FID 246 009170 EBB/ERP

October 26, 1994

Ms. Pamela A. Mylotta Hydrogeologist, Environmental Repair Program Wisconsin Department of Natural Resources 4041 North Richards Street Milwaukee, WI 53212

RE: TECUMSEH PRODUCTS COMPANY GRAFTON, WISCONSIN

Dear Pam:

Enclosed are two copies of the workplan for the second phase of the NR 700 investigation of the extent of chlorinated VOCs in groundwater at the above-referenced facility. This workplan contains a more detailed description of the activities that were recommended in the interim status report, along with the modifications that you and Scott Ferguson requested when we met on October 13, 1994.

We would appreciate receiving your approval or comments by November 4, as we plan to begin fieldwork on November 7 and to complete the well installations before Thanksgiving. Please call me if you have any questions.

Sincerely,

de Sheka

Linda E. Hicken, P.E. Senior Project Manager

gig

Enclosure

cc: Kerry DeKeyser, Tecumseh Products



RMT, INC. – MADISON, WI

744 HEARTLAND TRAIL = 53717-1934 P.O. Box 8923 = 53708-8923 608/831-4444 = 608/831-3334 FAX

3084,03 0000:MSE:mylotta

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

> PREPARED FOR TECUMSEH PRODUCTS COMPANY GRAFTON, WISCONSIN

OCTOBER 1994 FID 246009 170 ERR/ERP



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PHASE II WORKPLAN TO EVALUATE THE EXTENT OF CHLORINATED VOCS IN GROUNDWATER AT THE TECUMSEH PRODUCTS COMPANY GRAFTON, WISCONSIN FACILITY

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OCTOBER 1994 FID 246009 170 ERR/ERP

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Staff Hydrogeologist

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Section

TECUMSEH PRODUCTS

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Photovac® Technical Bulletin #27



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Section 1

INTRODUCTION

Background

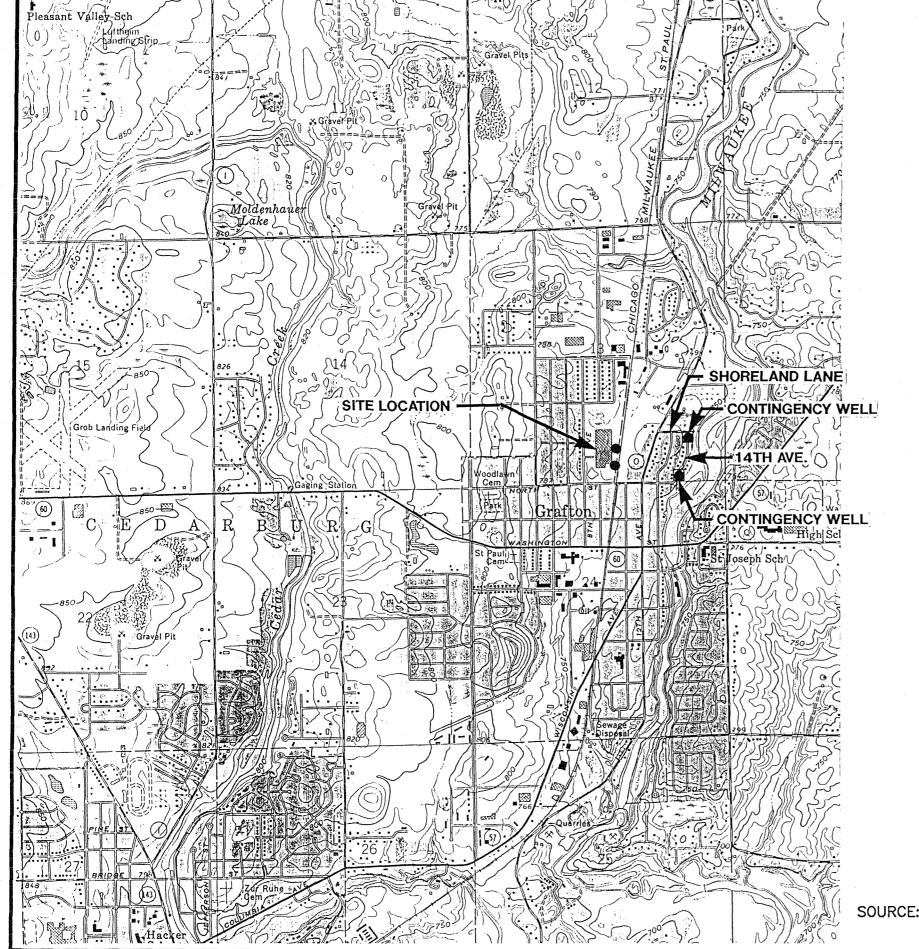
The Tecumseh Products Company operates a manufacturing facility located at 900 North Street in Grafton, Wisconsin (see Figure 1). During the 1960s, 1970s, and 1980s, the Grafton facility machined and assembled two-cycle gasoline engines. Vapor degreasing of parts and engines and painting of assembled engines were part of the manufacturing process. The process equipment and processes associated with the engine assembly operations included underground petroleum storage tanks for engine testing.

During the period of December 1988 through June 1992, eight underground storage tanks (USTs) were removed at the site. These tanks contained gasoline (leaded and unleaded), kerosene, Stoddard solvent, and motor oil. Evidence of releases was noted. During the course of investigating the extent of the release associated with the Stoddard solvent tank, chlorinated volatile organic compounds (VOCs) were detected in the groundwater.

RMT, Inc. (RMT), was hired to conduct additional investigative activities at the Grafton facility to better define hydrogeologic conditions beneath the facility, to evaluate the direction of groundwater migration, and to define the extent of chlorinated solvents identified in groundwater during the previous investigations. The first phase of this investigation was conducted in August 1994 and included the installation of eight soil borings, two of which were converted to water table wells, and three of which were converted to piezometers. The locations of the soil borings and monitoring wells are shown on Figure 2.

