augers (minimum inside diameter of 4.25 inches). The bottom of the well will be firmly seated on sand, and the sand pack (medium-grained silica sand) will extend approximately 2 feet above the well screen. A 2-foot-thick fine silica sand layer will be placed on top of the filter pack, and a 2-foot-thick bentonite pellet seal will be placed over the fine sand. The remaining annular space above the filter pack seal will be filled with a bentonite slurry grout. A lockable protective steel pipe will be placed over the riser pipe.

After completion of the well installations, the wells will be developed in accordance with NR 141, Wisconsin Administrative Code, to improve the hydraulic connection between the well and the surrounding formation and to reduce the amount of sediment in the groundwater samples. The wells will be developed by surging a dedicated bailer across the screened interval of the well for 10 minutes, and then pumping the well until 10 well volumes have been removed or until the well is bailed dry three times. Well development will be documented on WDNR Form 4400-113B.

The vertical elevation of the top of the PVC casing of each well will be surveyed to an accuracy of 0.01 foot and will be referenced to the National Geodetic Vertical Datum (NGVD). The horizontal location of each well will be referenced to a site datum.



**PIEZOMETER CONSTRUCTION DETAIL  
(FLUSH-MOUNT COVER)  
TECUMSEH PRODUCTS COMPANY  
GRAFTON, WISCONSIN**

	DWN. BY: DPR
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	FILE # 30840107

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**FIGURE A-3**

## 7. Groundwater Sampling and Decontamination Procedures

Static water levels will be measured in the monitoring wells prior to purging or sampling. All groundwater level measurements will be made using a reference point established on the well casing. The reference point will be the highest point of the well casing. In order to prevent cross-contamination, the water level measuring device will be decontaminated between wells by rinsing first with a soapy water solution and then with distilled water.

The monitoring wells will be purged to remove stagnant water to ensure that the samples collected are fresh formation water. Before sampling each well, a total of four well volumes will be purged.

Groundwater samples will be collected from the monitoring wells immediately after purging. The procedures for the sampling of the monitoring wells are as follows:

- Set up and prepare meters.
- Prepare bottles by writing the date, the sampler's name, and the time of day in the sampler section.
- Collect a sample from the monitoring well using a pre-cleaned PVC bailer and a bottom-discharging device to prevent excessive amounts of agitation and aeration. The bailer will be dedicated for use in only one well.
- Place the samples on ice immediately.
- Perform field measurements for pH, temperature, and specific electrical conductance.
- Complete documentation of the sample collected in the sample log book and on the chain-of-custody forms.

The tests conducted in the field and the instrument calibration procedures are described below:

- **Temperature** - The temperature of the groundwater sample will be recorded immediately after the sample is removed from the well.
- **Specific Conductance** - The specific conductance of the liquid will be measured in the same groundwater sample used for the temperature measurement. A portable specific conductance meter will be used to measure the specific conductance of the groundwater sample.

- **pH** - The pH measurements will be made electrometrically using a combination electrode and portable pH meter. Portable meters with provisions of temperature compensation will be used. The meter and electrode will be checked against standard buffer solutions of known pH values (4 and 7).

All equipment used for sampling that is not dedicated (e.g., bailers, water level measuring devices, etc.) will be decontaminated prior to initial use by the following methods:

- Prepare a soapy water bath using laboratory-grade detergent.
- Unwind and soak water level measuring devices in soapy water, and wipe clean with a cloth.
- Rinse all equipment with tap water.
- Rinse all equipment, except water level measuring devices, inside and outside with diluted 1:1 nitric acid.
- Rinse all equipment with deionized water.
- Dry all equipment, except water level measuring devices, in the oven at 105°C, and seal in polypropylene plastic to prevent contamination.

The procedures to be followed for cleaning equipment in the field between wells are as follows:

- Rinse all equipment with soapy water.
- Rinse all equipment with distilled water.
- Place rinseate in WDOT-approved 55-gallon drums, and store at an on-site location specified by the owner.

#### 8. **Hydraulic Conductivity Testing**

In-field hydraulic conductivity testing will be performed on monitoring wells completed in sandy soil. If visual observations of the soil suggest that the hydraulic conductivity of the soil is greater than  $10^{-4}$  cm/s, a pressure transducer coupled to a data logger will be used to record the recovery of hydraulic head following an instantaneous head change produced by a solid PVC slug. Lower permeability soil will be tested by removing water from the monitoring wells with a bailer and recording recovery rates with an electric water level indicator and clock.



The recovery data obtained by either of the methods described above will be analyzed using the methods described by the following:

Bouwer, H., and R.C. Rice. 1976. A slug test for determining hydraulic conductivity of unconfined aquifers with completely or partially penetrating wells. *Water Resour. Res.*, 12: 423-428.

