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Quality Assurance Project Plan 13160

Remedial Investigation And Feasibility Study Pagel's Pit Landfill Winnebago County, Illinois

Prepared for:

Respondent's Steering Committee

Prepared by:

Warzyn Engineering Inc.
Madison, Wisconsin

December 1987

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Remedial Investigation And Feasibility Study Pagel's Pit Landfill Winnebago County, Illinois

December 1987

QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR

PAGEL'S PIT LANDFILL
(Winnebago Reclamation Landfill)
Remedial Investigation/Feasibility Study

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1.0 INTRODUCTION

The United States Environmental Protection Agency requires that PRP-lead investigations under CERCLA have an approved Quality Assurance Project Plan (QAPP) covering environmental measurements. It is the responsibility of the Respondents or their representatives to implement minimum procedures to assure that the accuracy, precision, completeness and representativeness of data collected are known and documented.

This QAPP presents the organization, objectives, functional activities and specific quality assurance (QA) and quality control (QC) activities associated with the Remedial Investigation/Feasibility Study (RI/FS) at the Pagel's Pit Landfill (also known as Winnebago Reclamation Landfill) located near Rockford, Illinois. The QAPP is designed to achieve the specific data quality goals of the RI/FS.

The QAPP has been prepared using the following guidance documents:

- U.S. EPA, Region V, December 1985, Preparation of Federal-Lead Remedial Investigation Quality Assurance Project Plans for Region V.
- U.S. EPA, December 1980, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, QAMS-500/80.
- U.S. EPA, June 1986, Data Quality Objectives for the RI/FS Process, Doc. No. 9355.0-7A.

2.0 TABLE OF CONTENTS

SECTION		PAGE	REVISIONS	DATE
1.0	TITLE PAGE	1	0	9/26/86
	INTRODUCTION		1	8/12/87
2.0	TABLE OF CONTENTS	2	2	12/30/87
3.0	PROJECT DESCRIPTION	5	2	12/30/87
4.0	PROJECT ORGANIZATION AND RESPONSIBLITY	19	2	12/30/87
5.0	QUALITY ASSURANCE OBJECTIVES	21	2	12/30/87
6.0	SAMPLING PROCEDURES	24	1	8/12/87
7.0	SAMPLE CUSTODY	24	1	8/12/87
8.0	CALIBRATION PROCEDURES, FREQUENCY AND PREVENTATIVE MAINTENANCE FOR FIELD INSTRUMENTS	25	1	8/12/87
9.0	ANALYTICAL SERVICES	26	1	8/12/87
	9.1 Hazleton		1	8/12/87
	9.2 Warzyn		1	8/12/87
	9.3 Radian		0	8/12/87
10.0	QUALITY ASSURANCE REPORTS	30	1	8/12/87

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LIST OF TABLES

TABLE NO.	TITLE	PAGES	REVISION	DATE
1	Maximum Concentration of Volatiles and Semi-Volatile organics in Groundwater during the 1984 E.C. Jordan study of the ACME - Pagel's Pit Landfill area	1	0	9/26/86
2	Maximum Concentrations of Volatile Organics observed in groundwater monitoring wells during the 1984 - 1985 Warzyn study of the ACME - Pagel's Pit Landfill area	1	0	9/26/86
3	Sample types and estimated sample numbers, Pagel's Pit Landfill RI/FS	2	2	12/30/87
4	Sample quantities, bottles, preservations and packaging for water, sediment and leachate samples from Pagel's Pit Landfill	1	1	8/12/87

LIST OF FIGURES

FIGURE NO.	TITLE	PAGES	REVISION	DATE
C 12660-B1	Location map for the Pagel's Pit - ACME Solvents area	1	0	9/26/87
Attachment 1A, 1B	Project schedule for Pagel's Pit Landfill RI/FS	2	1	8/12/87
C 12660-A1	Project Organization	1	1	8/12/87
C 12660-B4	Site Features Map	1	0	9/26/87

LIST OF APPENDICES

APPENDIX	TITLE	PAGES	REVISION	DATE
A	EPA Hazardous Substance List and Requested Detection Limits for Analyses Based on Contract Laboratory Program Protocols	7	1	8/12/87
B	Sampling Plan	23	1	8/12/87
C-1	Field Measurement of pH		0	9/26/86
C-2	Instruction Manual Orion Model 211 pH Meter		0	9/26/86

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LIST OF APPENDICES
(Continued)

APPENDIX	TITLE	REVISION	DATE
C-3	Field measurement of Specific Conductance and Temperature	0	9/26/86
C-4	Operating Instructions YSI Model 33 Conductivity Meter	1	8/12/87
C-5	Calibration and Maintenance of Photovac TIP	0	9/26/86
C-6	Calibration and Maintenance of HNu Photoionizer	0	9/26/86
C-7	Calibration and Maintenance of Organic Vapor Analyzer (OVA)	0	9/26/86
	Extracted from Manufacturers Instruction Manual		
C-8	Calibration and Maintenance of GPK Gas Meter	0	9/26/86
	Extracted from Manufacturers Instruction Manual		
C-9	Air Monitoring Sampling Equipment and Analysis Methods	0	8/12/87
	Extracted from Manufacturers Instruction Manual		
D	Standard Operating Procedures for Analysis of Water, Leachate and Sediment using non-CLP Protocols	2	12/30/87
E	Field Measure of Permeability using the Bail-down Test	0	9/26/87
F	Example Sample Documentation Forms	0	8/12/87
G	Document Control and the Evidentiary File System for Pagel's Pit Landfill RI/FS	0	8/12/87
H	Acknowledgment of Ability to Perform Analyses by Performing Laboratories	1	12/30/87
I	Internal Chain of Custody Procedures for Hazleton Laboratories America	0	8/12/87

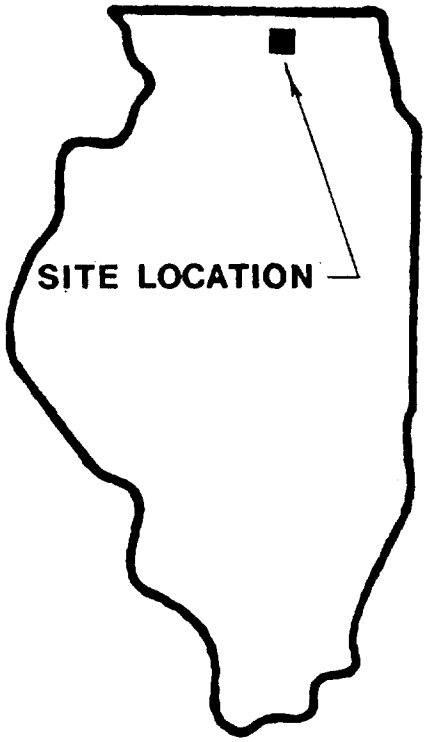
3.0 PROJECT DESCRIPTION

The RI portion of the study is designed to gather specific information needed to determine if Pagel's Pit landfill (also known as Winnebago Reclamation Landfill) presents a hazard to human health or welfare or to the environment and to evaluate potential remedial alternatives. Tasks are directed toward accomplishing these primary objectives. The Pagel's Pit landfill RI/FS is a PRP lead investigation.

3.1 BACKGROUND

Pagel's Pit landfill is located about five miles south of Rockford, Illinois (see Drawing C 12660-B1). Land use around the landfill is primarily agricultural. The land surface is gently rolling, exhibiting 10 to 200 feet of relief. Surface topography is a reflection of glacial deposition. The primary drainage out in the area is via Killbuck Creek, which flows to the northwest converging with the Kishwaukee and Rock Rivers.

The site is situated in a transition area between outwash deposits to the north, associated with the Kishwaukee River, and a clay till to the south. The glacial deposits range in thickness from five feet or less east of the landfill to 60 feet or greater west of the landfill. The immediately underlying glacial deposit is bedrock of the Galena Dolomite Formation. The Galena Dolomite is characterized as a yellowish gray to buff or brown, medium to coarsely crystalline dolomite. Beneath Galena Dolomite are the Decorah and Platteville Dolomite Formations. Based upon published information and observations made during prior drilling, all the dolomite units are assumed to be interconnected and Beneath the Ordovician Dolomites is the Glenwood Formation, which is an



STATE LOCATION MAP

REGIONAL TOPOGRAPHY, VICINITY AND STATE LOCATION MAPS

WINNEBAGO RECLAMATION SERVICE LANDFILL
PARTS OF
SECTION 36, T.43N., R.1E.,SECTION 31, T.43N., R.2E.
WINNEBAGO COUNTY, ILLINOIS

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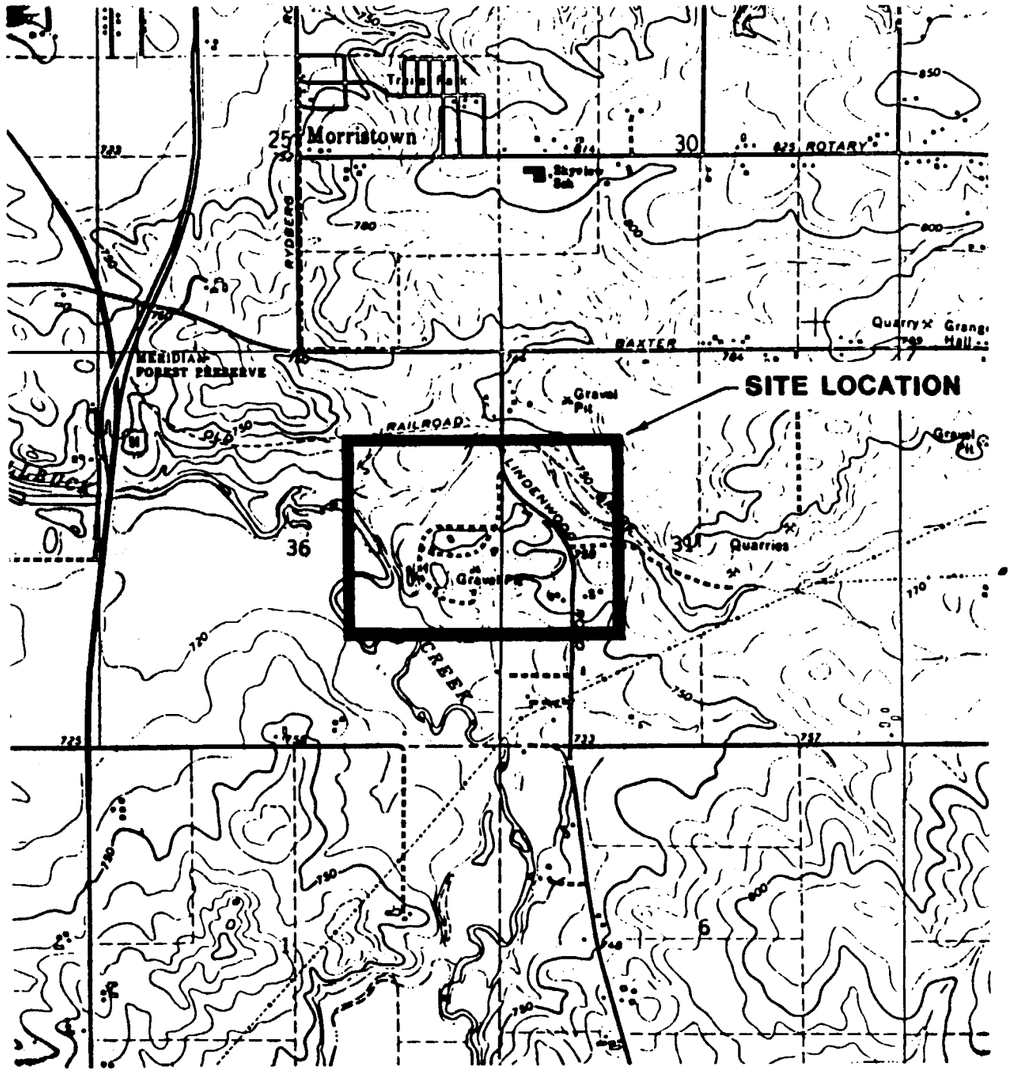
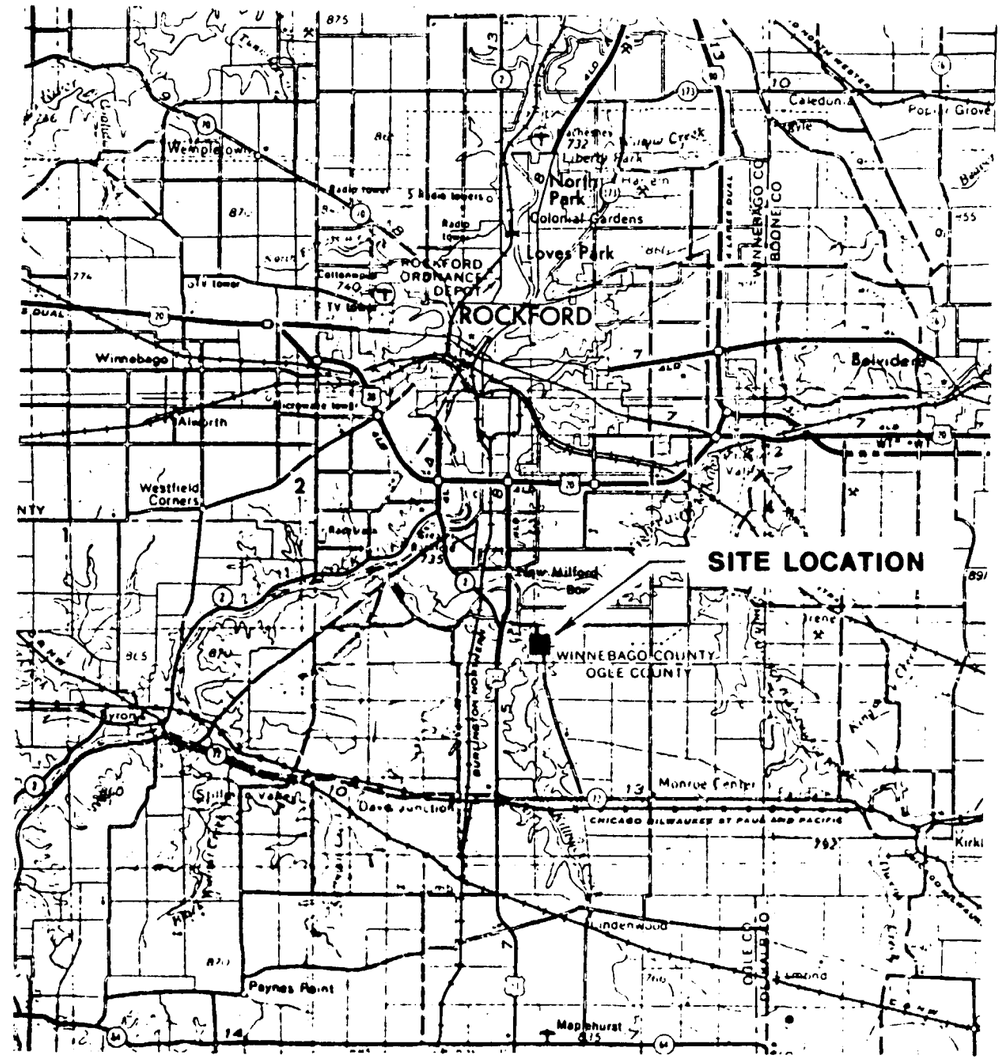
	CHECKED RJK	DATE 11/21/86	DRAWING NO. C12660-BI
	APPROVED Daniel W. Hall		
	REFERENCE		PRINTED DEC 29 1987