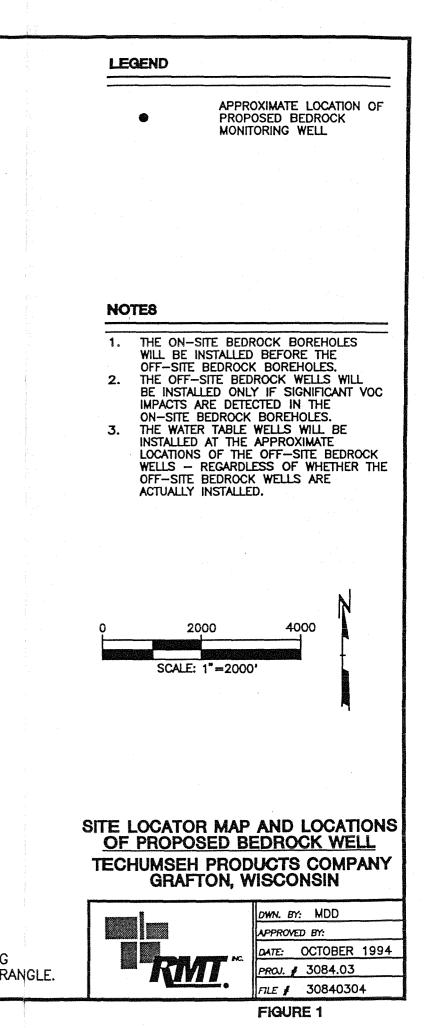
The findings and conclusions of this initial phase of investigation were summarized in an interim status report that was submitted to the WDNR on October 10, 1994 (RMT, 1994). The interim status report also contained recommendations for a second phase of investigation which would include installation of monitoring wells in the bedrock. A meeting among representatives of the WDNR, Tecumseh Products and RMT was held on October 13, 1994, to discuss the contents of the interim status report. The WDNR representatives generally

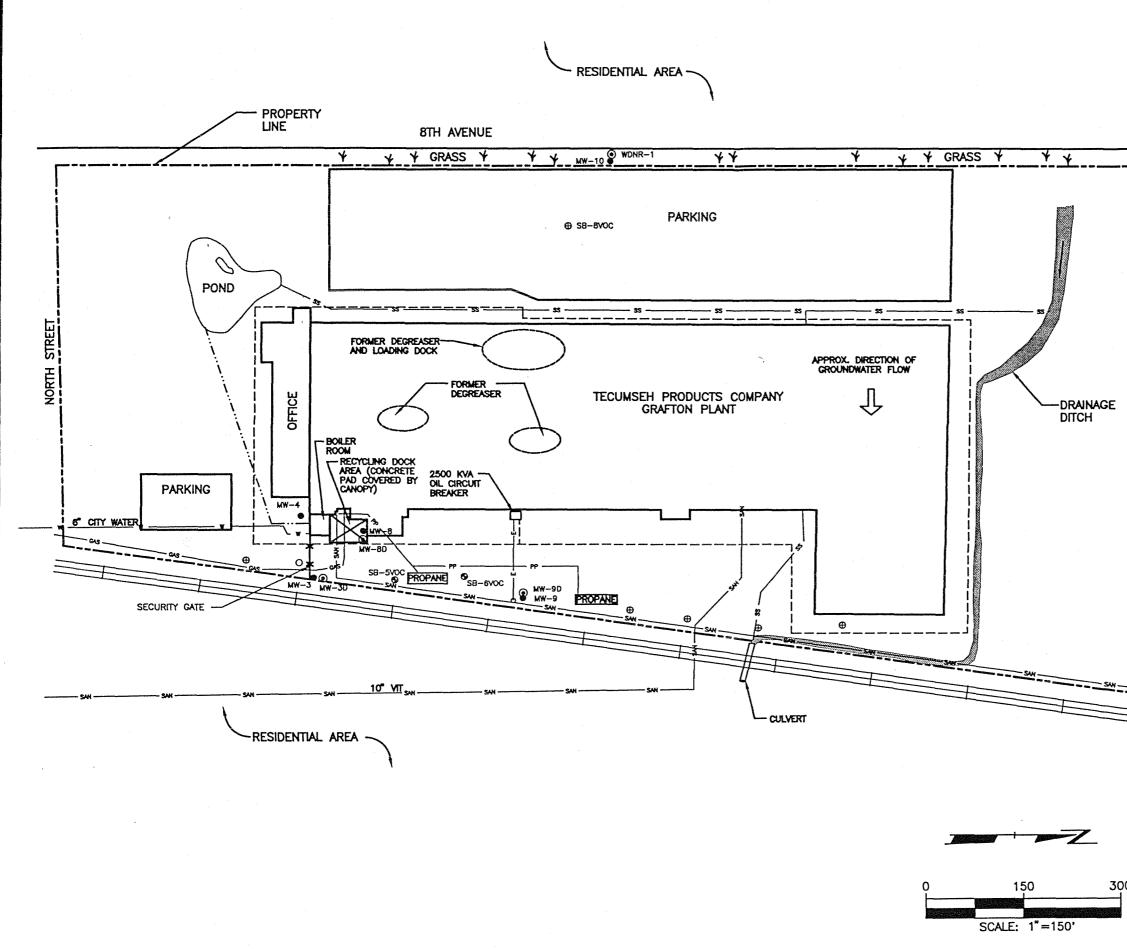




SOURCE: BASE MAP FROM CEDARBURG WI. 7.5 MINUTE USGS QUADRANGLE.

STATE LOCATION





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

	LEGEND				
	● WW-10 WATER TA	BLE WELL			
	● MW-90 PIEZOMETE				
		R (OWNED BY			
	SB-BVOC SOIL BORI	NG			
		SOIL BORING			
•		BEDROCK WELL			
	w WATER				
	GAS				
	PP PROPANE	WED			
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		PROJ. # 3084.03			
		FILE # 30840305			
and Theory of Contraction of Contrac		FIGURE 2			

RMT WORKPLAN TECUMSEH PRODUCTS

concurred with the recommendations outlined in the interim status report, but asked that some modifications be made. The RMT recommendations, with the WDNR's modifications, are presented in this workplan.

Purpose and Scope

The purpose of this workplan is to describe in more detail the field activities and laboratory analyses that will be performed during the second phase of the investigation.

The scope of the investigation includes installing additional shallow soil borings, water table wells, and piezometers in the unconsolidated soils, as well as installing monitoring wells with multiple sampling ports in the bedrock. The drilling activities will be conducted both on-site and in the Village right-of-way on 14th Avenue, which is between the Tecumseh facility and the Milwaukee River. The scope of this second phase of work also includes an assessment of the feasibility of investigating potential source areas under the plant building.

Objectives

The objectives of the second phase of investigation of groundwater at the Tecumseh Products Grafton facility are as follows:

- To determine the northern extent of the trichloroethene (TCE)-dominated plume along the eastern property line.
- To verify that the southern extent of the 1,1,1-trichloroethane (TCA)-dominated plume along the eastern property line does not extend significantly further south than MW-3.
- To assess the horizontal extent of the VOC plume in the shallow aquifer east of the plant.
- To assess whether, and to what extent, there is downward migration of VOCs in the bedrock aquifer beneath the site.
- To assess the horizontal extent of VOCs in the bedrock aquifer and the degree to which the Milwaukee River intercepts the groundwater in the bedrock aquifer that is affected by VOCs if concentrations of VOCs in the bedrock aquifer beneath the site are greater than or equal to an Enforcement Standard (ES) as specified under NR 140 WAC, Groundwater Quality.
- To evaluate the feasibility of investigating potential source areas under the plant building.

The activities that will be conducted to address these objectives are described in Section 2.