Hvorslev, M.J. 1951. Time lag and soil permeability in groundwater observations. U.S. Army Corps of Engineers, Waterways Expt. Sta. Bull 36.

#### 9. Chain-of-Custody Procedures

The possession of samples will be traceable from the time of collection through the use of chain-of-custody procedures. Specific chain-of-custody forms will accompany all sample shipping containers to document the transfer of the shipping containers and samples from the field collection point to the laboratory receiving the samples for analysis. The procedures to be implemented are as follows:

- Properly identify and label the samples in the field.
- Complete a chain-of-custody form in the field, indicating sample identification, containers filled, sampling date, sampling time, and sample collector's name.
- Pack shipping container with samples, chain-of-custody forms, and ice. The sample container should be assigned a chain-of-custody form, which travels with the container.
- Seal, date, and ship the coolers to the appropriate laboratory using an overnight delivery service or via RMT field vehicle.
- Receive and check shipping containers in the laboratory for broken seals or damaged sample containers.



**APPENDIX B**

**RMT STANDARD OPERATING PROCEDURE FOR  
PORTABLE GAS CHROMATOGRAPH ANALYSIS**

**(Previously submitted to the WDNR on August 2, 1994)**

## APPENDIX B

### RMT STANDARD OPERATING PROCEDURE FOR PORTABLE GAS CHROMATOGRAPH ANALYSIS

#### 1. Parameters To Be Analyzed

The four parameters to be analyzed by the portable GC are trichloroethene, 1,1,1-trichloroethane, toluene, and total xylenes. These parameters were selected on the basis of previously identified constituents in groundwater and soil, and on the sensitivity of the portable GC to these parameters. The peak area of targeted parameters will be determined for each sample. Quantitation of the specified compounds is determined by comparing peak areas of standards to those of samples.

#### 2 & 3. Range of Measurement and Limits of Detection

The anticipated working linear range and limits of detection are presented in Table 1. These detection limits are based on the concentration represented by the minimum recorded peak area for the GC (0.1 v-sec) at normal operating conditions. The maximum measurable concentrations for the different parameters are around 1,000  $\mu\text{L/L}$  (parts per million by volume). The  $\mu\text{L/L}$  (or ppm) units will be used to avoid confusion between concentration units in water and air.

#### 4. Sample Matrix

As a means of screening soil samples for VOC contamination, the headspace over the soil sample is measured for the VOCs. Thus, the sample matrix is air that has been contacted with the sample. Headspace measurement evaluates soil concentrations indirectly by determining the parameter concentration in the air in contact with the soil (i.e., the headspace). Parameter distribution between the soil and headspace is affected by a number of soil and parameter properties, including the parameter Henry's Law constant, and soil organic and water content and consistency. Since some of these properties can vary between samples, the parameter distribution between the soil and headspace can vary between samples. Headspace measurement thus does not give a direct measurement of parameter concentration in the soil, but rather gives a qualitative indication of the soil concentration.

<b>TABLE B-1</b>	
<b>DETECTION LIMITS FOR CHLORINATED AND BTEX COMPOUNDS ANALYZED BY PORTABLE GC</b>	
<b>Parameter</b>	<b><math>\mu\text{L/L}</math></b>
Trichloroethene	0.01
1,1,1-Trichloroethane	0.3
Toluene	0.03
m,p-xylene	0.03
o-xylene	0.04

VOC concentrations in water can be estimated by measuring the headspace concentration over the water in a VOA vial. At equilibrium, the VOCs partition between the headspace and water in accordance with Henry's Law, as follows:

$$H = \frac{\text{concentration in air}}{\text{concentration in water}}$$

where H is a constant

Standards will be prepared using the procedures suggested by Photovac® Technical Bulletin #27 (Attachment 1). The standards are analyzed at the same time as the samples, and each parameter's operational partitioning coefficient is calculated. The operational partitioning coefficient is the reciprocal Henry's Law constant, with the effects of temperature, equilibration time, kinetics, and relative volumes of water and headspace included.