REGIONAL TOPOGRAPHY MAP

SCALE: 1" = 2000'



VICINITY MAP

SCALE : 1" = 4 MILES

NOTES

1) BASE MAP FOR THE REGIONAL TOPOGRAPHY MAP OBTAINED FROM THE ROCKFORD SOUTH U.S.G.S. 7 1/2 MIN. QUADRANGLE MAP DATED 1971.

2) BASE MAP FOR THE VICINITY MAP OBTAINED FROM THE U.S.G.S. ROCKFORD, ILL. MAP NO. NK 16-4.

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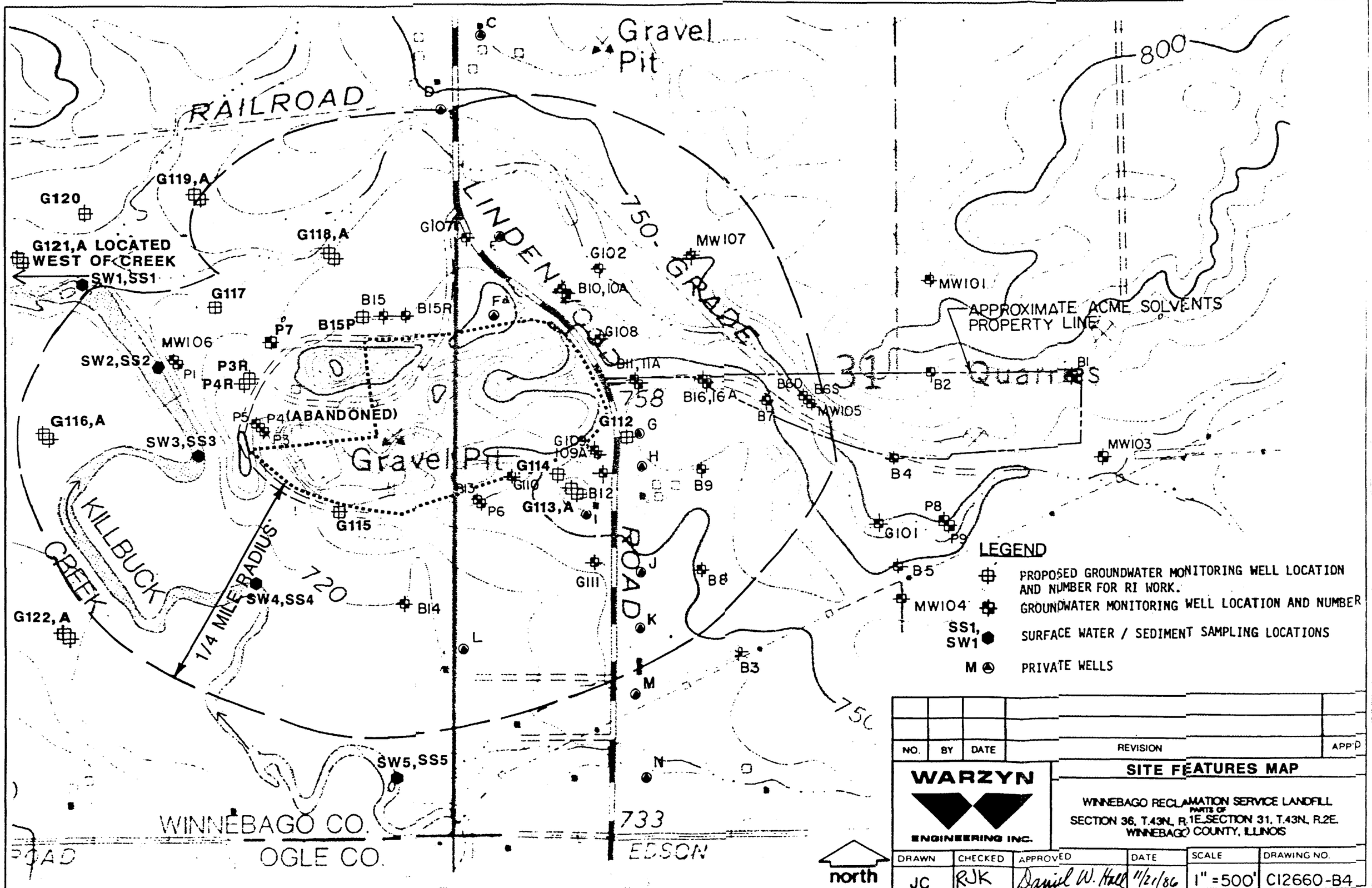

NO.	BY	DATE	REVISION	APP'D

WARZYN ENGINEERING INC.

SITE FEATURES MAP

WINNEBAGO RECLAMATION SERVICE LANDFILL
PARTS OF
SECTION 36, T.43N., R.1E., SECTION 31, T.43N., R.2E.
WINNEBAGO COUNTY, ILLINOIS

DRAWN	CHECKED	APPROVED	DATE	SCALE	DRAWING NO.
JC	RJK	Daniel W. Hall	11/21/86	1" = 500'	C12660-B4

interbedded dolomite, sandstone and shale. The Glenwood Formation may provide some hydraulic separation of the overlying Ordovician Formations from the underlying early Ordovician and Cambrian Sandstones.

Pagel's Pit landfill occupies approximately 60 acres west of Lindenwood Road (see Drawing C 12660-B4) and has been licensed and in operation since 1972. The facility has a bituminous liner with an overlying sand granular blanket and leachate collection system. Leachate is collected and disposed of off-site. Wastes accepted at the landfill are primarily municipal refuse and sewage sludge. A very limited quantity of special wastes, primarily liquid wastes containing metals, were disposed of at the facility prior to December, 1975. All special wastes accepted at the facility were permitted by IEPA. Permits for disposal from IEPA were obtained for some wastes which were not disposed of at the facility.

In 1979, methane gas was detected in the landfill. To prevent accumulation and to put the methane gas to constructive use, the facility operators installed a gas venting and collection system. Collected gas is currently cleaned and used as an energy source in a nearby grain alcohol plant and to dry municipal sewage sludge prior to landfilling.

Adjacent Facilities

East of the Pagel's Pit landfill on an approximately 20-acre parcel, is Acme Solvents Reclamation Inc. (ACME). The ACME site was active from 1960 to 1973 and was used for disposal of waste generated by the company's solvent reprocessing facilities in Rockford, Illinois. The materials disposed of at

the site are generally undocumented, but are known to have included still bottom sludges, non-recoverable solvents, paints and oils.
Waste materials were transported to the ACME site in drums that were either emptied into unlined disposal lagoons or stockpiled. The IEPA indicates that four lagoons were actively used for the disposal of waste materials on site. IEPA additionally indicates that, when the site was closed, between 10,000 and 15,000 drums may have been present on site. The total quantity of waste disposed of at the site during its operation is unknown.

An IEPA inspection in late 1972 and early 1973 indicated that waste materials in the ACME ponds were not removed, but were covered with soils borrowed from other portions of the site. It is also reported that an unknown number of drums stored on-site were crushed and buried rather than removed. Clean-up of the ACME site began in August, 1986. Initial remedial activities include removal of buried drums and contaminated soils.

Groundwater Flow

Groundwater flow is generally from east to west, from ACME toward Pagel's Pit landfill. Killbuck Creek, west of the landfill, probably acts as a local groundwater discharge area. An intermittent stream between the two sites may also seasonally charge groundwater. The water table is located in bedrock at the ACME site. Farther west, beneath Pagel's Pit landfill, bedrock dips off sharply and the water table is found in glacial outwash. Groundwater is generally found at a depth of approximately 20 feet west of Pagel's Pit landfill and at about 40 feet beneath the ACME site.

Prior Investigations

The Pagel's Pit landfill has been peripheral to investigations at ACME due to its proximity to the ACME site, which is currently on the NPL. Previous investigations at ACME are as follows:

- "Extent of Sources of Groundwater Contamination - ACME Solvents, Pagel Pit area near Morrisville, Illinois", Ecology and Environment Inc. (E&E) March, 1983.
- "ACME Solvents Superfund Site, Winnebago County, Illinois, Remedial Investigation", E.C. Jordan Company, September, 1984.
- "ACME Solvents Superfund Site, Winnebago County, Illinois, Preliminary Feasibility Study", E.C. Jordan Company, February, 1985.
- "Data Analysis and Summary Report for Deep Groundwater Assessment, ACME Solvents Superfund Site", E.C. Jordan, January, 1986 (Draft).

These investigations have identified a volatile organic compound (VOC) plume eminating from ACME and moving toward Pagel's Pit landfill, which is composed primarily of chorinated ethenes and ethanes (Table 1). Chlorinated ethenes and ethanes have also been observed in groundwater near Pagel's Pit landfill; however, limited testing of landfill leachate has not indicated a major presence of these compounds. Discontinuities in the contaminant plume between the two sites and changes in the relative proportion of chlorinated ethene and ethane species between observation points has caused prior investigations to conclude that separate plumes were emitting from the ACME and Pagel's Pit landfill sites. The Field Investigation Team Report (FIT) investigation also indicated the presence of As, Cd and Ba (maximum concentrations 30, 17 and 1200 ug/L, respectively) in groundwater adjacent to the landfill site.

In response to nomination for inclusion on the NPL, a supplemental investigation was undertaken by Pagel's Pit landfill to better define groundwater flow and chemistry between the landfill and ACME and to attempt to separate groundwater quality impacts of the two sites. Results of this investigation are reported in:

> "Supplemental Investigation, Winnebago Reclamation Landfill, Rockford, Illinois", Warzyn Engineering Inc., March, 1985.

The study indicated that the composition of the contaminant plume eminating from ACME was generally similar to that observed in prior studies (Table 2). However, no evidence was obtained in this study to support the contention that Pagel's Pit landfill is a major source of the noted groundwater contaminants. Examination of subsurface geology indicated that fracture flow could account for apparent plume discontinuities indicated by the current data base. In addition, known decomposition reactions for chlorinated ethenes and ethanes were found to be consistent with differences in relative amounts of contaminant species with position in the plume. Hence, the presence of different VOC species in the contaminant plume near the landfill are not necessarily indications of separate sources of contaminants.