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Section 2

SITE INVESTIGATION

2.1 Field Investigative Activities

The approximate locations of the planned soil and bedrock borings and the monitoring wells are shown on Figures 1 and 2. The drilling program is summarized in Table 1. The field methods and the portable GC operating procedures are contained in Appendices A and B, respectively.

Soil Borings and Monitoring Wells

 Install up to five on-site soil borings to the bedrock surface, which is approximately 40 feet below grade. As the borings are advanced, collect soil samples with a 2-foot-long split-spoon sampler at 2.5-foot intervals until the water table is encountered. Collect soil and groundwater samples at alternating 4-foot intervals below the water table until bedrock is reached. Modify the groundwater sampling intervals as necessary to be able to collect the groundwater samples from sandy soils. Describe the soil samples according to the Unified Soil Classification System (USCS). Collect the groundwater samples using a Hydropunch® sampler.

Analyze the headspace concentrations of groundwater samples for the following VOCs using a portable GC: dichloroethenes (1,1-dichloroethene [DCE], cis-1,2-DCE, and trans-1,2-DCE), TCE, TCA, toluene, and total xylenes. Vertically profile the distribution of these constituents throughout the unconsolidated material.

If results of the portable GC screening indicate that the northern edge of the TCE-dominated plume (defined as less than the ES of 5 μ g/L for TCE) is located more than approximately 100 feet north of MW-9, install a water table well and a piezometer near the northern edge of the plume. Set the screen for the piezometer in the interval that corresponds to the highest VOC concentrations. If installed, the boreholes for the water table well and the piezometer will be blind-drilled.

- Install two soil borings to the bedrock surface on the west side of the Milwaukee River within the public right-of-way on the east side of 14th Avenue.
 Place the boreholes just over the curb in a grass-covered area.
- Sample the soil and groundwater in each of the off-site soil borings as describe above for the on-site soil borings. Vertically profile the headspace

TABLE 1									
PROPOSED DRILLING PROGRAM									
Proposed Boring	Type of Boring or Monitoring Well	Estimated Depth (feet)	Primary Objective of Sample Point/Well						
SB-9VOC	Soil boring	40	To verify that the southern extent of the TCA-dominated plume along the eastern property line does not extend significantly further south than MW-3						
SB-10VOC	Soil boring	40	To determine the northern extent of the TCE-dominated plume along the eastern property line						
SB-11VOC ¹	Soil boring	40	To determine the northern extent of the TCE-dominated plume along the eastern property line						
SB-12VOC ¹	Soil boring	40	To determine the northern extent of the TCE-dominated plume along the eastern property line						
SB-13VOC ¹	Soil boring	40	To determine the northern extent of the TCE-dominated plume along the eastern property line						
MW-10 ²	Water table well	16	As necessary, to monitor shallow groundwater quality and water table elevation at the northern edge of the TCE-dominated plume.						
MW-10D ²	Piezometer	40	As necessary, to monitor deeper groundwater quality and piezometer head in the unconsolidated material aquifer at the northern edge of the TCE-dominated plume.						
MW-11	Water table well	20	To assess the horizontal extent of VOCs in groundwater in the shallow aquifer east of the plant						
MW-12	Water table well	20	To assess the horizontal extent of VOCs in groundwater in the shallow aquifer east of the plant						
MW-3BR	Bedrock monitoring well	Up to 160	To assess whether, and to what extent, there is downward migration of VOCs in the bedrock aquifer beneath the site						
MW-10BR ³	Bedrock monitoring well	Up to 160	To assess whether, and to what extent, there is downward migration of VOCs in the bedrock aquifer beneath the site						

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TABLE 1 (Continued)										
PROPOSED DRILLING PROGRAM										
Proposed Boring	Type of Boring or Monitoring Well	Estimated Depth (feet)	Primary Objective of Sample Point/Well							
MW-11BR⁴	Bedrock monitoring well	Up to 140⁵	 As necessary: To assess the horizontal extent of VOCs in the bedrock aquifer To assess the degree to which the Milwaukee River intercepts groundwater in the bedrock aquifer affected by VOCs 							
MW-12BR⁴	Bedrock monitoring well	Up to 140⁵	 As necessary: To assess the horizontal extent of VOCs in the bedrock aquifer To assess the degree to which the Milwaukee River intercepts groundwater in the bedrock aquifer affected by VOCs 							
 NOTES: SB-11VOC, SB-12VOC, and SB-13VOC are contingency soil borings. The northern extent of the TCE-dominated plume will be established first by installing SB-10VOC (and vertically profiling groundwater quality using the portable GC), and then, as necessary, moving northward with SB-11VOC, SB-12VOC, and SB-13VOC until the concentrations of the selected VOCs are nondetectable on the portable GC. MW-10 and MW-10D are contingency wells. They will be installed if the results of the field-screening performed on SB-10VOC indicates that the northern edge of the TCE-dominated plume is located more than 100 feet north of MW-9D. MW-10BR will be installed in approximately the center of the TCE-dominated plume. This will be south of MW-10 and MW-10D if they are installed. MW-11BR and MW-12BR are contingency wells. They will be installed if the on-site bedrock borings/wells (MW-3BR and MW-10BR) indicate significant concentrations of VOCs in the bedrock aquifer beneath the site. Depth contingent upon vertical VOC extent noted in borings MW-3BR and MW-10BR. 										

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concentrations of the same group of VOCs listed above throughout the boreholes using the portable GC. Blind-drill boreholes next to each of these two locations and install water table wells.

- Install the water table wells and piezometers in accordance with Chapter NR 141, Wisconsin Administrative Code.
- Develop the water table wells and shallow piezometers by surging and bailing, in accordance with NR 141 rules.
- Conduct single-well response tests in the water table wells installed on 14th Avenue to further characterize the hydraulic conductivity of geologic materials.

Bedrock Borings and Monitoring Wells

Install two borings into the bedrock on-site along the eastern property line: one in the TCA-dominated area near MW-3D, and one in the center of the TCE-dominated area (to be determined during the installation of the on-site soil borings described above). The on-site bedrock borings will be installed to either the bottom of the VOC plume (defined as concentrations less than ESs specified in NR 140 WAC) or to a maximum depth of 120 feet into bedrock, which is below the bottom of the casing in Village Well 1—whichever comes first (total depth of boring up to 160 feet).

If the on-site bedrock borings/wells indicate concentrations of VOCs in the bedrock aquifer are greater than ESs specified in NR 140 WAC, install two additional bedrock borings/monitoring wells on the west side of the Milwaukee River within public right-of-ways in the residential area, adjacent to the new water table wells (MW-11 and MW-12). The off-site bedrock borings will be installed to either the bottom of the VOC plume or to a maximum depth of 100 feet into bedrock—whichever comes first (total depth of boring up to 140 feet).