The operational partitioning coefficient (OPC) is calculated as follows:

$$\text{OPC} = \frac{\text{original concentration in water, } \mu\text{g/L} \times (\text{gain}) \times (\text{injection volume})}{\text{concentration in headspace, v-sec}}$$

Water concentrations are determined by preparing samples and standards, and then multiplying the sample concentration by the OPC and dividing by gain and injection volume, as follows:

$$\text{Water concentration, } \mu\text{g/L} = \frac{(\text{OPC})(\text{GC response, v-sec})}{(\text{gain})(\text{injection volume, } \mu\text{L})}$$

##### **5. Principle, Scope, and Application**

Headspace analysis is a convenient means of screening for VOC contamination. In principle, VOCs contained in soil or water will partition between the matrix and any air in contact with the matrix, with partitioning dependent on the VOC concentration in the matrix as well as on several other factors (Devitt, et al., 1987). Analysis of the soil headspace can be done in the field using a portable gas chromatograph, and can provide near real-time screening analysis of VOCs. Due to the variety and variability of factors that influence VOC partitioning between the matrix and air, headspace analysis is best used for screening VOC levels rather than for quantitatively analyzing the VOC content in the field. However, measurement of the VOC concentration in the gas itself is a quantitative measurement.

Headspace screening is useful for locating areas of contamination and relative levels of contamination, for selecting samples for further laboratory analysis, and for determining the areas that should be further investigated (e.g., soil borings or monitoring well installation).

#### **6. Interferences and Corrective Action**

Interferences are uncommon in the analysis for the parameters analyzed under standard operating conditions. However, components of gasoline coelute with di- and trichloroethylene and, if gasoline is present, it is difficult to quantify di- and trichloroethylene. Generally, however, gasoline and the chlorinated ethylenes are not found in the same sample. In addition, ethylbenzene and m- and p-xylene peaks overlap. If both peaks are present, the peak is calculated as if it contained the constituent giving the large peak, and a note is made that both compounds were present.

Dirty apparatus can cause problems with a portable GC, since many surfaces (notably teflon) can adsorb and desorb gaseous constituents. Several precautions and cleanup steps are used to avoid such cross-contamination. First, standards are transported separately from the syringes, septa, etc., so that no cross-contamination during transport occurs. Second, column and syringe blanks are run whenever contamination is suspect. Third, syringes are cleaned between each run by removing the plunger and allowing the contamination to disperse, or by purging the syringe or plunger with compressed air from the air cylinder. If syringe contamination is suspect, a syringe blank is run in which room air or compressed air is injected into the GC with the syringe in question. Syringes that remain contaminated after purging are baked overnight in a portable oven. Typically, ambient air contamination has not been found to be a problem.

#### **7. Safety Precautions**

Samples are contained in 40-mL VOA vials, and the analyst does not come into direct contact with the soil. If soil needs to be transferred from one vial to another, or if a vial breaks, then the analyst will take the normal precautions used when working with chemicals, e.g., washing hands after working with the material, not eating while handling the material, etc. All pure solvents used for making standards are likewise kept in 40-mL VOA vials, and the analyst does not come into contact with the solvents.

Standard laboratory practice precautions are taken when working with the gas cylinders used for gas supply to the GC. The cylinders are either secured to the wall by chains or, in places where no wall mountings are available, supported by a gas cylinder stand.

**8. Sample Size, Collection, Preservation, and Handling.**

Water and soil samples are collected in 40-mL VOA vials with a septum top. If the samples are analyzed in the field, the vials are filled approximately half full with soil when the sample is collected. If the samples are analyzed in the laboratory, the vials are filled completely in the field, and a second vial is then filled half full in the laboratory. The vials are then allowed to equilibrate at room temperature for at least 30 minutes. No preservatives are used in the samples. After the equilibration time, an aliquot of the headspace is removed with a syringe through the septum, and is injected into the GC for analysis. Injections are repeated until the peaks of interest are on scale. The sample is discarded by either returning the vials to RMT Laboratories or by returning the soil to the sampling area. At RMT Laboratories, the vials are opened in a hood, and the VOCs are allowed to volatilize, after which the soil is discarded. After the soil has been removed from the vials, the empty vials are discarded.

**9. Apparatus**

The GC used is a Photovac® Model 10S50 Portable Gas Chromatograph. Other sampling equipment used for the headspace analysis is listed in Table 2.

**10. Routine Preventive Maintenance**

The routine preventive maintenance procedures used in day-to-day operation of the GC are described in section 14 below. The procedures include running column and syringe blanks at the start of a day's operation, and when syringe or instrument contamination is suspected. The injection port septa is changed after 50 to 75 injections. VOA vial blanks are run if vial contamination is suspected. Vial contamination would be suspected if similar peak patterns were found in several soil samples from different areas.