TABLE 1

MAXIMUM CONCENTRATION OF VOLTILE AND SEMI-VOLTILE ORGANICS IN GROUNDWATER DURING THE 1984 E.C. JORDAN STUDY OF THE ACME - PAGEL'S PIT LANDFILL AREA

COMPOUND	MAXIMUM CONCENTRATION (ug/L)
Volatile Compounds	
trichloroethene	170
1,2 dichloroethene	2400
vinyl chloride	160
1,1,1 trichloroethane	240
1,1 dichloroethane	150
benzene	7.9
1,1 dichloroethene	13
ethylbenzene	31
toluene	40
xylene	280
Semi-Volatile Compounds	
bis (2-ethylhexyl) phthalate	220

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TABLE 2

MAXIMUM CONCENTRATIONS OF VOLATILE ORGANICS
OBSERVED IN GROUNDWATER MONITORING WELLS DURING THE 1984-1985
WARZYN STUDY OF THE ACME-PAGEL'S PIT LANDFILL AREA

COMPOUND	MAXIMUM CONCENTRATION (ug/L)
tetrachlorethene	68
trichlorethene	130
1,2 dichloroethene	1020
vinyl chloride	140
1,1,1 trichloroethane	28
1,1 dichloroethane	170
chloroethane	5.9
benzene	42
chorobenzene	3.5
2 chloroethyl vinyl ether	1.5
1,4 dichlorobenzene	11
1,2 dichloroethane	8.5
1,1 dichloroethene	4.4
dichloromethane	62
1,2 dichloropropane	30
cis- 1,3-dichloropropane	2.2
ethylbenzene	18
toluene	55
m-xylene	30
o,p xylene	29
acetone	210
methyl ethyl ketone	21
tetrahydrofuran	130
trichlorofluoromethane	1

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3.2 PROJECT OBJECTIVES AND USE OF DATA

The RI will be performed to gather and assess information needed to accomplish the following objectives:

- Define the extent to which observed releases of volatile organic compounds in the groundwater are attributable, if at all, to Pagel's Pit Landfill.
- Evaluate the performance of the landfill, with emphasis on whether there are, have been or are likely to be releases of contaminants into the groundwater system.
- Provide additional definition of the groundwater flow system and groundwater quality between Pagel's Pit landfill and ACME Solvents, and determine the source(s) of any hazardous releases.
- Perform an endangerment assessment (EA) to evaluate the effects of any known or potential hazardous releases from Pagel's Pit landfill on the public health, welfare and/or the environment.
- Develop and evaluate remedial alternatives that may be needed for the Pagel's Pit landfill facility.

All tasks, subtasks, and activities are directed toward the accomplishment of these primary objectives.

Data from prior investigations are not sufficient to clearly define independent effects the ACME and Pagel's Pit landfill sites have on the local groundwater system. The proposed study will evaluate potential releases from Pagel's Pit landfill through examination of landfill leachate and groundwater adjacent to and downgradient of the landfill site. Results will be used to evaluate potential hazards associated with releases and to evaluate whether a separate source of VOCs may be eminating from the landfill. Additional definition of the flow system between the two sites will be made to evaluate contaminant transport and potential contaminant sources.

The majority of data collected will be used for site characterization and risk assessement purposes. Some data collected using direct-reading, portable instruments will be used primarily for health and safety purposes. Separate determination of cis- and trans- 1, 2-dichloroethene will be performed to aid in interpretation of spatial changes in relative quantities of plume components resulting from reductive dechlorination reactions. As the cis-isomer is the primary decomposition product of trichloroethene, its concentration in the plume will aid in determining whether changes in plume composition are the result of such reactions or an indication of a separate contaminant source. The method used for VOC determination will also provide lower detection limits then CLP RAS methods, which will allow greater sensitivity for delineating any plume boundary.

3.3 Specific Project Subtasks and Activities

Subtask 3A - Landfill Characterization
Subtask 3B - Hydrogeologic Investigations
Subtask 3C - Sediment Sample Collection
Subtask 3D - Surface Water Investigation
Subtask 3E - Air Monitoring

The QAPP is limited to applicable activities within the above subtasks as discussed below. More complete descriptions of activities within subtasks can be found in the Work Plan and Sampling Plan.

Activity 3A.2 - Leachate Sampling

Up to five rounds of leachate sampling are proposed to determine the composition of landfill leachate for comparison with groundwater composition. In each round, samples will be obtained from two manholes which serve as collection points for two independent leachate collection systems. Additional samples will be obtained from several of the 70 existing gas extraction leachate

head wells. The leachate collection area will be divided into quadrants with one well from each quadrant sampled during each successive sampling round. Specific locations to be sampled are given in the Sampling Plan (Appendix B).

Activity 3A.3 - Water Balance Evaluation

A water balance will be prepared for the landfill using the HELP model in order to evaluate and compare potential leachate supplies with amounts of leachate collected. Required data will include precipitation, water level measurements in leachate head wells and amount of leachate pumped from the site.

The USEPA HELP model is a quasi-3 dimensional landfill hydrologic model which uses daily data in predicting cover runoff, evapotranspiration, lateral drainage and percolation. The model may also be used for analysis of leachate collection system and landfill liner performance. The model accepts daily precipitation data and monthly average data on insolation and temperature, soils characteristics, growing season and leaf area index data to compute the water balance using a layered hydrologic routing model. Details of the development and operation of the model are contained in Schroeder, et al (1984)[1] and examples of calibration and verification of the model are described in Peyton and Schroeder (1986)[2] and Peters, et al (1986)[3].

1 Schroeder, P.R., Morgan, J.M., Walski, T.M., and Bigson, A.C., "The Hydrologic Evaluation of Landfill Performance (HELP) Model, Volume 1" EPA/530/SW-84-009, U.S. EPA, Cincinnati, OH, 1984, 120 pp.

2 Peyton, L., and Schroeder, P.R., "Verification of the Hydrologic Evaluation of Landfill Performance (HELP) Model using Field Data" (authors draft), to be published in Land Disposal of Hazardous Waste - Proceedings of the Annual Research Symposium, U.S. EPA, Cincinnati.

3 Peters, N., Warner, R.C., Coates, A.L., and Logsdon, D.S., "Applicability of the HELP Model in Multilayer Cover Designs; A field verification and modeling assessment", (authors draft), to be published in Land Disposal of Hazardous Waste - Proceedings of the Annual Research Smyposium, U.S. EPA, Cincinnati.

The HELP model analysis will be performed using available local climatological data. Daily precipitation data were obtained from National Oceanic and Atmospheric Administration (NOAA) for the period 1952 to the present for the Rockford Reporting Station. Editing of data to remove years containing substantial missing data will occur. Average monthly temperature data was also obtained from NOAA records for Rockford. Insolation and growing season data will be taken from the records for St. Cloud, Minnesota contained within the HELP model default data files. Selection of soil characteristics will be taken from the 20 sets of default soils data contained in the HELP model, based upon on-site soil classifications, and on test data for the clay barrier soils.

The HELP model analysis for the 6 inch topsoil/24 inch clay cover for closed areas and an analysis using 6 inches of sand cover for the active areas will be made using the layering and soils characteristics described in the proceeding paragraph. A sensitivity analysis will be performed to evaluate the effects of differences in rooting depths, vegetation density and competency of the clay barrier on predicted percolation rates.

Activity 3B.1 - Groundwater Monitoring System

Fifteen additional wells will be installed to supplement the existing groundwater monitoring network. At each location where a single well or nest of wells is installed, soil from the deepest boring will be sampled at five foot intervals to its terminus. Samples will be retained by Warzyn or the PRPs for inspection for a period of 1 year. No chemical analyses of these samples are planned.

An additional five wells may be installed if initial groundwater monitoring indicates a need.

Activity 3B.2 - Surveying

Groundwater monitoring wells will be surveyed for location elevation. Well locations will be surveyed to ± 1.0 foot using an electronic "total station" instrument, which uses an infrared light source to determine distances. Elevations of the top of the inner and outer well casings will be obtained to an accuracy of ± 0.01 foot. Ground elevation at each well will be obtained to an accuracy of ± 0.10 foot. Elevations will also be obtained using the "total station" system. All total station output is received as LED output. The instrument is calibrated on a monthly basis.

Activity 3B.3 - Groundwater Flow Characterization

Water levels in all functional groundwater monitoring wells will be measured on a monthly basis for a nine-month period. On site permeability tests will be conducted at 15 wells using a baildown and recovery method (Appendix E). The wells selected will provide permeability information on bedrock, sand and gravel, and clay till soils.

Water levels will be obtained from groundwater monitoring wells with a fiberglass measuring tape and attached steel sounding device. The tape is calibrated in one hundredths of a foot intervals and is used to measure the depth to water from the surveyed top of the well casing. The sounding device makes a popping noise when contact with the groundwater surface is encountered. Water level measurements are considered accuarate to ±0.01 foot.

Activity 3B.4 - Groundwater Quality Monitoring

A maximum of four rounds of monitoring are proposed. The initial two rounds will include 43 monitoring well samples and 1 private well sample. Samples from the private well will not be filtered prior to analysis for metals. A reduced number of samples is anticipated in rounds three and four (a minimum of 15 monitoring wells and 1 private well). Samples for metals analyses from the private well will similarily be unfiltered and, thereby, reflect total metals content.

Subtask 3C - Sediment Sample Collection

Stream sediment samples will be collected from Killbuck Creek at five locations in order to evaluate the nature and extent of contamination that may be present. One upstream location and four downstream locations will be sampled. Sampling will proceed from furthest downstream location to furthest upstream location to minimize contamination from sediments disturbed in the sampling process.

Subtask 3D - Surface Water Investigations

Surface water samples will be collected at sediment sampling sites on two occasions. Sampling will be concurrent with first round groundwater sampling, and if needed, concurrent with third round groundwater sampling.

TABLE 3
SAMPLE TYPES AND ESTIMATED SAMPLE NUMBERS
PAGEL'S PIT LANDFILL

(1) MATRIX	(2) LAB	NO. OF SAMPLES	FIELD DUPLICATES	(3) FIELD BLANKS	(4) MATRIX SPK/DUP	(5) GC/MS CONFIRM	(6) TOTAL SAMPLES	(7) TEST PARAMETERS
GROUNDWATER								
ROUND 1	HAZLETON	44	5	3	6	5	63	VOLATILES HAZLETON'S METHOD
	HAZLETON	17	2	1	2		22	EPA HSL BNA'S AND PEST/PCB'S
	HAZLETON	17	2	1			20	EPA HSL METALS, DISSOLVED
	WARZYN	17	2	1			20	CYANIDE
	WARZYN	44	5	3			52	PHENOL
	WARZYN	44	5	3			52	ALKALINITY, CHLORIDE
	HAZLETON	27	3	2			32	METALS, DISSOLVED (As, Ba, Cd)(8)
ROUND 2	HAZLETON	44	5	3	6	5	63	VOLATILES HAZLETON'S METHOD
	WARZYN	44	5	3			52	PHENOL
	WARZYN	44	5	3			52	ALKALINITY, CHLORIDES
	HAZLETON	44	5	3			52	METALS, DISSOLVED (As, Ba, Cd)(8)
ROUND 3 (optional)	HAZLETON	16	2	1	2		21	VOLATILES HAZLETON'S METHOD
	WARZYN	16	2	1			19	PHENOL
	WARZYN	16	2	1			19	ALKALINITY, CHLORIDES
	HAZLETON	16	2	1			19	METALS, DISSOLVED (As, Ba, Cd)(8)
ROUND 4 (optional)	HAZLETON	16	2	1	2		21	VOLATILES HAZLETON'S METHOD
	WARZYN	16	2	1			19	PHENOL
	WARZYN	16	2	1			19	ALKALINITY, CHLORIDES
	HAZLETON	16	2	1			19	METALS, DISSOLVED (As, Ba, Cd)(8)
LEACHATE								
ROUND 1	HAZLETON	6	1	1	2	1	11	VOLATILES HAZLETON'S METHOD
	HAZLETON	6	1	1	2		10	EPA HSL BNA'S AND PEST/PCB'S
	HAZLETON	6	1	1			8	EPA HSL METALS, TOTAL
	WARZYN	6	1	1			8	CYANIDE
	WARZYN	6	1	1			8	PHENOL
	WARZYN	6	1	1			8	ALKALINITY, CHLORIDE
ROUND 2	HAZLETON	6	1	1	2	1	11	VOLATILES HAZLETON'S METHOD
	WARZYN	6	1	1			8	PHENOL
	WARZYN	6	1	1			8	ALKALINITY, CHLORIDES
	HAZLETON	6	1	1			8	METALS, TOTAL (As, Ba, Cd)
ROUND 3	HAZLETON	6	1	1	2	1	11	VOLATILES HAZLETON'S METHOD
	WARZYN	6	1	1			8	PHENOL
	WARZYN	6	1	1			8	ALKALINITY, CHLORIDES
	HAZLETON	6	1	1			8	METALS, TOTAL (As, Ba, Cd)
ROUND 4	HAZLETON	6	1	1	2	1	11	VOLATILES HAZLETON'S METHOD
	WARZYN	6	1	1			8	PHENOL
	WARZYN	6	1	1			8	ALKALINITY, CHLORIDES
	HAZLETON	6	1	1			8	METALS, TOTAL (As, Ba, Cd)