All of the bedrock borings will be blind-drilled from the ground surface to the top of bedrock using the mud rotary drilling method. Bedrock is expected to occur at a depth of approximately 40 feet below ground surface. Rock drilling will be performed using the air rotary drilling method.

- In order to avoid potential downward migration of constituents at the <u>on-site</u> bedrock boring locations, install a 6-inch-diameter steel well casing through the unconsolidated material at these locations. Cement the casings into place.
- Install 6-inch-diameter steel surface casings through the unconsolidated material at the <u>off-site</u> bedrock boring locations only if the vertical profiling of the shallow aquifer groundwater quality at these locations indicates the presence of significant VOC concentrations.

RMT WORKPLAN	OCTOBER 1994
TECUMSEH PRODUCTS	FINAL

- Advance each 6-inch air rotary borehole in 20-foot intervals. Perform packer tests of each 20-foot interval to collect information on the presence of fractures, hydraulic effectiveness, and connection to VOC sources. Collect groundwater samples from each 20-foot interval if fractures are encountered. Analyze the groundwater samples for the selected VOCs using a portable GC.
- Perform a downhole video inspection of each borehole to visually determine where the fractures are located.

Unless the boring extends less than 40 feet into bedrock, complete each boring as a multiple-port monitoring well system. Construct each well using 3inch-ID Schedule 80 PVC casing that will have up to three, 5-foot-long screened intervals. Each screened interval will be separated by at least 20 feet of 3-inch Schedule 80 PVC riser. The locations of the screened intervals will be determined on the basis of the results of the packer testing, portable GC analysis, and video logging.

- Install a multiple-port monitoring system in the 3-inch PVC casing in each bedrock well. The system will be comprised of PVC sampling/pressure ports and permanent, positive-seal packers built into a PVC casing string. Dedicated pumps and pressure transducers will be placed within each sample port for measuring hydraulic head.
- If the bedrock boring is less than 40 feet into bedrock, complete the boring as a piezometer in accordance with NR 141.

Soil and Bedrock Borings and Wells

- Survey the location and elevation of the new soil borings and monitoring wells relative to a site datum and to mean sea level, respectively.
- Collect a complete round of groundwater level measurements from the previously installed and new monitoring wells. If free product is found in MW-2, remove it using a bailer.
- Collect one round of groundwater samples from the new monitoring wells and from existing wells MW-3D, MW-8D, and MW-9D. Analyze the samples for VOCs using USEPA Method 8021. Also analyze a trip blank for quality control purposes.
- Place the drill cuttings in properly labeled 55-gallon drums, and store the drums at an on-site location. Collect a composite sample of the drill cuttings for disposal characterization.
- Collect the decontamination, purge, and development waters in a 2,000-gallon tanker. Based on the results of the portable GC analysis, periodically discharge the collected decontamination water to the sanitary sewer with the permission of the Village of Grafton.

2.2 Evaluation of Potential Source Areas Under the Plant Building

An assessment of the feasibility of drilling soil borings at potential source areas inside the plant (i.e., the former degreasers and loading dock on the west side of the plant [see Figure 2]) will be made by conducting a site visit and interviews with appropriate plant personnel in order to develop a map of the facility that will show the locations of the potential source areas, current facility operations, underground utilities, and the aisle clearance necessary to avoid disruption of plant operations.

2.3 Report Preparation

Upon completion of the fieldwork and laboratory analyses described above, RMT will prepare a second interim status report for review by the WDNR. A meeting will be scheduled with the WDNR and Tecumseh Products to discuss the findings and conclusions of the investigative activities to date and to determine whether sufficient information has been collected to support the evaluation of remedial options. If the degree and extent of VOC impacts have been adequately characterized upon completion of the second phase of investigation, a report will be prepared and submitted to the WDNR documenting both phases of study.

RMT WORKPLAN TECUMSEH PRODUCTS

Section 3

SCHEDULE

The schedule for completing the second phase of the project is shown on Figure 3.

Project Schedule Tecumseh Products - Grafton Operations Phase II Investigation

	Oct	tob	er	I	Nov	emt	ber	T	De	ecen	nbei	•		Jan	uar	/	1	Febi	ruar	у
Activity	17	2	4 3	31	7	14	21	28	5	12	19	26	2	9	16	23	30	6	13	20
Prepare and submit the workplan to the WDNR																				
WDNR's review and approval of the workplan			₿																	
Install borings and monitoring wells							1													
Collect groundwater samples								8-1												
Laboratory analysis and data validation								┣												
Evaluate the data and prepare a second interim status report for submittal to the WDNR																	th	e WĽ	g with NR i ebrua	'n

Section 4

REFERENCES CITED

RMT, 1994a. Interim status report on the subsurface investigative activities at the Tecumseh Products Company Grafton Operation. October 1994.

RMT WORKPLAN TECUMSEH PRODUCTS

APPENDIX A

FIELD METHODS

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 of 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 long. 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. The split-spoon will be decontaminated before each sample is collected. (See Subsection 4.2 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.

2. Plugging and Sealing of Drill Holes

Soil borings that are not converted to groundwater monitoring wells will be abandoned by backfilling the borehole to the ground surface with bentonite chips. 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.

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3. Groundwater Sampling using a Hydropunch® Sampler

Groundwater samples will be collected at 4-foot intervals in the unconsolidated material 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 analysis. 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 portable GC laboratory for analysis.

The Hydropunch II will be decontaminated between each sample using the procedures outlined in Subsection 4.2.

4. Decontamination Methods

4.1 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 a 2,000-gallon tanker and, based on the results of the portable GC analysis, will be discharged periodically to the sanitary sewer, as verbally approved by the Village of Grafton.

4.2 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.

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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.

5. Investigation-Derived Wastes

Soil and rock cuttings generated during the performance of the soil and bedrock borings will be placed in WDOT-approved, 55-gallon drums, and will be labeled and stored on-site. A composite soil sample will be collected and analyzed for the parameters needed to determine the disposal requirements for the investigation-derived waste.

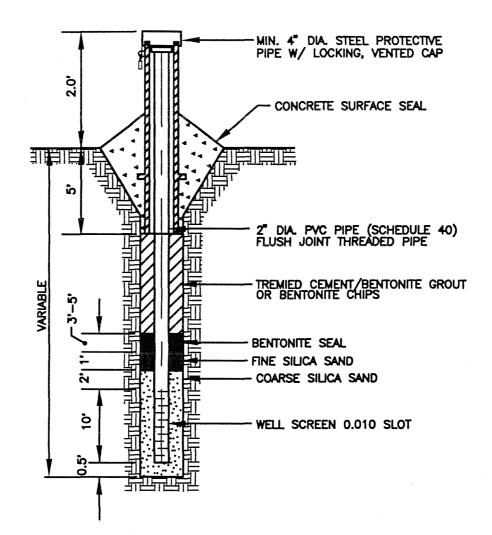
Purge and development water will be placed in a 2,000-gallon tanker and, based on results of the portable GC analysis, will be discharged periodically to the sanitary sewer, per the verbal approval of the Village of Grafton.