TABLE B-2

PORTABLE GC FIELD EQUIPMENT CHECKLIST

<u>EQUIPMENT</u>	<u># NEEDED</u>	<u>CHECKOFF</u>
INSTRUMENTS		
Photovac® 10S50		
Battery pack for oven		
Electrical cord for using GC with 110 V		
Electrical cord for charging battery pack		
Electrical cord for GC from battery pack		
Gas flow meter and connecting gas lines		
Gas tank regulator and connecting lines or internal tank refill gas line		
0.1-ppm grade air tank		
GC SUPPLIES		
Plotter pens		
Plotter paper		
Extra UV lamp		
White Teflon®-coated septa for GC		
PAPERWORK		
Field GC logbook		
Field notebook		
Photovac® GC instruction manual		
SYRINGES		
10 µL		
25 µL		
100 µL		
250 µL		
1,000 µL		
Syringe needles		

TABLE B-2 (CONTINUED)

PORTABLE GC FIELD EQUIPMENT CHECKLIST

<u>EQUIPMENT</u>	<u># NEEDED</u>	<u>CHECKOFF</u>
SAMPLE CONTAINERS		
VOA vials		
1-L gas sample bottles		
250-mL gas sample bottles		
Green septa for sample bottles		
Labels for vials/sample bottles		
Standards (pure solvent/gas STD)		
TOOLS		
Adjustable wrench for gas cylinder		
Small wrench for gas line fittings		
Slotted screwdriver		
Phillips head screwdriver		
MISCELLANEOUS		
Kim Wipes®		
Paper hand towels		
Markers		
Pens		
Calculator		
Knife		
Rubber bands		
Paper clips		
Water bottle		

The neat solvents used for preparing standards are stored and transported separately from the other GC equipment to avoid cross-contamination. Septa, syringes, and the plastic portions of the gas sample bottles are stored in organic contamination-free areas.

A common analytical problem is clogging or partial clogging of the injection syringe needles. Occurrence of partial clogging is determined by running replicate samples. If the results cannot be replicated after several runs, the syringe needle is changed. Clogged syringes are detected by injecting air into a water-filled vial. If no bubbles are observed, then the needle is changed.

#### **11. Reagents and Calibration Standards**

Pure solvents and calibration standards are purchased from chemical supply companies. Commercially prepared gas standards are purged into the gas sample bottles from pressurized gas cylinders. An example of standard concentrations is shown in Table 3. Water standards are prepared by appropriate dilution of pure solvent. Water standards are prepared in 40-mL VOA vials using methanol and water as diluents. The final working solution contains 20 mL of liquid in the 40-mL VOA vial. The constituents in water are allowed to come into equilibrium with the headspace of the VOA vial prior to injection of this headspace to the GC. A new standard is prepared when the septum on the gas bottle becomes too perforated, or when the solvent volatilizes through the septum. A Photovac® Model 10S50 can analyze only phase-phase samples, so all samples and standards must be in the gas phase prior to injection.

Chromatograms of the standard are compared with previous standard chromatograms to ensure that the standard was prepared properly.

#### **12. Calibration Procedures**

A. Standards should be prepared according to the protocol given above.

<b>TABLE B-3</b>	
<b>EXAMPLE OF A COMMERCIALY PREPARED STANDARD</b>	
<b>Compound</b>	<b>Gas Concentration ppm (<math>\mu\text{L/L}</math>)</b>
Trichloroethene	15.4
1,1,1-Trichloroethane	150.0
Toluene	19.9
Total xylenes	82.0

- B. A standard should be run after the column blank when starting the instrument. For the most accurate results, standards should be prepared daily. The standard can be used for calibrating the instrument response factor on the first run or runs, and then for peak identification for the rest of the day.
- C. After the instrument is calibrated, a second standard should be run to verify that the first standard is reasonable. If the results differ by more than 5 percent, rerun and recalibrate (if necessary) the instrument using a different syringe or a different needle.
- D. Record on the strip chart both the V-sec and ppm readings on standards that are used for calibration, so that the calibration factor the instrument is using can be calculated.
- E. If there is a question on the identity of a given peak in a sample run, run a standard to determine the retention time of the compound of interest. Peak identification by the GC can be in error if the airflow rate or column temperature drift. The analyst should be familiar with the peak patterns of the compounds of interest, check to ensure that the GC is correctly identifying peaks, and check retention times against standards if any questions arise.

### **13. Sample Preparation**

Sample preparation is discussed in Section 8 above.

### **14 & 15. Step-by-Step Analytical Methodology**

The daily operation of the GC, including routine procedures, preventive maintenance, and daily quality control procedures, is described below:

- A. Connect the power supply for the GC (unless using the internal battery).
- B. Connect the battery for the column oven to the external DC input connector. Set column to the desired temperature, and allow to heat for approximately 30 minutes to reach operating temperature.
- C. Connect the exhaust gas lines to the gas flow meter. The left side of the flow meter measures flow through the detector and is connected to the "Detector Out" port, while the right side is connected to the needle valve on the "Aux Out" post.
- D. Connect the input gas lines, turn the gas flow on, adjust the flow through the column, and backflush to the appropriate values. The backflush flow should be set slightly higher than the column flow. If using the internal tank, fill the tank before adjusting the flow. The gas flow through both the detector and backflush lines is set by the "A + B" valve at the center left of the front panel. The backflush flow is also controlled by the "Aux Out" needle valve. Note that the internal gas pressure should be set at 40 psi.