TABLE 3
SAMPLE TYPES AND ESTIMATED SAMPLE NUMBERS
PAGEL'S PIT LANDFILL
(Continued)

MATRIX (1)	LAB (2)	NO. OF SAMPLES	FIELD DUPLICATES	FIELD BLANKS (3)	MATRIX SPK/DUP (4)	GC/MS CONFIRM (5)	TOTAL SAMPLES (6)	TEST PARAMETERS (7)
ROUND 5	HAZLETON	6	1	1	2	1	11	VOLATILES HAZLETON'S METHOD
	WARZYN	6	1	1			8	PHENOL
	WARZYN	6	1	1			8	ALKALINITY, CHLORIDES
	HAZLETON	6	1	1			8	METALS, TOTAL (As, Ba, Cd)
SURFACE WATER								
ROUND 1	HAZLETON	5	1	1	2	1	10	VOLATILES HAZLETON'S METHOD
	WARZYN	5	1	1			7	PHENOL
	WARZYN	5	1	1			7	ALKALINITY, CHLORIDES
	HAZLETON	5	1	1			7	METALS, TOTAL (As, Ba, Cd)
ROUND 2	HAZLETON	5	1	1	2	1	10	VOLATILES HAZLETON'S METHOD
	WARZYN	5	1	1			7	PHENOL
	WARZYN	5	1	1			7	ALKALINITY, CHLORIDES
	HAZLETON	5	1	1			7	METALS, TOTAL (As, Ba, Cd)
SEDIMENT								
	HAZLETON	5	1				6	EPA HSL VOLATILES
	HAZLETON	5	1				6	EPA HSL BNA'S AND PEST/PCB'S
	HAZLETON	5	1				6	EPA HSL METALS
	WARZYN	5	1				6	CYANIDE
AIR								
	RADIAN	4	3	1			8	VOLATILES

(1) All samples are considered low concentration samples
(2) Hazleton - Hazleton Laboratories America
3301 Kinsman Boulevard
Madison, Wisconsin 53704
Warzyn - Warzyn Engineering Inc.
One Science Court
University Research Park
Madison, Wisconsin 53705
Radian - Radian Corporation
Progess Center
3200 E. Chapel Hill Rd/Nelson Hwy.
P.O. Box 13000
Research Triangle Park, North Carolina
(3) A trip blank for volatiles will be included with each shipment.
(4) Sample numbers reflect extra volume required for matrix spike/matrix spike duplicate analyses.
(5) Ten percent of all volatiles analyses by Hazleton's method will be confirmed by GC/MS analysis.
(6) Sample totals do not include trip blanks for volatiles.
(7) See Appendix A for CLP TCL list. See Appendix D for analyte list for volatiles analysis by Hazleton's method. See Appendix F for analyte list for volatiles analysis of air samples using Radian's method.
(8) Groundwater samples from private wells will not be filtered

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SAMPLE QUANTITIES, BOTTLES, PRESERVATIVES AND PACKAGING
FOR WATER, SEDIMENT AND LEACHATE SAMPLES FROM PAGEL'S PIT LANDFILL

Analysis	Bottles and Jars	Preservation	Holding Time	Volume of Sample	Shipping	Normal Packaging
WATER AND LEACHATE						
Low Concentration (Organics)						
Acid Extractables, base/neutral extractables, pesticides/PCBs	Two 1/2-gallon amber[1] bottles (teflon-lined caps)	Iced to 4°C	5 days until extraction	Fill bottle to neck	Shipped Daily by Overnight Carrier	No. 1 foam liner or vermiculite
Volatiles (GC)	Four 40-ml volatile organic analysis (VOA vials)	Iced to 4°C	7 days (48 hours for surface water)	Fill completely no headspace	Shipped Daily by Overnight Carrier	No. 1 foam liner or vermiculite
Low Concentration (Inorganics)						
Metals (groundwater)	One 1-liter high density polyethylene bottle	Filter through 0.45 um filter, HNO_3 to pH <2 Iced to 4°C, Optional	6 months	Fill to shoulder of bottle	Shipped Daily by Overnight Carrier	No. 2 foam liner or vermiculite
Metals (leachate and surface water)	One 1-liter high density polyethylene bottle	HNO_3 to pH <2 Iced to 4°C	28 days	Fill to shoulder of bottle	Shipped Daily by Overnight Carrier	No. 2 foam liner or vermiculite
Cyanide	One 1-liter high density polyethylene bottle	NaOH to pH >12 Iced to 4°C	14 days	Fill to shoulder of bottle	Shipped Daily by Overnight Carrier	No. 2 foam liner or vermiculite
Other Analysis						
Phenolics, Total	1/2 liter glass bottle teflon lined cap	H_2SO_4 to pH <2 Iced to 4°C	28 days	Fill 3/4 full	Shipped Daily by Overnight Carrier	Vermiculite
Chlorides, Alkalinity	One 1-liter high density polyethylene	Iced to 4°C	28 days (14 days for alkalinity)	Fill to shoulder of bottle	Shipped Daily by Overnight Carrier articles	No. 2 foam liner or vermiculite
SEDIMENTS						
Low or Med Concentration (Organics)						
Acid extractables, base/neutral extractables, pesticides/PCBs	One 8-oz wide mouth glass jar	Iced to 4°C	10 days until extraction	Fill 3/4 full	Shipped Daily by Overnight Carrier	Foam liner No. 3 (Med in cans/ vermiculite)
Volatiles	Two 120-ml VOA vials	Iced to 4°C	10 days	Fill completely no headspace	Shipped Daily by Overnight Carrier	Vermiculite (Med in cans/ vermiculite)
Low or Med Concentration (Inorganics)						
Metals and Cyanide	One 8-oz wide mouth glass jar	Iced to 4°C	Not established	Fill 3/4 full	Shipped Daily by Overnight Carrier	Foam liner No. 3 (Med in cans/ vermiculite)
AIR SAMPLES						
Volatiles	Tenax/Tenax-charcoal	None	14 days	Not applicable	Shipped Daily by Overnight Carrier	Capped, sealed in glass vials, packed in vermiculite

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Subtask 3E - Air Monitoring

Air sampling will be conducted at three downwind and one upwind locations at property boundaries in order to obtain background data and evaluate potential impacts to downwind receptors. An attempt will be made to collect samples during "worst case" conditions.

Weather data will be collected concurrent with air monitoring. Weather-related information will be collected using the TERRA 8 datalogger. The TERRA 8 datalogger is a fully programmable computer/data collection system that can acquire weather-related data from a variety of sensing transducers for extended periods of time. The TERRA 8 is programmable and data collected is recorded in a non-volatile RAM memory for download to the user via cassette.

The TERRA 8 is housed in a weather proof box and, when established in the field, requires minimal maintenance. Weather data is collected via specific sensing transducers, which sense and monitor information and relay it back to the TERRA 8. The information is read into the datalogger according to specific tailer-made programs by the user or by the Standard Users Program, which is company-installed into the datalogger. There are a variety of sensing transducers, i.e., dry-bulb temperature, wind speed, wind direction, relative humidity, etc. Once data has been acquired, it is kept in the TERRA 8's non-volatile RAM memory for download to the user via cassette. Data collected and stored in memory may be withdrawn with various programs that allow data files to be read from cassette tape for analysis.

Specific procedures to be used in sample collection for the various sample matrixes are outlined in the Sampling Plan (Appendix B). A summary of sample numbers and matrices is given in Table 3. A summary of sample containers, sample volumes, preservation and shipment methods to be used is given in Table 4. A project schedule is given in Attachments 1A and 1B.

4.0 PROJECT ORGANIZATION AND RESPONSIBILITY

4.1 OVERALL RESPONSIBILITY

- Gary L. Marzorati
  Pagel's Pit landfill PRP Steering Committee
- James A. Hill, Project Manager
  Warzyn Engineering
- Karen Waldvogel, Remedial Project Manager
  U.S. EPA, Region V
- RI/FS Reports and technical memoranda prepared by Warzyn Engineering

4.2 MONITORING AND SAMPLING OPERATIONS AND QC

- Principal Engineering Firm - Warzyn Engineering
- Drilling - Exploration Technology Inc.
- Sampling, Monitoring and Survey - Warzyn Engineering
- Quality Control - Richard W. Maurer, Warzyn Engineering
  (Quality Assurance Officer or his designate

4.3 LABORATORY ANALYSES AND QC

- EPA HSL volatiles, semi-volatiles, pesticides/PCBs and metals using CLP protocols (see Appendix A for HSL Analyte List) and volatiles by GC (see Appendix D for method and analyte list) - Hazleton Laboratories America, Inc., 3301 Kinsman Boulevard, Madison, Wisconsin 53704
- cadmium, barium, aresenic, phenols, chloride, total cyanide and alkalinity - Warzyn Engineering
- Field analysis of pH and specific conductance - Warzyn Engineering

4.4 SPECIALIZED RESPONSIBILITIES FOR LABORATORY SERVICES

- Hazleton Data

  - analytical protocol specified - Warzyn Engineering
  - review of analytical specifications - U.S. EPA, Region V, QA Office
  - review and approval of performing laboratory - U.S. EPA, Region V, QA Office

  - internal QA/QC - Hazleton staff
  - final data review and validation - Warzyn Engineerng
  - review of tentatively identified compounds and assessment of need for confirmation - Warzyn Engineering

- Warzyn Data

  - review of analytical specifications - U.S. EPA, Region V, QA Office
  - review and approval of performing laboratory - U.S. EPA, Region V, QA Office
  - internal QA/QC - Warzyn Engineering
  - final data review and validation - Warzyn Engineering

4.5 QUALITY ASSURANCE

- Overall QA Responsibility - Warzyn Quality Assurance Officer (QAO)

- Warzyn subcontracted activities - Warzyn Engineering

- Review of QAPP - U.S. EPA, Region V, QA Office

- Field analyses - Warzyn Engineering

4.6 PERFORMANCE AND SYSTEMS AUDITS

- Field Operations - QAO, Warzyn Engineering

- Analytical Laboratories - U.S. EPA, Region V, QA Office

- Evidence Audit - QAO, Warzyn Engineering

A project organizational chart is shown in Drawing C 12660-A1.

5.0 QUALITY ASSURANCE OBJECTIVES

The overall QA objective is to develop and implement procedures of field sampling, chain-of-custody, laboratory analysis and quality control (QC) reporting that will provide legally defensible results of documentable quality. Specific procedures to be used for sampling, chain-of-custody, calibration, laboratory analysis, reporting, internal quality control, audits, preventative maintenance and corrective actions are described in other sections of this QAPP. This section defines the goals for the of QC effort (accuracy, precision, and sensitivity of analyses and completeness, representativeness and comparability of measurement data from analytical laboratories) and presents quality control objectives for field measurements.

5.1 LEVEL OF QUALITY CONTROL EFFORT

5.1.a Field Sampling Program

The quality of data from the field sampling program will be evaluated through the collection of field duplicates, matrix spike/matrix spike duplicates and field and trip blanks. Duplicate samples will be used to assess the combined effects of sample collection, handling and analysis on data precision. Blank samples will be used to check for procedural contamination or ambient conditions at the site that may cause contamination. The general level of effort for all matrices will be one field duplicate per 10 investigative samples. Field blanks will be collected for water and leachate samples at a frequency of one per collection method per sampling time on day. For organics analysis of water and leachate samples, additional sample volume will be collected for matrix spike/matrix spike duplicate analyses at a frequency of one per 20 investigative samples. One trip blank will be included with each batch of water samples for volatiles analysis.

5.1.b Laboratory Analysis

Hazleton

Hazleton will perform analyses for purgable organics by GC using the method described in Appendix D. Level of QA effort for this method is included with the method description. Selected samples will be analyzed for purgables by GC/MS for confirmation purposes. Analyses will follow CLP protocols except that a 25 ml purge volume will be used to achieve detection limits comparable to those obtainable by the GC method (see Appendix A for analyte list and detection limits). Hazleton will perform analyses for semi-volatiles, pesticides/PCBs and metals (see Appendix A for analyte lists) using CLP protocols. Level of QA effort for CLP protocol analyses are described in IFBs WA-85-GJ664/J680 (or most recent) for organics and WA-85-J838/J839 (or most recent) for inorganics.