6. Portable Gas Chromatograph Analyses

Groundwater samples will be field-screened for selected VOCs using a Photovac® Model 10S50 GC portable gas chromatograph (GC). The portable GC analysis will be used to characterize the presence and relative concentration of selected VOCs in the headspace on a near real-time basis. The headspace will be analyzed for dichloroethenes (1,1-DCE, cis-1,2-DCE, and trans-1,2-DCE), trichloroethene (TCE), 1,1,1-trichloroethane, toluene, and xylenes. Standard operating procedures for the portable GC are included in Appendix B.

7. Shallow Monitoring Well Installation and Development

The water table wells and piezometers in the unconsolidated material will be constructed using 2-inch-diameter Schedule 40 PVC with flush-threaded joints. The water table wells will have 10-foot-long screens and will be constructed as shown on Figures A-1 or A-2, depending on the surface finish. The piezometer wells will have 5-foot-long screens and will be constructed as shown on Figures A-3 or A-4, depending on the surface finish. All of the wells located in Village rights-of-way will be completed with locking flush-mount surface covers. The on-site

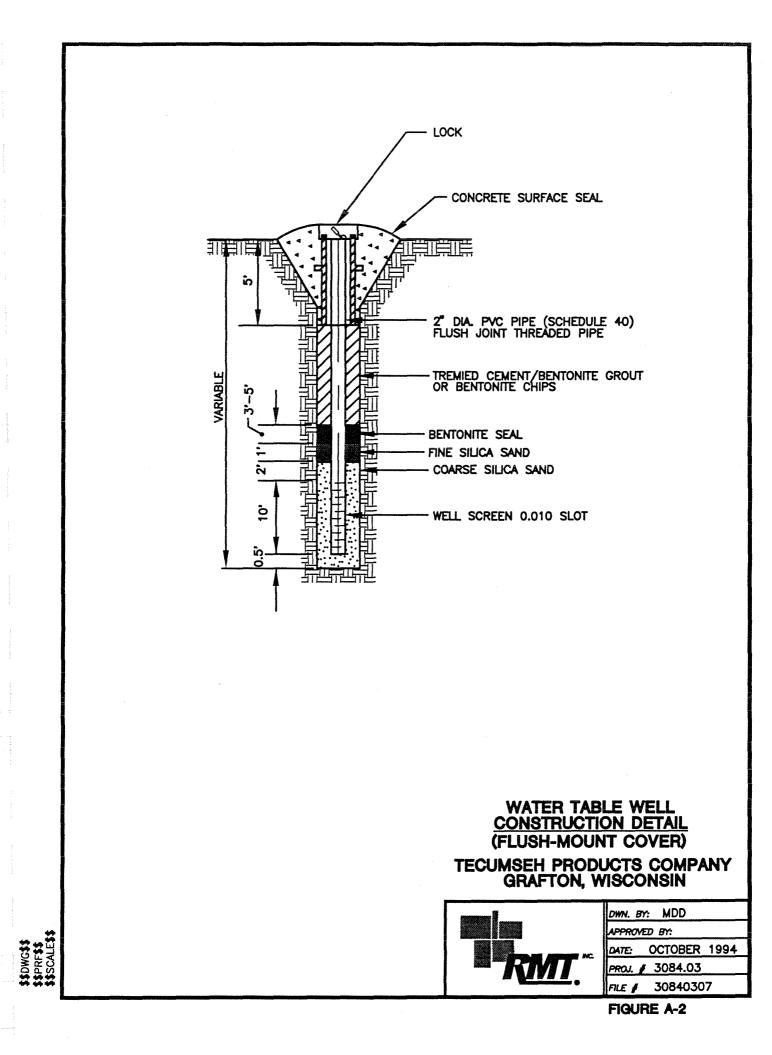


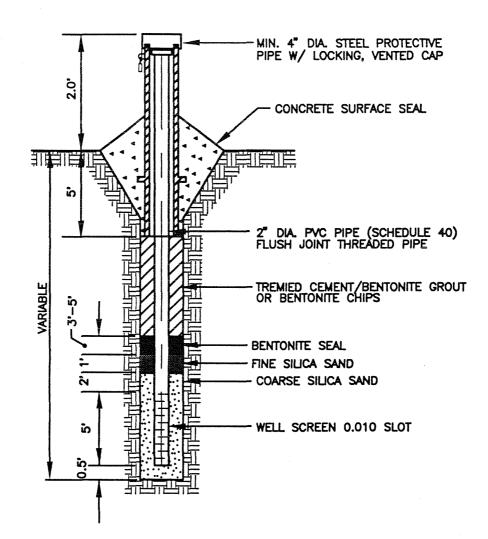
tspwcst tsprfst tscalets

WATER TABLE WELL <u>CONSTRUCTION DETAIL</u> (STICK-UP SURFACE CASING) TECUMSEH PRODUCTS COMPANY GRAFTON, WISCONSIN

	DWN. BY: MDD
	APPROVED BY:
	DATE: OCTOBER 1994
	PROJ. # 3084.03
•	FILE # 30840306

FIGURE A-1





\$\$PRF\$\$ \$\$PRF\$\$ \$\$SCALE\$\$

PIEZOMETER CONSTRUCTION DETAIL <u>(STICK-UP SURFACE CASING)</u> TECUMSEH PRODUCTS COMPANY GRAFTON, WISCONSIN



DWN. BY: MDD						
APPROVED BY:						
DATE: OCTOBER 1994						
PROJ. # 3084.03						
FILE # 30840308						

FIGURE A-3

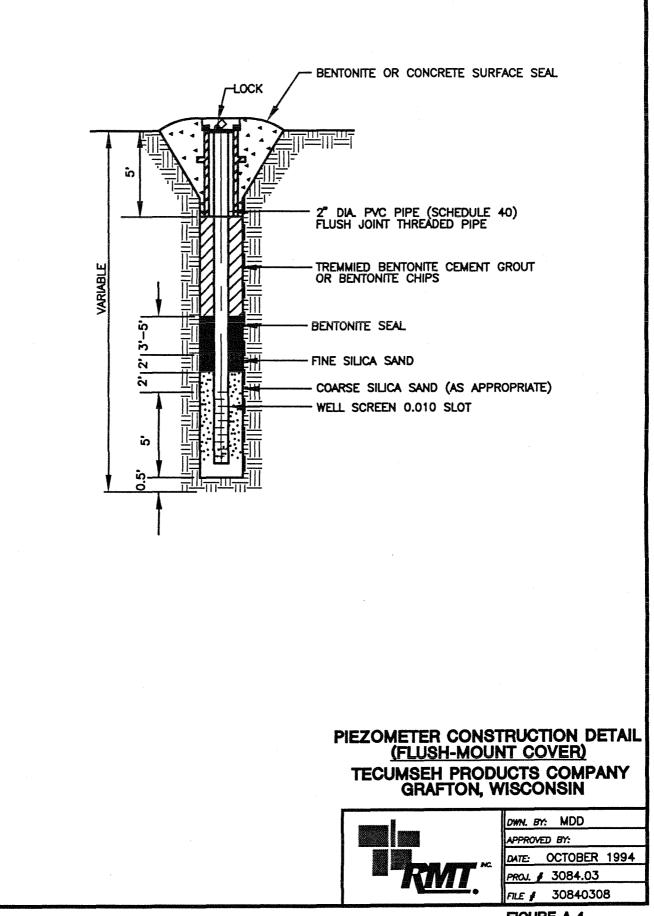


FIGURE A-4

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wells will have a locking protective steel pipe over the stick-up casing unless traffic patterns in the area create too much potential for damage to the surface casing. As-built well construction diagrams will be prepared on WDNR Form 4400-113A.