- E. Turn the instrument on (Note: gas must be flowing past detector before the lamp is turned on). The instrument will read "LAMP NOT READY. PLEASE WAIT" for a few minutes after turning the instrument on. If the lamp does not come on after several minutes, as indicated by the instrument reading "READY," turn the instrument off ("OFF", then "ENTER") and then back on, and wait for a minute. If the lamp still does not turn on, use the special Teflon® screwdriver to adjust the lamp power supply on the lamp box inside the unit. If the lamp still does not light, change the detector bulb.
- F. Set daily information, using USE button. Also enter project information, if necessary, using INFO button.
- G. If you are not sure of the GC's valve timing, press "TEST" then "ENTER." The GC will print out the Event timing. Event 1 should be set for an ON time of 8 (sec) and one OFF time of 10 (sec). Event 1 controls the buzzer for sample injection, and the injection port sequence. Event 3 controls the backflush start time. The ON time should be 0 (sec), and the OFF time should be one-fourth to one-fifth of the retention run time of the slowest analyte of interest. The run time is set by the CYCLE button.
- H. Set gain to desired value. (Note: Gain defaults to 2 when instrument is turned off.)
- I. Change septum on injection port, if necessary (septa are good for approximately 50 injections).
- J. Prepare daily injection log. All runs should be recorded on log.
- K. Run a column blank (no injection) and a syringe blank.
- L. Prepare appropriate standards (if necessary). Directions for standards preparation are given in Section II.
- M. Run standard. Recalibrate instrument, if necessary (see Calibrating the GC in Section II-12). If possible, obtain the peak areas in V-sec as well as ppm-V, so that the instrument calibration factor (in  $\mu\text{L/L}/\text{V-sec}$ ) can be determined.
- N. If time permits, run a syringe blank (an injection of room air or 0.1 air) to ensure syringe cleanliness using the syringes that will be used in the day's work.
- O. Run samples. Adjust the injection volume until the peaks of interest are on scale and preferably more than 1/4 of maximum size for chart paper. If time permits, runs should be duplicated and average values should be used for quantification.
- P. Record sample ID, injection volume, and gain on the injection log. Also record sample ID and injection volume on chromatogram at the start of the run.
- Q. Record the results of the run on the calculation sheet for the project. Correct the GC output for injection volume (and gain if results are in mV or V-sec) to those values used for standard. When two replicate runs are made, calculate for both separately, and then average the results.

- R. Record the corrected results on the results sheet for the project. One copy of the results sheet should be given to PM/person-in-charge, and one copy should be placed in the project notebook.
- S. Clean syringes by removing the plunger after an injection and letting the plunger air.
- T. If replicate runs are not satisfactory (< 10 percent difference is a suggested guideline), reinject sample. If third run is still inconsistent, replace the needle on the syringe, and then rerun the sample. Also check for syringe contamination by running a syringe blank. Check for plugged syringes by injecting air into a vial filled with water.
- U. If instrument is not recognizing obvious peaks, recalibrate the known peak. If unsure of peak ID, run standard for peak identification. (Note: Once the septa is punctured in the gas sample bottle, the VOCs are slowly lost from the bottle. Therefore, the standard should be used for calibrating the concentration only shortly after the standard is prepared. The standard can still be used for peak identification until the peaks disappear.)
- V. Standards should be run periodically throughout the day, for peak identification (not for recalibration of concentration) and at any time when questions on peak identification arise. At least three standards should be run each day.
- W. Column and syringe blanks should be run periodically throughout the day, and if any questions of syringe contamination arise. Column and syringe blanks are particularly important when the sample concentration is low, or if there is a sharp drop in sample concentration (< factor of 5 change in concentration).
- X. Under normal operation, the maximum gain is 20, and under all conditions, the maximum injection volume is 1,000  $\mu$ L.
- Y. After 50 to 60 injections, change the injection septum.
- Z. After the last run has been completed, disconnect the oven battery from the instrument, and then shut the power off to the instrument. After the instrument has turned off, turn off the airflow, either by closing the tank or decreasing the regulator (if using an external tank). If using the internal tank, turn off the flow by using the instrument flow control knob.
- AA. At the end of a day's run, tear off the chart paper and mark the end with the date, project name and number, and if possible, the samples run.
- BB. If the instrument is using the internal power supply, then it should be recharged overnight. Fill the internal gas tank in the instrument, and bring the GC to a place where there is a 110 v power supply. Plug the instrument in, turn the airflow to a very slow rate (5 to 10 mL/min), and then turn the instrument on overnight. Be sure the air tank is full or that it has sufficient air to keep air running through the detector throughout the night.