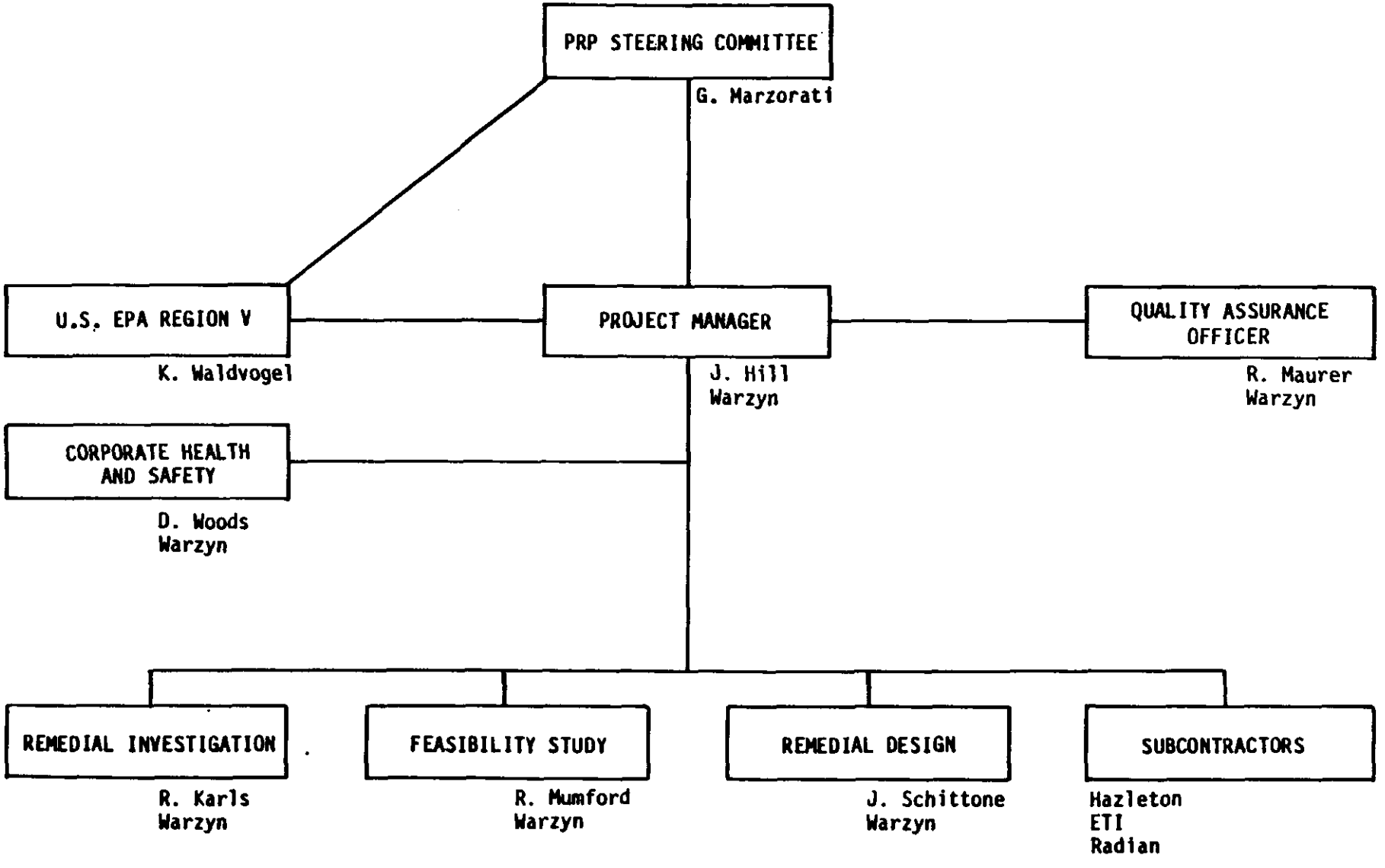
Warzyn

Level of QA effort for analyses performed by Warzyn are included with methods descriptions in Appendix D.

Radian

Level of QA effort for analyses of air samples by Radian is given in the method QA plan in Appendix C-9.

PROJECT ORGANIZATION CHART
PAGEL'S PIT LANDFILL RI/FS



DRAWING 12660-A1

5.1.c Field Measurement of pH and Specific Conductance

Level of QA effort for field measurement of pH will consist of precalibration using two buffer solutions and calibration verification at regular intervals (at least every ten samples). Level of QA effort for specific conductance measurements will consist of initial and continuing (at least every ten sample) calibration verification using a standard solution of known conductivity.

5.2 Accuracy, Precision and Sensitivity of Analyses

The QA objectives of analyses with respect to accuracy, precision and sensitivity are to achieve acceptable data based on specified performance criteria. Accuracy and precision requirements and method detection limits for CLP protocol analyses are described in IFB's WA-85-J664/J680 (or most recent) for organics and WA-85-J838/J839 (or most recent) for inorganics. Accuracy and precision criteria and required detection limits for other proposed analyses are listed with method descriptions in Appendix D.

Accuracy of field measured pH will be judged from agreement of instrument readings with standard buffer solutions. Agreement with standards will be within $\pm$ 0.05 pH units or the unit will be recalibrated. Field measurements will be made to 0.01 unit. Measurement precision will be estimated by periodically (1 per 10 samples) making duplicate readings of samples.

Accuracy of the conductivity meter will be assured by daily calibration verification with a check standard. If readings vary more than 5% from expected values, the unit will be replaced.

5.3 Completeness, Representativeness and Comparability

It is anticipated that at least 95% of analyses will provide results meeting acceptance criteria. Sampling methods and locations are designed to provide results representative of the matrix at the sampling point. Analytical methods used will provide comparable data which will supplement prior data collected at the site.

6.0 Sampling Procedures

Specific sampling procedures used are documented in the Sampling Plan (Appendix B). Containers, preservatives, holding times, transport and storage methods are summarized in Table 4.

7.0 Sample Custody

Sample documentation will follow guidelines in National Enforcement Investigations Center Policies and Procedures, May 1978 (Revised May 1986), except that organics and inorganics traffic reports will not be used. Sample documentation will include chain-of-custudy forms, sample tags, custody seals, sample identification records, container labels and field note books. Examples of documentation to be used are given in Appendix F. Original field notes and field documents will be maintained by Warzyn in a final evidence file. Format and maintenance of the final evidence file is given in Appendix G.

8.0 Calibration Procedures, Frequency and Preventative Maintenance for Field Instruments

Calibration and maintenance of pH and specific conductance meters are detailed in Appendices C-1 and C-2, respectively.

Calibration of survey instruments used for health and safety purposes will follow procedures recommended by the manufacturer (Appendices C-3 through C-8). The HNu and TIP will be calibrated at the beginning of each work day using standard calibration gas (isobutylene) supplied by HNu. The OVA will be calibrated once per month using methane, as specified by the manufacturer, unless conditions warrant more frequent calibration. These conditions might include extensive use of instruments, particularly rough treatment or alteration of initial calibration conditions (i.e., turning of knob settings, replacement of burner chamber, etc.).

HCN Monitox detectors will be checked for accuracy each work-day morning using a gas generation unit. If detectors are not in calibration, they will be replaced with ones which are.

All Rad-Mini radiation meters used on-site have been calibrated at the factory within the past year (in accordance with manufacturer's directive). A thorium camping lantern mantle will be kept on-site to verify operation of the meters.

9.0 Analytical Services

9.1 Hazleton

A. Sample Custody

Internal custody procedures for Hazleton are documented in Appendix I. When analyses and validation of results are completed, the laboratory will deliver all custody forms, sample tags, and all original documents, printouts or data files generated during analyses to Warzyn for inclusion in the final evidence file. A final evidence file will be maintained by Warzyn as described in Appendix G.

B. Analytical and Calibration Procedures

Samples analyzed by Hazleton for HSL semi-volatiles, pesticides/PCBs and inorganics (see Appendix A for analyte list) will follow CLP protocols outlined in IFBs WA-85-J664/J680 or most recent) for organics and WA-85-J838/J839 (or most recent) for inorganics. Analysis for HSL volatiles (see Appendix A for analyte list) in sediment will also follow the above protocols. Volatile organics analysis for groundwater, surface water and leachate samples will be performed by using the method described in Appendix D. Analysis of ten percent of water and leachate samples for volatiles will be confirmed by GC/MS using CLP protocols with the exception that a 25 ml purge volume will be used. Analyte list and detection limits are given in Appendix A.

C. Internal Quality Control

Internal quality control for CLP based analyses will follow specifications in IFB documents WA-85-J664/J680 (or most recent) for organics and WA-85-J838/J839 (or most recent) for inorganics. Internal quality control requirements for volatiles analysis by GC using Hazleton's method are described with analytical methodology in Appendix D.

D. Performance and Systems Audits

The Region V QAO will audit the performing laboratories as a basis for approval or disapproval of the labortory for the requested analyses. Performance audits are to be made as specified in the appropriate IFB documents for analyses by CLP protocols and in Appendix D for analysis of volatiles by GC.

E. Data Assessment

Data assessment will be performed by Warzyn Engineering. Assessment of organics data will be performed using guidelines developed in Technical Directive Document No. HQ-8410-01, Laboratory Data Validation, Functional Guidelines for Evaluating Organics Analyses, May 1985. Inorganics data assessment will be performed based on guidelines in Laboratory Data Validation, Functional Guidelines for Evaluating Inorganics Analyses, November, 1985. Validation of volatile analyses by GC with follow guidelines listed in the method description in Appendix D.

F. Accuracy/Precision Definitions

Accuracy and precision definitions are described in the above IFB documents for analyses performed using CLP protocols and with the method description in Appendix D for volatiles analysis by GC.

G. Corrective Action

If quality control audits result in detection of unacceptable conditions or data, the Warzyn QAO will be responsible for developing and initiating corrective action. Corrective action may include:

- Re-analysis of the samples, if holding time criteria permits;
- Resampling and re-analysis;
- Evaluating and amending sampling and analytical procedures; and
- Accepting data, acknowledging level of uncertainty.

9.2 Warzyn

A. Sample Custody

Samples are delivered to Warzyn under chain-of-custody. A designated sample custodian accepts custody of the shipped samples and verifies that the chain-of-custody seals have not been broken. The sample custodian reviews the information on the smaple tags/labels with that on the chain of custody records. Pertinent information as to shipment, pickup, courier, etc., is entered in the remarks section. The custodian then enters that sample tag/label data into a bound logbook which is arranged by project code and station number. The sample custodian must acknowledge receipt on the chain-of-custody form. Any comments pertaining to the shipment should be made under "Remarks".

The sample custodian will use the sample tag/label as identification information to assign a unique sequenial laboratory number to each sample. This laboratory number is entered on the chain-of-custody form. The information is logged into the laboratory logbook. The sample custodian will transfer all samples to the proper analyst or store the sample in the appropriate refrigerator. The chain-of-custody and testing request forms are forwarded to the laboratory supervisor.

The laboratory is a secured area with strict limited access. All data files and doors are locked daily. Laboratory personnel are responsible for the care and custody of samples from the time they are recieved until the sample is returned to the custodian or refrigerated.

When sample analyses and necessary quality assurance checks have been completed by the laboratory or after a 3 month time period, whichever is longer, the unused portion of the sample should be disposed of properly. All identifying tags, data sheets, and laboratory records shall be retained as part of the permanent documentation of the project and forwarded to the project manager for inclusion in the final evidence file.

B. Analytical and Calibration Procedures

Analytical and calibration procedures are documented in Appendix D.

C. Internal Quality Control

Required internal quality control analyses and their frequency are specified with analyses procedures in Appendix D.

D. Performance and Systems Audits

The Region V QAO will audit the laboratory as a basis for approval or disapproval for the requested analyses. Performance audits with independent QC samples are to be performed as specified in methods descriptions in Appendix D.

E. Data Assessment

Data assessment will be performed by Warzyn Engineering. Criteria to be used for assessment are listed with methods descriptions in Appendix D.

F. Accuracy/Precision Definitions

Accuracy and precision definitions are given with methods descriptions on Appendix D.

G. Corrective Action

If quality control audits result in detection of unacceptable conditions or data, the Warzyn QAO will be responsible for developing and initiating corrective action. Corrective action may include:

- Re-analysis of the samples, if holding time criteria permits;
- Resampling and re-analysis;
- Evaluating and amending sampling and analytical procedures; and
- Accepting data, acknowledging level of uncertainty.

9.3 Radian

Analysis of VOCs in ambient air will be performed by Radian. Methods, sample documentation and QA/QC for this method are described in Appendix C-9. The Region V QAO will audit the laboratory as a basis for approval in disapproval of the required analysis.

10.0 Quality Assurance Reports

No separate QA report for this project is planned. The final RI report and FS report will contain separate QA sections that summarize quality of data collected during the project.