The water table wells will be installed in soil borings that will extend to approximately 7 feet below the water table. The piezometer will be completed at the northern edge of the TCE plume, as determined by the portable GC results. The piezometer 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 chip seal will be placed over the fine sand. The remaining annular space above the filter pack seal will be filled with bentonite chips or a bentonite slurry grout. A lockable protective steel pipe or surface cover 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 30 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.

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8. Bedrock Monitoring Well Installation

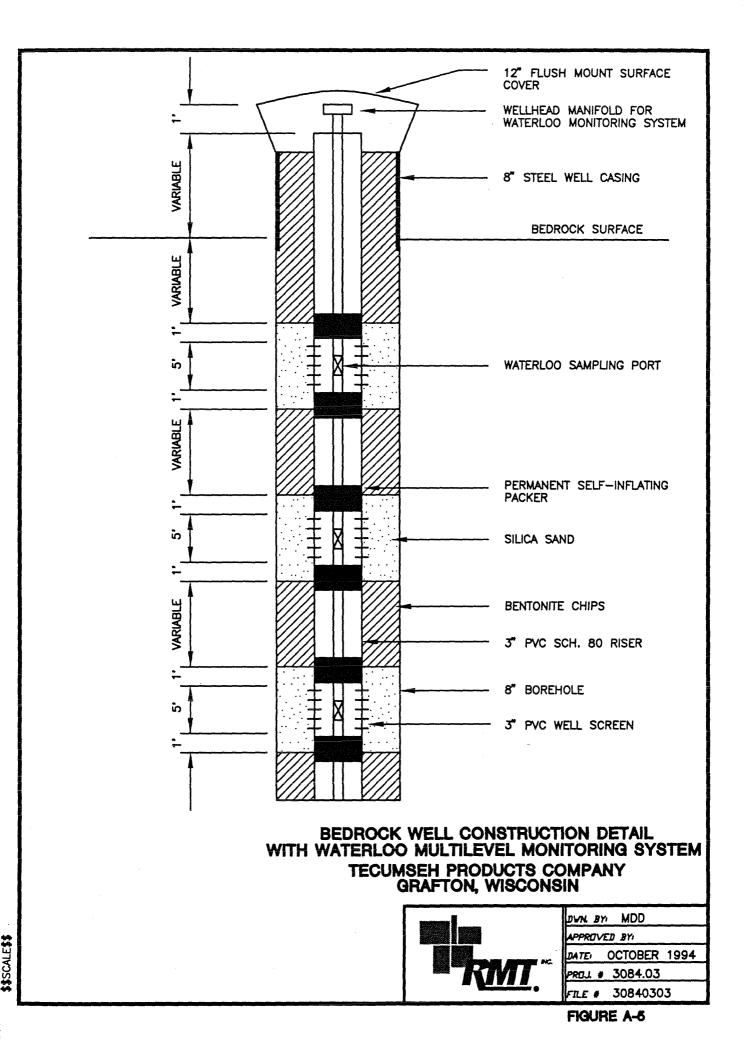
The boreholes for bedrock well installation will be advanced through the unconsolidated material using 12-inch mud rotary. The boreholes will be drilled without sampling shallow soil or groundwater. After bedrock is reached, 8-inch steel well casing will be installed in the boreholes and will be grouted in place using neat cement. The boreholes will be advanced through the bedrock using 8-inch air rotary once the cement around the 8-inch casing has set. The on-site bedrock wells will be advanced to the bottom of the VOC plume or to a maximum depth of 160 feet below ground surface, whichever comes first. The off-site bedrock wells will be advanced to the bottom of 140 feet below ground surface, whichever comes sill be collected from the airstream as they are brought to ground surface to allow classification of subsurface material.

Drilling into the bedrock will be completed in 20-foot intervals to allow groundwater samples to be collected using a single-packer system and a Grundfos 2-inch sampling pump. At each 20-foot interval, the pump will be lowered downhole and the packer will be inflated above the pump. Each interval will be pumped for approximately 15 minutes (if the interval yields water) before a sample is collected. Pumping rate will be recorded with clock and bucket methods and hydraulic head declines will be noted with a pressure transducer. The sample will be collected under low pumping flow conditions and transferred to a glass vial with a Teflon® septum seal. The vial will be taken to the portable GC laboratory area for immediate analysis.

After the boreholes are advanced to completion depth, a downhole video inspection will be performed on each borehole to determine where the fractures are located. A maximum of three monitoring zones will be selected based on the fracture locations identified with the video inspection, the pumping rate and head observations, and on the results of the in-field GC analysis.

The bedrock wells will be constructed using 3-inch ID Schedule 80 PVC casing that will have up to three 5-foot-long screened intervals. Each screened interval will be separated by at least 20 feet of 3-inch Schedule 80 PVC riser. The locations of the screened intervals will be determined based on the results of the packer testing, portable GC analysis, and video logging. The wells will be constructed as shown on Figure A-5. A No. 30 sand pack will be

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installed in the annular space between each screen and the borehole, extending from approximately one foot below the bottom of the screen to one foot above the top of the screen. A one-foot-thick fine sand seal will be installed above the filter sand pack in each screened interval. The screened intervals will be separated by bentonite chips. The bentonite chips will be placed from the top of the fine sand seal in one screened interval to approximately 1-foot below the well screen in the next screened interval. The bentonite chips will be added through a tremie pipe to minimize the problem of bridging or void formation. The volume of the borehole will be determined prior to installing the chips to estimate the volume of chips needed for the annular space seal. The chips will be placed in one continuous operation in order to not disturb the integrity of the filter pack. As the chips are added to the borehole, the depth to the top of the chips will be continuously measured to determine when a sufficient seal has been placed. The estimated and actual volume of sealing material will be calculated and reported to the WDNR.