**16. Data Treatment**

The Photovac® measures the area under peaks for the compounds in the injected gas sample. When using a "Library," the area, in V-sec or MV-sec, is converted to μL/L (ppm-V) in the gas phase by calibrating the instrument using a known concentration of the compound in question (with gas standards). The instrument compares areas of standards with sample peak areas to determine the concentration of the unknown. The instrument corrects for gain, but not for injection volume. The instrument's concentration output should be corrected for injection volume as follows:

$$(actual\ conc.,\ \mu L/L) = (instrument\ reading,\ \mu L/L) \left( \frac{injection\ volume\ of\ standard}{injection\ volume\ of\ sample} \right)$$

If the reading is given in V-sec, a correction needs to be made for gain as well as for injection volume, i.e.,

$$(actual\ conc.,\ \mu L/L) = (instrument\ reading,\ V-sec) \left( response\ factor\ \frac{\mu L/L}{V-sec} \right) \left( \frac{injection\ volume\ of\ standard}{injection\ volume\ of\ sample} \right) \left( \frac{gain\ setting\ for\ standard}{gain\ setting\ for\ sample} \right)$$

where the injection volume and gain are the values for the standard at the time the instrument was calibrated.

The response factor is the conversion factor for the V-sec given by the detector at a given standard concentration, i.e.,

$$response\ factor = \left( \frac{standard\ concentration,\ \mu L/L}{instrument\ reading,\ V-sec} \right)$$



The response factor is specific for the injection volume, gain, and detector response.

Gas measurements are commonly reported in ppm or  $\mu\text{g}/\text{m}^3$ . A gas phase unit of ppm is a  $\mu\text{L}/\text{L}$ . To avoid confusion between measurements made on different matrices (air, water, or soil) units of  $\mu\text{L}/\text{L}$  are used in the calculations.

#### 17. Data Deliverables

Portable GC Results Notebooks will be prepared. The notebook will consist of the following sections:

- A. Project information
- B. Standards preparation and SOP for the sample handling and GC operation
- C. Injection logs
- D. Calculation sheets
- E. Results summary sheets
- F. Replicate comparison and standards results sheets
- G. Chromatograms

After the fieldwork, the chromatograms are xeroxed and placed in the appropriate section. Replicates are recorded separately, and compared in a separate section of the notebook. The calculation and results summary sheets should be checked by the QC reviewer. The notebook should be comb bound and placed in the project file. The calculation and results summary sheets can be xeroxed and stored at the GC operator's desk for later reference, if appropriate.

The notebooks contain the information required to follow the results from the original chromatogram to the final results sheet. Standards are included so that retention times can be checked. However, syringe or column blanks, runs that went off-scale, and other miscellaneous runs are not included. The original chromatograms are stored in the RMT Applied Chemistry Laboratory.

### **18. Quality Control**

The degree of QA/QC for the portable GC use is dependent on the use of the results, and should be adjusted as appropriate. It should be recognized that the GC itself is a precise analytical instrument, capable of providing as consistent and reliable results as a laboratory GC, if used under optimum conditions. However, use in the field under less controlled conditions increases the analytical variation in the results. Further, and more important, soil headspace analysis is designed to be a screening method, giving approximate indicators of soil concentrations. There is inherent variability in a soil headspace measurement due to changes in the soil itself that cannot be eliminated by QC procedures during the analysis. The extent of QC should be appropriate to the end use of the results.

During field operation of the GC, the operator checked for syringe clogging or contamination, machine malfunction, and miscalibration as discussed in Sections 14 and 15. If replicate runs vary by more than 10 percent difference between the results, the sample is reanalyzed by repeating the injections until the results do replicate or the cause of the poor replication is identified.

The purpose of the QA/QC procedures can be divided into three areas, as follows:

#### A. Verification

Much of the verification of peak identification, syringe cleanliness, and proper operation is done during machine operation, and is discussed in Section 12, Calibration Procedures. During the QC check of the results, the following steps are needed:

- i. Sample and standard peak retention times and peak patterns are compared. GC identification of the peaks is checked. Incorrect GC peak identification is noted on the chromatogram.
- ii. If a question of peak identification arises that cannot be resolved by comparing sample peak retention time with that of the standard, relative retention times for the unknown and for a known compound are calculated and compared between the sample and standard.
- iii. If a question of peak identification arises after a relative retention time check, the peak is identified as the compound of interest with a note saying "Tentative Identification."