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APPENDICES

A

APPENDIX A

EPA Hazardous Substance List and Requested Detection Limits for Analyses Based on Contract Laboratory Program Protocols

CLP TARGET COMPOUND LIST
AND DETECTION LIMITS*

Volatiles	CAS Number	Detection Limits(1) Low Water(2) ug/l	Detection Limits(1) Low Soil Sediment(3) ug/kg
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-35-4	5	5
9. 1,1-Dichloroethane	34 75-35-3	5	5
10. trans-1,2-Dichloroethene	156-60-5	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-3	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	75-27-4	5	5
18. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
19. 1,2-Dichloropropane	78-87-5	5	5
20. trans-1,3-Dichloropropene	10061-02-6	5	5
21. Trichloroethene	79-01-6	5	5
22. Dibromoclorometh ane	124-48-1	5	5
23. 1,1,2-Trichloroethane	79-00-5	5	5
24. Benzene	71-43-2	5	5
25. cis-1,3-Dichloropropene	10061-01-5	5	5
26. 2-Chloroethyl Vinyl Ether	110-75-8	10	10
27. Bromoform	75-25-2	5	5
28. 2-Hexanone	591-78-6	10	10
29. 4-Methyl-2-pentanone	108-10-1	10	10
30. Tetrachloroethene	127-18-4	5	5
31. Toluene	108-88-3	5	5
32. Chlorobenzene	108-90-7	5	5
33. Ethyl Benzene	100-41-4	5	5
34. Styrene	100-42-5	5	5
35. Total Xylenes		5	5

Semi-Volatiles	CAS Number	Detection Limits(1) Low Water(4) ug/l	Low Soil Sediment(5) ug/kg
36. Phenol	108-95-2	10	330
37. bis(2-Chloroethyl)ether	111-44-4	10	300
38. 2-Chlorophenol	95-57-8	10	330
39. 1,3-Dichlorobenzene	541-73-1	10	330
40. 1,4-Dichlorobenzene	106-46-7	10	330
41. Benzyl Alcohol	100-51-6	10	330
42. 1,2-Dichlorobenzene	95-50-1	10	330
43. 2-Methylphenol	95-48-7	10	330
44. bis(2-Chloroisopropyl)ether	39638-32-9	10	330
45. 4-Methylphenol	106-44-5	10	330
46. N-Nitroso-Dipropylamine	621-64-7	10	330
47. Hexachloroethane	67-72-1	10	330
48. Nitrobenzene	98-95-3	10	330
49. Isophorone	78-59-1	10	330
50. 2-Nitrophenol	88-75-5	10	330
51. 2,4-Dimethylphenol	105-67-9	10	330
52. Benzoic Acid	65-85-0	50	1600
53. bis(2-Chloroethoxy)methane	111-91-1	10	330
54. 2,4-Dichlorophenol	120-83-2	10	330
55. 1,2,4-Trichlorobenzene	120-82-1	10	330
56. Naphthalene	91-20-3	10	330
57. 4-Chloroaniline	106-47-8	10	330
58. Hexachlorobutadiene	87-68-3	10	330
59. 4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	10	330
60. 2-Methylnaphthalene	91-57-6	10	330
61. Hexachlorocyclopentadiene	77-47-4	10	330
62. 2,4,6-Trichlorophenol	88-06-2	10	330
63. 2,4,5-Trichlorophenol	95-95-4	50	1600
64. 2-Chloronaphthalene	91-58-7	10	330
65. 2-Nitroaniline	88-74-4	50	1600
66. Dimethyl Phthalate	131-11-3	10	330
67. Acenaphthylene	208-96-8	10	330
68. 3-Nitroaniline	99-09-2	50	1600
69. Acenaphthene	83-32-9	10	330
70. 2,4-Dinitrophenol	51-28-5	50	1600
71. 4-Nitrophenol	100-02-7	50	1600
72. Dibenzofuran	132-64-9	10	330
73. 2,4-Dinitrotoluene	121-14-2	10	330
74. 2,6-Dinitrotoluene	606-20-2	10	330
75. Diethylphthalate	84-66-2	10	330

Semi-Volatiles		CAS Number	Detection Limits(1) Low Water(4) ug/l	Low Soil Sediment(5) ug/kg
76.	4-Chlorophenyl Phenyl ether	7005-72-3	10	330
77.	Fluorene	86-73-7	10	330
78.	4-Nitroaniline	100-01-6	50	1600
79.	4,6-Dinitro-2-methylphenol	534-52-1	50	1600
80.	N-nitrosodiphenylamine	86-30-6	10	330
81.	4-Bromophenyl Phenyl ether	101-55-3	10	330
82.	Hexachlorobenzene	118-74-1	10	330
83.	Pentachlorophenol	87-86-5	50	1600
84.	Phenanthrene	85-01-8	10	330
85.	Anthracene	120-12-7	10	330
86.	Di-n-Butylphthalate	84-74-2	10	330
87.	Fluoranthene	206-44-0	10	330
88.	Pyrene	129-00-0	10	330
89.	Butyl Benzyl Phthalate	85-68-7	10	330
90.	3,3'-Dichlorobenzidine	91-94-1	20	660
91.	Benzo(a)anthracene	56-55-3	10	330
92.	bis(2-ethylhexyl)phthalate	117-81-7	10	330
93.	Chrysene	218-01-9	10	330
94.	Di-n-octyl Phthalate	117-84-0	10	330
95.	Benzo(b)fluoranthene	205-99-2	10	330
96.	Benzo(k)fluoranthene	207-08-9	10	330
97.	Benzo(a)pyrene	50-32-8	10	330
98.	Indeno(1,2,3-cd)pyrene	193-39-5	10	330
99.	Dibenz(a,h)anthracene	53-70-3	10	330
100.	Benzo(g,h,i)perylene	191-24-2	10	330

Pesticides	CAS Number	Detection Limits(1) Low Water(6) ug/l	Low Soil Sediment(7) ug/kg
101. alpha-BHC	319-84-6	0.05	8.0
102. beta-BHC	319-85-7	0.05	8.0
103. delta-BHC	319-86-8	0.05	8.0
104. gamma-BHC (Lindane)	58-89-9	0.05	8.0
105. Heptachlor	76-44-8	0.05	8.0
106. Aldrin	309-00-2	0.05	8.0
107. Heptachlor Epoxide	1024-57-3	0.05	8.0
108. Endosulfan I	959-98-8	0.05	8.0
109. Dieldrin	60-57-1	0.10	16.0
110. 4,4'-DDE	72-55-9	0.10	16.0
111. Endrin	72-20-8	0.10	16.0
112. Endosulfan II	33213-65-9	0.10	16.0
113. 4,4-DDD	72-54-8	0.10	16.0
114. Endosulfan Sulfate	1031-07-8	0.10	16.0
115. 4,4'-DDT	50-29-3	0.10	16.0
116. Endrin Ketone	53494-70-5	0.10	16.0
117. Methoxychlor	72-43-5	0.5	80.0
118. Chlordane	57-74-9	0.5	80.0
119. Toxaphene	8001-35-2	1.0	160.0
120. AROCLOR-1016	12674-11-2	0.5	80.0
121. AROCLOR-1221	11104-28-2	0.5	80.0
122. AROCLOR-1232	11141-16-5	0.5	80.0
123. AROCLOR-1242	53469-21-9	0.5	80.0
124. AROCLOR-1248	12672-29-6	0.5	80.0
125. AROCLOR-1254	11097-69-1	1.0	160.0
126. AROCLOR-1260	11096-82-5	1.0	160.0

Notes:

(1) Detection limits listed for soil/sediment are based on wet weight. The detection limits calculated by the laboratory for soil/sediment will be on dry weight basis and will be higher.

(2) Medium Water Detection Limits (DL) for Volatile CLP Target Compounds are 100 times the individual Low Water RDL.

(3) Medium Soil/Sediment DL for Volatile CLP Target Compounds are 100 times the individual Low Water DL.

(4) Medium Water DL for Semi-Volatile CLP Target Compounds are 100 times the individual Low Water DL.

(5) Medium Soil/Sediment DL for Semi-Volatile CLP Target Compounds are 60 times the individual Low Soil/Sediment DL.

(6) Medium Water DL for Pesticide CLP Target Compounds are 100 times the individual Low Water DL.

(7) Medium Soil/Sediment DL for Pesticide CLP Target Compounds are 15 times the individual Low Soil/Sediment DL.

* Specific detection limits are highly matrix dependent. The detection limits listed herein are provided for guidance and may not always be achievable.

[cac-79-13]

ELEMENTS DETERMINED BY INDUCTIVELY COUPLED PLASMA EMISSION
OR ATOMIC ABSORPTION (AA) SPECTROSCOPY

Metal:	Required Detection Level (ug/l)
Alumium	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	5
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20

[cac-79-14]

METHOD DETECTION LIMITS
VOLATILE HSL COMPOUNDS
25 mL PURGE METHOD

HAZLETON LABORATORIES

Instrument: FINN 5100A
Calibration date: 02-06-87

in ug/L

Laboratory ID COMPOUND	LAM217 1 ppb	LAM218 1 ppb	LAM219 1 ppb	STD DEV	M.D.L.
Chloromethane	1.000	0.705	0.758	0.16	0.47
Bromomethane	1.000	0.975	0.888	0.06	0.18
Vinyl Chloride	1.000	0.815	0.777	0.12	0.36
Chloroethane	1.000	1.639	1.531	0.34	1.03
Methylene Chloride	1.000	1.220	1.107	0.11	0.33
Acetone	1.000	1.626	1.511	0.33	1.00
Carbon Disulfide	1.000	1.075	0.972	0.05	0.16
1,1-Dichloroethene	1.000	0.902	0.891	0.06	0.18
1,1-Dichloroethane	1.000	0.959	0.936	0.03	0.10
Trans-1,2-Dichloroethene	1.000	0.965	0.944	0.06	0.19
Chloroform	1.000	1.116	1.012	0.06	0.19
1,2-Dichloroethane	1.000	0.956	1.015	0.03	0.09
2-Butanone	ND	ND	ND		
1,1,1-Trichloroethane	1.000	0.694	0.706	0.17	0.52
Carbon Tetrachloride	1.000	0.860	0.868	0.08	0.24
Vinyl Acetate	1.000	0.972	0.987	0.01	0.04
Bromodeichloromethane	1.000	0.874	0.901	0.07	0.20
1,2-Dichloropropane	1.000	0.871	0.899	0.07	0.20
Trans-1,3-Dichloropropane	0.780	0.691	0.728	0.04	0.13
Trichloroethene	1.000	0.877	0.904	0.06	0.19
Dibromochloromethane	1.000	0.893	0.994	0.06	0.18
1,1,2-Trichloroethane	1.000	0.912	1.006	0.08	0.23
Benxene	1.000	0.871	0.910	0.06	0.20
cis-1,3-Dichloropropane	1.220	0.957	1.022	0.14	0.41
2-Chloroethylvinylether	1.000	0.722	0.923	0.14	0.43
Bromoform	1.000	0.927	1.038	0.06	0.17
4-Methyl-2-Pentanone	1.000	0.992	1.123	0.07	0.22
2-Hexanone	1.000	0.780	0.919	0.11	0.33
Tetrachloroethene	1.000	0.946	0.943	0.03	0.10
1,1,2,2-Tetrachloroethane	1.000	0.801	0.924	0.10	0.30
Toluene	1.000	0.925	0.929	0.11	0.33
Chlorobenzene	1.000	0.977	1.000	0.01	0.04
Ethylbenzene	1.000	1.050	1.023	0.03	0.08
Styrene	1.000	1.049	1.021	0.02	0.07
Total Xylenes	1.000	1.057	1.006	0.03	0.09

ND - Not Detected
For 2-Butanone, a detection limit of 2 ppb has been set based on lab experience.
Compounds spiked into clean lab water.

B

APPENDIX B

Sampling Plan

TABLE OF CONTENTS

INTRODUCTION..... 1

SAMPLING LOCATIONS AND NUMBERS..... 2

Sample Locations..... 2
Groundwater Samples..... 2
Sediment Samples..... 4
Surface Water Samples..... 4
Leachate Samples..... 4
Air Samples..... 5

SAMPLE DESIGNATION..... 7

Project Identifier..... 7
Sample Type and Location Code..... 7
Sampling Event..... 8
Examples of Sample Numbers..... 8

GENERAL SAMPLING EQUIPMENT AND PROCEDURES..... 9

Existing Wells and Piezometers..... 9
Soil Borings/Well Installation..... 9
Groundwater Sample Collection..... 13
Sediment Sampling..... 14
Surface Water Sampling..... 15
Leachate Sampling..... 15
Air Sampling..... 16

SAMPLING HANDLING AND ANALYSES..... 17

Parameters..... 17
Groundwater..... 17
Sediment..... 18
Surface Water..... 19
Leachate..... 19
Air Samples..... 20
Sample Preservation..... 20

SAMPLE DOCUMENTATION..... 21

SCHEDULING..... 22

SAMPLING TEAM MEMBERS..... 22

[sss 800-89]

SAMPLING PLAN
PAGEL'S PIT LANDFILL RI/FS

INTRODUCTION

This sampling plan documents procedures and practices to be used in obtaining soil, bedrock, groundwater, surface water, stream sediment, leachate and air samples at or near the Pagel's Pit Landfill near Rockford, Illinois. A phased approach will be used for monitoring well installation and groundwater sampling. Fifteen monitoring wells will be installed during Phase 1. Groundwater samples will be collected for analyses on two occasions from the 15 Phase 1 monitoring wells, three (3) proposed wells on the Acme Solvents site which will be installed by the Acme Solvents PRPs, one (1) private well and 25 previously installed wells.

If the interim groundwater analysis indicates that additional monitoring is necessary, up to five (5) additional wells may be installed during Phase 2. Two additional sets of groundwater samples will be collected from 15 selected monitoring wells and one (1) private well as part of the Phase 2 investigation.