A Waterloo Multilevel Groundwater Monitoring System, manufactured by Solinst Canada Ltd., of Ontario, Canada, will be installed inside the 3-inch PVC casing. The Multilevel Monitoring System is comprised of PVC sample/pressure ports, permanent positive-seal packers, various casing lengths, a base plug, and a surface manifold. Permanent self-inflating packers will be installed at the top and bottom of each monitoring port to provide a continuous seal within the borehole. The packers and sampling ports will effectively isolate each monitoring interval and allow the monitoring of discrete zones within the bedrock. Each monitoring port will be fitted with a dedicated sampling pump and pressure transducer for collecting water levels, water samples, and hydraulic conductivity measurements.

The modular components of the Multilevel Groundwater Monitoring System will be threaded together in the appropriate order and lowered down the borehole one section at a time. Casing clamps will be used for support and water will be added to the casing string to overcome buoyancy, as required. After installation of the Waterloo system, water will be added to the casing string, causing packer expansion. It will take approximately 24 to 48 hours for the packers to effectively seal the borehole.

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The bedrock wells will be completed with permanent 12-inch flush mount surface covers. The PVC riser will be cut off approximately 1-foot below ground surface to allow room for the multi-level wellhead manifold to be installed within the flush mount surface cover.

The Waterloo Monitoring System will only be used if the VOC plume extends more than 40 feet into bedrock. If results of the in-field GC analysis indicate that the bottom of the VOC contamination is less than 40 feet below the bedrock surface, the bedrock wells will be constructed using standard 2-inch- diameter Schedule 40 PVC with flush-threaded joints. The wells will have 5-foot long screens and will be constructed as shown on Figure A-5.

9. Groundwater Sampling and Decontamination Procedures

Static water levels will be measured in the monitoring wells prior to purging or sampling. Groundwater level measurements in the monitoring wells installed in the unconsolidated material 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 with distilled water. Groundwater level measurements in the bedrock monitoring well will be made using a dedicated in-line pressure transducer. The transducer will be collected to a GK-403 vibrating wire readout unit for automatic water level readings.

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. The monitoring wells in the unconsolidated material will be purged using a precleaned PVC bailer. The bailer will be dedicated for use in only one well.

Groundwater samples in the bedrock monitoring wells will be collected using a dedicated inline double-valve pump system. The drive pressure for the pump will be provided by a nitrogen tank and an automatic pump control unit. Using the vent cycle of pumping, water flows from the formation into the pump and associated tubing. Water is displaced to the surface from the pump and associated tubing during the pressure cycle of pumping. The pump is cycled from vent to pressure as needed until the appropriate purge volume is obtained. All sampling ports within a well will be purged simultaneously.

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 samples from the monitoring wells in the unconsolidated material 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.
- Collect samples from the bedrock monitoring wells directly from the dedicated dual-line tubing.
- 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 measured 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.

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- 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.

10. 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.

11. 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.

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- 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.

4

APPENDIX B

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APPENDIX B

RMT STANDARD OPERATING PROCEDURE FOR PORTABLE GC ANALYSIS OF GROUNDWATER SAMPLES

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APPENDIX B

RMT STANDARD OPERATING PROCEDURE FOR PORTABLE GC ANALYSIS OF GROUNDWATER SAMPLES

1. Parameters To Be Analyzed

The parameters to be analyzed by the portable GC for this investigation are the dichloroethenes (1,1-DCE, cis-1,2-DCE, and trans-1,2-DCE), trichloroethene, 1,1,1-trichloroethane, toluene, and 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 the field samples.

2 & 3. Range of Measurement and Limits of Detection

The anticipated limits of detection are presented in Table B-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 μ L/L (parts per million by volume). The μ L/L (or ppm) units will be used to avoid confusion between concentration units in water and air.

4. Sample Matrix

The concentrations of VOCs in water will 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 = (concentration in air)(concentration in water), where H is a constant

Standards will be prepared using the procedures suggested in the Photovac® Technical Bulletin #27 (Attachment 1). The standards will be analyzed at the same time as the samples, and each parameter's operational partitioning coefficient will be 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.

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TABLE B-1		
DETECTION LIMITS FOR SELECTED CHLORINATED VOCs AND XYLENES ANALYZED BY PORTABLE GC		
Parameter	μL/L	
1,1-Dichloroethene	0.05	
cis-1,2-Dichloroethene	0.05	
trans-1,2-Dichloroethene	0.05	
Trichloroethene	0.1	
1,1,1-Trichloroethane	0.3	
Toluene	0.03	
m,p-xylenes	0.03	
o-xylene	0.04	

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

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

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

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

5. Principle, Scope, and Application

Headspace analysis is a convenient means of screening for VOC contamination. In principle, VOCs contained in water will partition between the water and the air in contact with the water, with partitioning dependent on the VOC concentration in the water as well as on several other factors (Devitt, et al., 1987). Analysis of the headspace gas can be done in the field using a portable gas chromatograph, and can provide near real-time screening analysis of VOCs. Due to the variability of factors that influence VOC partitioning between the water 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 trichloroethlene and, if gasoline is present, it is difficult to quantify di- and trichloroethlene. 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.

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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 suspected. 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 suspected, 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. Ambient air contamination has not typically been found to be a problem.

7. Safety Precautions

Samples will be contained in 40-mL VOA vials, and the analyst will not come in direct contact with the sample. If samples need to transferred from one vial to another, or if a vial breaks, the analyst will follow the standard procedures used when working with chemicals. All pure solvents used for making standards will also be kept in 40-mL VOA vials, and the analyst will not come into contact with the solvents.

Standard laboratory practices will be taken when working with the gas cylinders used for gas supply to the GC. The cylinders will be 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 samples will be collected in 40-mL VOA vials with a septum top. The vials will be filled approximately half full. The vials will be allowed to equilibrate at room temperature for at least 30 minutes. No preservatives will be used in the samples. After the equilibration time, an aliquot of the headspace will be removed with a syringe through the septum, and injected into the GC for analysis. Injections will be repeated until the peaks of interest are on scale. The sample will be disposed of with other potentially affected waters.

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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 B-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.

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

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

11. Reagents and Calibration Standards

Pure solvents and calibration standards will be purchased from chemical supply companies. Commercially prepared gas standards will be purged into the gas sample bottles from pressurized gas cylinders. An example of standard concentrations is shown in Table B-3. Water standards will be prepared by appropriate dilution of pure solvent. Water standards will be prepared in 40-mL VOA vials using methanol and water as dilutents. The final working solution will contain 20 mL of liquid in the 40-mL VOA vial. The constituents in water will be 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 need to be in the gas phase prior to injection.