B. Data and Calculation Checks

- i. All results to be reported should be recorded on the calculation sheet.
- ii. The results should be corrected for injection volume and gain. Note: the Photovac® automatically corrects for gain if the reports are presented in ppm, but does not correct for gain if the results are presented as V-sec.
- iii. Further calculations should be recorded on the DATA CALCULATION SHEET.
- iv. The corrected results should be recorded on the sample results sheet. All results should be reported as  $\mu\text{L/L}$  (ppm-V).
- v. All data transcriptions and calculations should be checked by a QC person, unless otherwise instructed. The QC person should check the results for accuracy of transcription, and spot check the calculations. Furthermore, the QC person should compare the final results for reasonableness, based on previous results or anticipated results.
- vi. A xerox of the chromatograms used for calculation should be made. The xerox facilitates QC checking and project file documentation. If duplicate injections were made, both chromatograms should be recorded. Further, if there is any uncertainty about peak identification, then the standards used for retention time calibration should be xeroxed also.
- vii. The QA/QC person should initial and date each page of data calculation or results sheet checked and any corrections that are made. Corrections should be in a different color ink (e.g., blue or red) than the original.

C. Documentation

- i. A portable GC project notebook should be used unless there are instructions not to. The project notebook should consist of the following sections:

*Section Topic*

- a. General Project Information
  - Includes the proposed Scope of Services if available
- b. Standard Operating Conditions
  - Standards preparation forms
  - Standards chromatogram
  - Calculation equations
  - Sample handling instructions (if prepared)
- c. Results summary forms
- d. Daily injection log

- e. Calculation sheets and chromatograms
  
- ii. While the project is active, the notebook should be kept in a 3-ring binder (unless otherwise specified).
  
- iii. After the project is completed, the notebook should be comb bound, with a cover page, and stored in the project files. The GC operator is responsible for putting the final notebook together.
  
- iv. The original chromatograms should be stored in the Applied Chemistry Laboratory. The chromatograms should be organized by project and date.

**19. References**

Devitt, D.A., R.B. Evans, W.A. Jury, T.R. Starks, B. Eklund and A. Gnoison. 1987. Soil gas sensing for detection and mapping of volatile organics. EPA 600/8-87-036, USEPA, Las Vegas, Nevada.

Photovac® - Technical bulletin #27. Photovac, Inc., Huntington, New York.

**ATTACHMENT 1**  
**PHOTOVAC® TECHNICAL BULLETIN #27**

# PHOTOVAC

## Technical Bulletin

# 27

PREPARATION OF AQUEOUS STANDARDS FOR  
GROUNDWATER ANALYSIS

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Many of the chemicals mentioned herein are of a hazardous nature. Photovac expressly disclaims liability for any loss or injury arising out of the use of information, materials, equipment or practices described. Safe use of any procedure, equipment or material is the responsibility of the user.

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## PREPARATION OF AQUEOUS STANDARDS FOR GROUNDWATER ANALYSIS

This procedure is based on an actual site investigation conducted with E.P.A. approval and is intended to provide a guide for similar investigations. Local site conditions and the type of compounds present will obviously be different and approval of the planned procedure will need local E.P.A. approval.

### PREPARATION OF WATER STANDARDS

This is a general guideline to be used when making water standards of volatile compounds. The example given is for Benzene and TCE, two compounds commonly found in groundwater contamination. The volumes used for generating the initial stock solution and subsequent dilutions are readily dispensable to allow the preparation of low concentration standards.

The accuracy of the standards is dependent on the precautions taken in the transfers of liquids and care taken to prevent headspace loss. Cross contamination by using contaminated syringes must also be avoided.

APPARATUS: The following apparatus is required, with care being taken to decontaminate the various items using only spectroscopic grade solvents:

1. 40 ml. VOA bottles
2. Gas tight syringes (50 ul to 100 ul).
3. Volumetric pipettes to measure 20 ml and 40 ml volumes. (Other glassware of compatible accuracy can be used as alternatives).
4. Analytical balance (and pan balance if available).

### REAGENTS

1. 100ml Methanol
2. Approximately 20 ml each of other desired reagents.
3. Organic-free water.

### PROCEDURE

If analytical balances are available, then the preferred method of making standards is to weigh small quantities of the volatile organic compounds in gas-tight syringes. These quantities are transferred to 20 ml of Methanol and the whole reweighed (See appendix A "Procedure for weighing liquids with a syringe"). Concentration is then calculated directly on a wt/wt basis ( $\text{ppm} = \text{ug/g}$ ).