Five (5) surface water samples will be collected for chemical analyses from Killbuck Creek on two occasions coinciding with the first and third rounds of groundwater sampling. Stream sediment samples will be collected at the five (5) surface water sampling locations concurrent with Phase 1 drilling. Six leachate samples will be collected from site risers and manholes on a bi-monthly basis for approximately eight months (5 sampling rounds). Air monitoring samples will be collected at four (4) locations at the property boundaries.

SAMPLING LOCATIONS AND NUMBERS

Sample Locations

Soil bedrock, groundwater, surface water, sediment, leachate and air samples will be collected from various locations at and in the vicinity of Pagel's Pit Landfill. Drawing C 12660-B4 of the QAPP illustrates proposed sampling locations. Locations of Phase 1 monitoring wells and surface water/sediment sampling locations were agreed upon and staked during the site visit with U.S. EPA and USGS (October 16, 1986). Phase 2 locations were not located at the time of the site visit. Specific sampling locations and the numbers of samples to be obtained are listed below.

Groundwater Samples

The hydrogeologic investigation will be conducted in two phases. In Phase I 13 monitoring wells will be installed at the southeastern and western margins of the Pagel's Pit Landfill site. In addition, two (2) wells will be installed west of Killbuck Creek to monitor groundwater quality on the opposite side of the creek.

Two (2) rounds of groundwater samples will be collected during Phase 1, including all new Phase 1 wells plus existing wells B-4, B-6S, B-6D, B-7, B-9, B-10, B-11, B-11A, B-12, B-14, B-13, B-15, B-16, B-16A, G-107, G-108, G-109, G-109A, G-110, G-111, P-1, P-6, P-7, MW-105, MW-106, replacement wells P-3R and P-4R and three (3) newly installed wells between the Pagel's Pit Landfill and ACME Solvents sites as part of an Acme Solvents site investigation. In addition, one (1) private well at the farm southwest of Pagel's Pit Landfill

(Private Well O) will be sampled, for a total of 44 wells per sampling round. Rounds 1 and 2 will occur approximately 6 weeks apart.

The scope or necessity of the Phase 2 hydrogeologic investigation is dependent on the results of the Phase 1 water quality analyses as presented in the Interim Groundwater Quality Evaluation. If the first two rounds of water quality analyses indicate that Pagel's Pit Landfill is, or appears likely to be responsible for contaminant impacts west of Killbuck Creek, five (5) additional monitoring wells will be installed west of the creek (Wells G-120, G-120A, G-121, G-122 and G-122A on Drawing C 12660-B4), and sampling rounds 3 and 4 will be conducted. Fifteen monitoring wells (including all Phase 2 Wells) and one (1) private well will be sampled in Rounds 3 and 4, except as modified by the Interim Groundwater Quality Evaluation.

If Pagel's Pit Landfill is not found to be responsible for impacts west of the Creek, then the Phase 2 wells will not be installed, and rounds 3 and 4 of groundwater quality monitoring and rounds 4 and 5 of leachate monitoring will not be performed. Quality control sample analyses for the sampling program are summarized in Table 3 of the QAPP.

The sampling program assumes that the wells referenced as part of the Acme Solvents investigation will be installed by ACME PRP's, and further, they will be accessible for sampling. Changes in the Acme Solvents well installation program or inability to gain access wells to under Acme Solvents control will necessitate changes in the Pagels Pit Landfill Work Plan. Changes will be by mutual consent of the Pagels Pit Landfill PRPs, Warzyn and U.S. EPA.

Sediment Samples

Five (5) sediment samples will be collected from Killbuck Creek. The sediment samples will be collected from the same locations where surface water samples are collected, concurrent with the Phase 1 drilling program. Results of sediment analyses for HSL parameters may modify the analytical parameter list for surface water samples. Sampling locations were agreed upon and staked in the field during the site visit. Qaulity control samples will consist of one field duplicate.

Surface Water Samples

Five (5) surface water samples will be collected from Killbuck Creek on two occasions. The first will be concurrent with the first round of groundwater sampling. The second will occur, if needed, after the interim groundwater quality evaluation. Four of the sample locations are located downgradient from the landfill. One sample will be collected at a location upstream from the landfill to provide background surface water quality data. Sampling locations were agreed upon and staked in the field during the site visit. Approximate surface water sampling locations are shown on Drawing C 12660-B4.

A field blank and field duplicate will be collected during each round. Two matrix spike/spike duplicate samples will be collected and a trip blank included for volatiles. A separate sample will be collect at one location for volatiles confirmation by GC/MS.

Leachate Samples

On a bi-monthly basis for approximately eight (8) months (5 sampling rounds), six (6) samples of leachate will be collected at the site at site manholes

and risers. The bi-monthly schedule may be adjusted to distribute sampling to better characterize seasonal variations in leachate quantity and quality. The scheduling will be such that three (3) rounds of leachate samples will be collected before issuance of the Interim Groundwater Evaluation (Subtask 3B.5). No more than five (5) sampling events will occur over the eight (8) month period. The samples will be taken to provide an overall indication of site leachate character. Manholes will be sampled during each round, however, risers sampled will be varied from round to round.

A field blank and duplicate will be collected during each sampling round. Matrix spike/matrix spike duplicate samples and a sample for volatiles confirmation by GC/MS will also be collected.

Air Samples

Air monitoring will be conducted at the property boundaries of the landfill site to determine the presence of volatile organic compounds in the air. The property boundary monitoring program will consist of the collection of four (4) Tenax/Tenax-charcoal samples over a 12-hour period on one occasion. Sampling will include one (1) upwind and three (3) downwind sampling stations. Sampling stations will be determined based on site conditions on the day of the test.

Specific sampling station locations will be determined in the field and will be subject to approval by U.S. EPA and USGS. A recording weather station will be used to provide a record of weather conditions during the test. The results will be used to evaluate the potential for health impacts to downwind receptors. Initially, one round of samples is planned during conditions approaching "worst case". The need for addtional rounds will

potentially impacted by migrating toxic gases, a plan to routinely monitor gases at site boundaries will be prepared.

Quality control samples will consist of a field blank and three samples at one of the downwind locations collected at a series of three flow rates above and below the normal sampling rate.

SAMPLE DESIGNATION

A sample numbering system will be used to identify each sample, including duplicates and blanks. Each sample identifier will have three components: a project identifier; a sample type and location code; and a numerical code indicating the sampling event. A listing of sample identification will be maintained in a log book kept by the site sampler.

Project Identifier

A 2-letter designation will be used to identify the sample collection site. For this project, the designation will be PP, which represents Pagel's Pit Landfill.

Sample Type and Location Code

Each sample collected will be identified by a 1- or 2-digit alpha code corresponding to the type of sample, followed by the sample location number. Alpha codes for monitoring wells are consistent with those of prior investigations. The alpha codes are as follows:

- G, B, P or MW-monitoring well
- SW - Surface water
- SS - Sediment
- LL - Leachate
- AA - air
- BB - bottle blank
- SB - sample blank
- PW - private well

Sample locations will be designated by a number. The numerical designation of the location will follow the alpha code for the type of sample collected. The location numbers used will be as shown on Drawing C 12660-B4 of the QAPP.

Sampling Event

Samples will have an identifier to indicate sampling event ("01", "02, etc.). Duplicate samples will be identified by "91" for the first sampling event, "92" for the second sampling and so on.

Examples of Sample Numbers

Examples of sample numbers are listed below:

- PP-G118-01 = Pagel's Pit Landfill monitoring well groundwater sample from Well G-118, sampling period 1;
- PP-SW02-92 = Pagel's Pit Landfill, surface water location 2, second round duplicate;
- PP-LL06-05 = Pagel's Pit Landfill, leachate sample location 6, round 5;
- PP-SB02-02 = Pagel's Pit Landfill, sample blank 2, second round

GENERAL SAMPLING EQUIPMENT AND PROCEDURES

Existing Wells and Piezometers

Forty-two existing wells and piezometers will be utilized during the remedial investigation. Twenty-five of the existing wells will be sampled during the first and second rounds of sampling. Functional wells will be monitored for water levels during the course of the RI. Table 1 summarizes the details of the existing wells and piezometers on site.

Soil Borings/Well Installation

The proposed monitoring well installation program at the site will be in two phases. Phase 1 wells include the installation of new wells B15P, G112, G113, G113A, G114, G115, G116, G116A, G117, G118, G118A, G119 and G119A. Replacement wells P3R and P4R (two well nest) will be installed to replace abandoned wells P3, P4 and P5. Other non-functional wells that were previously installed by Pagel's Pit Landfill representatives and are deemed necessary to the project, will also be replaced. Non-functional wells are those that are expected to be used during the investigation for water quality sampling as described in this plan, which have been destroyed, or are known to have questionable utility for water quality sampling. Agreements with U.S. EPA and USGS will be made on the necessity for replacement of any non-functional wells.

Attached Drawing C 12660-B4 shows approximate Phase 1 well locations. A brief discussion on each well location is provided below:

Phase I

P3R - Replacement for abandoned water table well P-3 to test for any releases west of the fill and to provide data on the relationship between groundwater flow and Killbuck Creek (estimated to be 40 feet deep).

P4R - Replacement for abandoned piezometer well P-4 having data objectives similar to those for Well P3R (estimated to be 60 feet deep).

B15P - Screened 30 feet below the water table to provide data on water quality at depth adjacent Well B-15 (estimated to be 70 feet deep).

G112 - A water table well (estimated to be 50 feet deep) between the landfill and two impacted water supply wells to the east (G and H). The well may provide an indication if the landfill is impacting the water quality at the water supply wells.

G113, G113A - Nested wells (estimated to be 50 feet and 80 feet deep, respectively) adjacent to a water supply well (I), which appeared to be impacted, but is not currently functional. Determination of flow direction and groundwater quality may provide an indication of the source of contamination at the former water supply well.

G114 - A water table well (estimated to be 60 feet deep) adjacent to the landfill in an area of contaminated groundwater. The well may provide an indication as to whether mounding is occurring under the landfill, and the origin of contaminated groundwater in the area.

G115 - A water table well (estimated to be 40 feet deep) located along the southwest perimeter of the landfill, to monitor for any releases from that portion of the landfill, to test for groundwater mounding, and to provide data on the relationship between groundwater flow and Killbuck Creek.

G116, G116-A - Nested wells (estimated to be 30 feet and 60 feet deep, respectively) to be placed west of Killbuck Creek to evaluate potential migration of contaminants beneath the creek, and to help characterize the hydraulic relationship between Killbuck Creek and groundwater flow.

G117 - A water table well (estimated to be 30 feet deep) adjacent to the landfill and near impacted monitoring wells (MW-106, P1, P7); this well may provide an indication of landfill releases, and data on the hydraulic relationship between groundwater flow and Killbuck Creek.

G118, G118-A - A nest of wells (estimated to be 30 feet and 60 feet deep) in an area which appears to be downgradient of monitoring wells B15 and B15R. These wells will help evaluate the extent of any releases from the landfill.

G119, G119A - A water table well (30 feet deep) and a piezometer (estimated to be 60 feet deep) to investigate the hydraulic relationship between groundwater flow toward Killbuck Creek, and to assist in the evaluation as to whether there are releases from the landfill.

New wells G120, G121, G121A, G122 and G122A are identified as Phase 2 wells (see Drawing C 12660-B4). These wells, or a modification thereof, will be installed, if the first two rounds of water quality monitoring data indicate that the Pagel's Pit Landfill is, or appears likely to be responsible for contaminant impacts west of Killbuck Creek.

Phase II

G120 - This well is estimated to be 30 ft deep and has a similar purpose to those stated for G119.

G-121, G121A - Nested wells (estimated to be 30 ft to 60 ft deep, respectively) placed west of Killbuck Creek, to evaluate potential migration of contaminants beneath the creek, and determine hydraulic relationship between groundwater flow and Killbuck Creek.

G 122, G-122A - These wells with estimated depths of 40 to 70 ft have the same purpose as G116 and G116A.

At each location where a single well or nest of wells will be installed, the deepest boring will be sampled to its terminus. The deepest borings at multiple well locations will be sampled with a split-spoon sampler at the surface and 5-ft intervals to the top of bedrock using ASTM Method D1586. Bedrock will be sampled by collection of HW cores (3.2 in. diameter) using ASTM Method D2113. If necessary, casing will be used to prevent caving of the borehole and to prevent loss of drilling fluid. Soil and rock samples will be logged in the field by a geologist who will be observing drilling operations. Measurements of recovered cores will be made to allow calculation of percent recovery and rock quality designation. Samples will be screened in the field with an OVA or HNu meter to determine the potential for contamination by organic compounds. Cutting will be wasted on site. Waste liquid will be drummed if screening with HNu or OVA shows above background readings.

Representative soil and rock samples will be provided to U.S. EPA representatives at their request, in the field if adequate sample volume is recovered. Soil and rock samples will be retained by Warzyn or the PRP's for a period of one year (per U.S. EPA request). U.S. EPA may review the samples at Warzyn's or the PRP's facility.