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CHECKOFF

TABLE B-2

PORTABLE GC FIELD EQUIPMENT CHECKLIST

EQUIPMENT

NEEDED

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

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TABLE B-2 (CONTINUED)

PORTABLE GC FIELD EQUIPMENT CHECKLIST

EQUIPMENT

NEEDED C

CHECKOFF

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

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TABLE B-3 EXAMPLE OF A COMMERCIALLY PREPARED STANDARD		
1,1-Dichloroethene	19.5	
cis-1,2-Dichloroethene	20.1	
trans-1,2-Dichloroethene	9.9	
Trichloroethene	15.4	
1,1,1-Trichloroethane	150.0	
Toluene	19.9	
m- and p-xylenes	40.8	
o-xylene	20.8	

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

12. Calibration Procedures

- A. Standards will be prepared according to the protocol given above.
- 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, and should check to ensure that the GC is correctly identifying peaks, and check retention times against standards if questions arise.

13. Sample Preparation

Sample preparation was 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.

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- 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 unsure 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 $(\mu L/L)/V$ -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.

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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 the technical coordinator, 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 (a < 10 percent difference is a suggested guideline), reinject the 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 <u>concentration</u> only shortly after the standard is prepared. The standard can still be used for <u>peak</u> 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 μ 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.

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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 μ L/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.,

 $(actual conc., \mu L/L) = (instrument reading, V-sec) (response factor <math>\frac{\mu L/L}{V-sec}$)

(injection volume of standard) (gain setting for standard) (gain setting for sample)

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

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

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

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

Gas measurements are commonly reported in ppm or μ g/m³. A gas phase unit of ppm is a μ L/L. To avoid confusion between measurements made on different matrices (air, water, or soil) units of μ L/L are used in the calculations.

17. Data Deliverables

Portable GC result 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 is completed, the chromatograms will be copied and placed in the appropriate section. Replicates will be recorded separately, and compared in a separate section of the notebook. The calculation and results summary sheets will be checked by the QC reviewer. The notebook will be comb bound and placed in the project file. The calculation and results summary sheets can be copied and stored at the GC operator's desk for later reference, if appropriate.

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

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 was 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."

TECUMSEH PRODUCTS

- 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 μ 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 copied too.
 - 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. <u>References</u>

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

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ATTACHMENT 1

PHOTOVAC® TECHNICAL BULLETIN #27

PHOTOVAC Technical Bulletin

27

PREPARATION OF AQUEOUS STANDARDS FOR

GROUNDWATER ANALYSIS

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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 abviously be different and approval of the planned procedure will need local E.P.A. approval.

PREPARATION OF WATER STANDARDS

Thisiis a ceneral duideline to be used when making water standards of volatile compounds. The example given is for Benzene and TCE, two compounds commonly found in aroundwater 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 cransfers 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).
- Volumetric pipettes to measure 20 ml and 40 ml volumes. (Other 3.
- classware of compatible accuracy can be used as alternatives). Analytical balance (and pan balance if available). 4.

REAGENTS

- 1. 100ml Methanol
 - 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 (ppm = uq/q).

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. Theprocedure consists of 3 parts: A) Making a stock solution in Methanol (approx. 1000-5000 ppm), E) 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 <u>.7915 o</u> at 20°C, 20 ml should weigh: mī x 20 ml = 15.83 g of Methanol <u>.7915_0</u> m1 2. Using a gas tight 100 µl syringe, transfer 40 µl of TCE 2. Using a gas tight not project, and the density of TCE is <u>1.465 a</u> ml at 20°C therefore 40 µl weighs $\frac{1.465}{m1} = \frac{1465}{m1} = \frac{1465}{m1} = \frac{1465}{1000} = \frac{1.465}{\mu1} = \frac{1.465}{\mu1}$ Therefore, 40 μ l × $\frac{1.465}{\mu l}$ = 58.6 mg TCE 3. With a clean syringe, transfer 60 µl of Benzene to the TCE/ Methanol mixture. The density of Eenzene at 20°C is .8794 g therefore 60 μ l = $\frac{0.8794}{m1} = \frac{0.8794}{\mu1}$ x 60 µl = 52.76 mg Benzene Concentration of stock solution for Benzene - 52.76 mg Benzene = 58.6 mg TCE + 15.83 g Methanci = <u>52.76 mg Benzene</u> = 15.94 g total = $\frac{52,760}{15.94} \mu q = \frac{3310}{1} \mu q = 3310 \text{ ppm}$ 58.60 mg TCE + 52.76 mg Benzene + 15.83 g Methanci for TCE - - $\frac{58.60}{15.94} = \frac{100}{100} = \frac{3676}{100} = \frac{3676}{100} = \frac{100}{100}$ = 3676 ppm This mixture is Solution A. B) 1st Dilution of Stock: Withdraw 50 µl of stock solution with a 100 µl 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. Concentration = <u>.050 ml stock</u> = 40 ml water = 4.6ppm TCE 1 800 and 4.14 ppm Benzene (Solution B) C) 2nd Dilution: Using Solution B, withdraw 40 µl and expel into 20 ml water in

a 40 ml vOA bottle. Invert the bottle and swirl a few minutes.

Let the solution sit 30 minutes to equilibrate before using.

<u>40 µl Solution B</u> 20 ml water Concentration :

dilution = 9.2 ppb TCE + 8.3 ppb Benzene (Solution) $\frac{-40}{20} \frac{\text{m}}{\text{m}}$ $= \frac{1}{500}$

Solution C is the working Standard.

NOTE: Follow chemical manufacturer's recommended safety information for the reagents used.

Precautionary Notes

- 1. Care should be taken not to inject liquid into the G. C.
- 2. When using a 10550 or 10570 G. C. use a separate library for water standard data. By listing the headspace concentration as the liquid concentration, from the standards, the G. C. will then print results based on liquid concentration for field samples. This approach will prevent confusion with air calibration data and negates the need for using Henry's Law to calculate vapor concentrations with respect to liquid samples.
- 3. When working with samples in the parts-per-billion concentration range, freshly prepared aqueous standards should be used on a daily basis. The standards should be stored with the septum screw capped VOA vial inverted.
- 4. Decending upon the volume of headspace used for injection into the G. C., using a clean gas-tight syringe, transfer the same volume of UZ air to replenish the headspace. Allow the standard to equilibrate for approximately 1/2 hour.

5. Typically, injection volume of headspace rance from 100-500 µl.

APPENDIX A

PROCEDURE FOR WEIGHING LIQUIDS WITH A SYRINGE

Cautions/Limitations:

- 1. The minimum amount of liquid to be weighed is 20 µl.
- Use only about 50-60% of the capacity of the syringe, therefore, the minimum size syringe that can be used is 50 µl.
- 3. Perform procedure as <u>auickly</u> as possible to avoid exposure and possible volatilization.
- For liquids that are very volatile, it is neccessary to draw total sample back into syringe barrel, before weighing, to prevent liquid volatilization during weighing procedure.

Procedure

- 1. Use a 50 µl syringe and withdraw at least 20 µl of liquid.
- 2. Carefully wipe the needle and syrince 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.