If analytical balances are not available, the densities of the compounds are used to determine the weight of known volumes. The following is an example for making water standards at low ppb concentrations of Benzene and TCE. The procedure consists of 3 parts: A) Making a stock solution in Methanol (approx. 1000-5000 ppm), B) 1/800 dilution in water, C) 1/500 dilution in water.

#### A) STOCK SOLUTION IN METHANOL

1. Using a 20 ml volumetric pipette, transfer 20 ml of Methanol

to a 40 ml VOA bottle. Cap the bottle immediately.  
The density of Methanol is  $\frac{.7915 \text{ g}}{\text{ml}}$  at 20°C, 20 ml should weigh:

$$\frac{.7915 \text{ g}}{\text{ml}} \times 20 \text{ ml} = 15.83 \text{ g of Methanol}$$

2. Using a gas tight 100  $\mu\text{l}$  syringe, transfer 40  $\mu\text{l}$  of TCE into the 20 ml of Methanol. The density of TCE is  $\frac{1.465 \text{ g}}{\text{ml}}$  at 20°C therefore 40  $\mu\text{l}$  weighs

$$\frac{1.465 \text{ g}}{\text{ml}} = \frac{1465 \text{ mg}}{\text{ml}} = \frac{1465 \text{ mg}}{1000 \mu\text{l}} = \frac{1.465 \text{ mg}}{\mu\text{l}}$$

$$\text{Therefore, } 40 \mu\text{l} \times \frac{1.465 \text{ mg}}{\mu\text{l}} = 58.6 \text{ mg TCE}$$

3. With a clean syringe, transfer 60  $\mu\text{l}$  of Benzene to the TCE/Methanol mixture. The density of Benzene at 20°C is  $\frac{.8794 \text{ g}}{\text{ml}}$  therefore 60  $\mu\text{l}$  =

$$\frac{0.8794 \text{ g}}{\text{ml}} = \frac{0.8794 \text{ mg}}{\mu\text{l}} \times 60 \mu\text{l} = 52.76 \text{ mg Benzene}$$

Concentration of stock solution

$$\text{for Benzene} - \frac{52.76 \text{ mg Benzene}}{52.76 \text{ mg Benzene} + 58.6 \text{ mg TCE} + 15.83 \text{ g Methanol}} =$$

$$\frac{52.76 \text{ mg Benzene}}{15.94 \text{ g total}} = \frac{52,760 \mu\text{g}}{15.94 \text{ g}} = \frac{3310 \mu\text{g}}{1 \text{ g}} = 3310 \text{ ppm}$$

$$\text{for TCE} - \frac{58.60 \text{ mg TCE}}{58.60 \text{ mg TCE} + 52.76 \text{ mg Benzene} + 15.83 \text{ g Methanol}} =$$

$$\frac{58.60 \text{ mg TCE}}{15.94 \text{ g total}} = \frac{3676 \mu\text{g}}{1 \text{ g}} = 3676 \text{ ppm}$$

This mixture is Solution A.

B) 1st Dilution of Stock:

Withdraw 50  $\mu\text{l}$  of stock solution with a 100  $\mu\text{l}$  syringe and transfer into 40 ml of organic-free water within a VOA bottle. Mix by shaking for a few minutes and let stand for 15 minutes.

$$\text{Concentration} = \frac{.050 \text{ ml stock}}{40 \text{ ml water}} = \frac{1}{800} = 4.6 \text{ ppm TCE}$$

and 4.14 ppm Benzene (Solution B)

C) 2nd Dilution:

Using Solution B, withdraw 40  $\mu\text{l}$  and expel into 20 ml water in a 40 ml VOA bottle. Invert the bottle and swirl a few minutes.



## APPENDIX A

### PROCEDURE FOR WEIGHING LIQUIDS WITH A SYRINGE

#### Cautions/Limitations:

1. The minimum amount of liquid to be weighed is 20  $\mu$ l.
2. Use only about 50-60% of the capacity of the syringe, therefore, the minimum size syringe that can be used is 50  $\mu$ l.
3. Perform procedure as quickly as possible to avoid exposure and possible volatilization.
4. For liquids that are very volatile, it is necessary to draw total sample back into syringe barrel, before weighing, to prevent liquid volatilization during weighing procedure.

#### Procedure

1. Use a 50  $\mu$ l syringe and withdraw at least 20  $\mu$ l of liquid.
2. Carefully wipe the needle and syringe dry.
3. Place syringe on analytical balance and weigh. (Tare and sample).
4. Depress plunger to empty syringe into a suitable flask containing solvent. To prevent volatilization of liquid, be sure to empty contents of the syringe under the surface of the solvent.
5. Reweigh syringe (tare), and by difference calculate the weight of the liquid taken.