The groundwater monitoring wells will be constructed as follows:

- Wells will be constructed with threaded, flush-joint, two-inch inside diameter stainless steel screens and riser pipe to a level at least 5 feet above the anticipated maximum groundwater elevation. Galvanized steel riser pipe will be used above the high groundwater elevation to approximately 2 feet above ground surface.
- Manufactured 0.010-inch slotted screens will be used. Water table wells will be instrumented with 10-foot (nominal length) screens and piezometers will be instrumented with 5-foot (nominal length) screens.
- The results of screening of the soil and rock samples with the OVA or HNu will be considered when determining the placement of piezometer screens.
- Wells will have a clean No. 30 flint sand pack placed around the well screens extending to two (2) feet above the top of the screen. A 2-foot bentonite pellet seal or bentonite slurry will be placed immediately above the sand pack. The remainder of the annular space will be backfilled with a bentonite or bentonite/cement grout. Where more than five (5) feet of grout needs to be placed, a tremie pipe will be used.
- A locking, steel protective casing will be installed over each well.

Drilling equipment and tools will be decontaminated between boreholes to minimize possible cross contamination of wells. Drilling rods, bits and tools used will be washed with 2.5 percent tri-sodium phosphate (TSP) solution and steam cleaned prior to drilling and between boring locations. Steam cleaning of the drill rig will be performed when necessary to avoid cross contamination between locations based on the judgement by the field geologist. The split-spoon samplers and core barrel will be washed with a TSP solution and rinsed with

clean water between samples. Well screen and pipe will be steam cleaned prior to well installation.

Groundwater Sample Collection

Prior to purging each well for sampling, a water level measurement will be taken using a fiberglass tape with a steel sounding device attached to the end. The fiberglass tape will be divided into 0.01-foot increments, the accuracy of measurements is ±0.02-foot. The tape will then be used to measure the total depth of each well. Measuring the total depth of each well will provide well identification at well nests and indicate the amount of sediment accumulation at the bottom of the well, if any.

The sampling sequence will be based upon observations made during drilling and analytical results from previous sampling of existing wells. Groundwater sampling will proceed from the anticipated least contaminated wells to the wells suspected of having greater amounts of contaminants. Each well to be sampled will be purged immediately prior to sampling using a stainless steel bailer attached to a 1/8-inch diameter stainless steel cable. A pitcher pump may be used for purging if groundwater is shallow enough to make use of the pump possible. The water removed from the well will be collected and measured so a minimum of four well volumes are removed prior to sample collection. If a pitcher pump is used for purging, an additional one well volume will be removed with a stainless steel bailer prior to sampling to remove all water subjected to the negative pressure of the pitcher pump. After well purging has been completed, the samples will be collected using a stainless steel bailer. Purge water will be screened with a photoioization meter and drummed if readings are above background.

Groundwater sample blanks will be collected by pouring deionized water into the bailer and filling the sample bottles from the bailer. Deionized water for all blanks will be obtained from the Warzyn analytical laboratory for which background data are available.

The bailer, cable, pump and water level measurement tape will be cleaned between wells with a 2.5 percent TSP solution followed by a rinse with reagent grade acetone and, finally, double rinsed with distilled water. The pitcher pump and riser pipe will be steam cleaned and then will go through decontamination with TSP, acetone and distilled water. The pitcher pump riser pipe and bailer, will be triple rinsed with distilled water poured directly from containers in order to remove all acetone residuals from decontamination. The sampling equipment will be laid out on and covered with clean polyethylene sheeting to air dry as much as possible before reuse.

Sediment Sampling

Sediment samples will be collected at each of the surface water sampling locations concurrent with Phase I drilling.

The sediment samples will be collected using a 2-inch diameter stainless steel hand corer or clam shell sampler. Samples will be grab samples taken at representative points on the eastern one half of the stream profile, or as otherwise agreed to with U.S. EPA.

Sampling equipment will be decontaminated between sampling locations using decontamination fluids and distilled water rinses as described for goundwater sampling.

Surface Water Sampling

Sampling of surface water will progress from the furthest downstream location (SW-1) to the furthest upstream location (SW-5) to limit influence of sediment suspended by sampling on susequent samples. Surface water samples will be collected from the lower portion of the stream channel using a Kemmerer sampler or by taking grab samples. Samples will not be filtered. A photograph of each sampling location will be taken. A sampling blank will be collected by pouring deionized water into the sampler and then transferring to a sample bottle.

Sampling equipment will be decontaminated between sampling locations using TSP followed by acetone and, finally, distilled water rinses, as described for groundwater sampling.

Leachate Sampling

Six (6) leachate samples will be collected on five (5) occasions during the RI. Two of the samples in each sampling round will be collected from the leachate extraction system manholes. The remaining four (4) samples in each sampling round will be collected from gas wells that are part of the gas collection system (see attached Drawing C 12660-B5). Four different gas wells will be sampled in each sampling round as per the following plan:

Round 1	Round 2	Round 3	Round 4	Round 5
A1	A3	C7	D1	C5
E7	B1	C3	E5	E3
F1	G3	H1	G5	J3
K2	G7	I5	I3	L2

The leachate samples will be collected using a stainless steel bailer attached to a 1/8-inch diameter stainless steel cable. Samples from the gas wells will be collected by moving the flexible well connector from the PVC riser and then using the bailer to collect the sample. Prior to removing the well connector on the gas wells, appropriate valves will be closed to prevent introduction of clean air into the gas collection system. Leachate collected in excess of sample needs will be combined with that being collected for off-site disposal.

The bailer and stainless steel cable will be decontaminated between sampling locations using decontamination fluids and distilled water rinses as described for groundwater sampling.

Air Sampling

The property boundary air monitoring program will consist of the collection of four Tenax/Tenex-charcoal samples over a 12 hour period. Pre- and post-calibrated personal sampling pumps will be used to collect airborne contaminants. Sampling pumps and gas flow calibrator to be used are described in Appendix C-9 of the QAPP.

A prefilter will be used to prevent particulates from entering the sampling train. Of volatile compounds detected in greatest amounts on site, vinyl chloride has the lowest time weighted threshold limit value and among the highest method detection limits. Hence, sampling parameters were developed based on those recommended by NIOSH for vinyl chloride. An approximate flow rate of 20 cubic centimeters per minute will be used over a 12-hour period. This provides a total sample volume that is less than the maximum volume per

gram of sorbent (charcoal) recommended by NIOSH with about a 50% safety factor for any humidity affects and provides sufficient sensitivity for evaluating the applicable threshold limit value. A simple field blank will be collected. The blank will be treated like other samples except that no air will be drawn through it. At one of the downwind locations three additional samples will be collected at inflow rates of 10, 30 and 40 cubic centimeters per minute. Those samples will serve as a check on artifact formation and to indicate whether breakthrough may be occurring.

A recording weather station will be installed on site to provide a record of pertinent weather parameters, including wind speed and direction, humidity, ambient temperature and barometeric pressure. Sampling will be performed during the day and during conditions approximating "worst case" conditions (i.e. an inversion) if possible. The need for any additional sampling will be assessed based on results of first round sampling.

SAMPLING HANDLING AND ANALYSES

Parameters

Groundwater

Groundwater samples from the following seventeen wells will be analyzed in Round 1 for the Hazardous Substance List (HSL) semivolatiles, pesticide/PCBs, total cyanide and inorganic parameters listed in Appendix A:

1. Monitoring Wells B-15R and B-15P, G-109 and G-109A, G-115, G-116 and G-116A, G-117, G-118, G-118A MW-106 and P-1, and the two replacement wells, P-3R and P-4R.

2. One three-well nest between the two sites (total of three samples), to be installed during Acme Solvent's site investigation.

Analyses for volatile organic compounds (VOCs) will be performed using a method developed by Hazleton (see Appendix D of QAPP for method description and analyte list).

The remaining 27 wells to be sampled in Round 1 will be analyzed for the following:

- VOC's using Hazleton's test procedure; 10% confirmation by Gas Chromatography Mass Spectroscopy (GC/MS).
- Total Phenolics
- Field pH
- Field specific conductance
- Total alkalinity
- Chlorides
- Arsenic (Dissolved for monitoring wells, total for residential well)
- Barium (Dissolved for monitoring wells, total for residential well)
- Cadmium (Dissolved for monitoring wells, total for residential well)

The second round sampling locations will be identical to the first round. Additional parameters may be added to the above list after mutual agreement by Pagel's Pit Landfill PRPs, U.S. EPA and Warzyn if it appears other parameters are important to defining impacts from Pagel's Pit Landfill.

The analyses will be for the parameters listed:

- VOC's using Hazleton's test procedure; 10% confirmation by Gas Chromatography Mass Spectroscopy (GC/MS).
- Total Phenolics
- Field pH
- Field specific conductance
- Total alkalinity
- Chlorides
- Arsenic (Dissolved for monitoring wells, total for residential well)
- Barium (Dissolved for monitoring wells, total for residential well)
- Cadmium (Dissolved for monitoring wells, total for residential well)

Samples collected in Rounds 3 and 4 (if necessary) will be analyzed for the same parameters analyzed in Round 2.

Sediment

The stream sediment samples will be analyzed for HSL organic and metals

Surface Water

The surface water samples will be analyzed for the following:

- VOC's using Hazleton's test procedure; 10% confirmation by Gas Chromatography Mass Spectroscopy (GC/MS).
- Total Phenolics
- Field pH
- Field specific conductance
- Total alkalinity
- Chlorides
- Arsenic (Total)
- Barium (Total)
- Cadmium (Total)

If the HSL analyses of the sediment samples (collected concurrent with Phase 1 drilling) prior to surface water collection detect parameters of interest that are important to defining impacts from Pagel's Pit Landfill other than those listed above, additional parameters may be required on surface water samples. The additional parameters for analyses would be by mutual agreement between U.S. EPA, Warzyn and the Pagel's Pit Landfill PRP's.

Leachate

The first round of leachate samples will be analyzed for HSL semivolatiles, pesticides/PCBs and metals listed in Appendix A using CLP protocols. Total cyanide will be determined by Warzyn using the method described in Appendix D. Analysis for VOCs will be performed using Hazleton's test procedure with 10 percent confirmation by Gas Chromatography Mass Spectroscopy (GC/MS). In addition to the HSL parameters, first round leachate samples will also be analyzed for the following:

- Field pH
- Field specific conductance
- Total Alkalinity
- Chloride
- Total phenolics

All subsequent rounds of leachate samples will be analyzed for the following parameters:

- VOCs using Hazleton's test procedures; 10% confirmation by Gas Chromatography Mass Spectroscopy (GC/MS).
- Total Phenolics
- Field pH
- Field specific conductance
- Total alkalinity
- Chlorides
- Arsenic (Total)
- Barium (Total)
- Cadmium (Total)

Additional HSL parameters may be added to the leachate analyses for the four later sampling events based upon first round results if they are important to defining impacts from Pagel's Pit Landfill. The addition of any parameters would be by mutual consent of U.S. EPA, Pagel Pit's Landfill PRPs, and Warzyn.

Air Samples

The air samples will be analyzed for volatile listed with the method description in Appendix C-9.

Sample Preservation

Samples will be collected and preserved in a manner appropriate for the analyses they receive [see Table 4 of the Quality Assurance Project Plan (QAPP)]. A portion of groundwater and surface water samples used for inorganic analyses will be filtered in the field through a 0.45 micron filter as soon as possible after collection using a pressure filtration device. All filtered portions of the samples will be preserved as appropriate immediately after filtration.

All sample fractions will be preserved before delivery to the laboratory according to the procedures shown in Table 4 of the QAPP. Preservatives added will be prepared using reagent grade chemicals.

SAMPLE DOCUMENTATION

All samples will be collected under chain-of-custody procedures. Standard forms including sample labels, chain-of-custody forms, and custody seals used for sample tracking will be maintained. A brief description of sample documents follows.

A. Chain of Custudy Form

1. One Form per shipping container (cooler).
2. Carrier service does not need to sign form is custody seals remain intact.
3. User for all samples

B. Chain of Custody Seals

1. Two seals per shipping container to secure the lid and provide evidence that samples have not been tampered with.
2. Cover seals with clear tape.
3. Record seal number on Chain of Custody Form.
4. Use for all samples.

C. Sample Tags

1. Each sample container must have a sample tag affixed to it.
2. Sample tag numbers are recorded on the Chain of Custody Forms.
3. Use for all samples.

D. Sample Identification Record Form

1. Will provide means of recording crucial sample shipping and tracking information.

2. Will contain information such as:

   . Sample number
   . Sample matrix
   . Sample location code
   . Sample round
   . Chain of custody number
   . Lab code
   . Date sampled
   . Date shipped
   . Airbill number
   . Sampling tag number

All paperwork accompanying the samples being shipped to the appropriate performing laboratory will be sealed in a plastic bag that is taped to the inside of the cooler lid. Copies of the chain-of-custody forms, and other paperwork (if possible), will be retained for the field files.

Two sample seals will be placed on opposite sides of the lid and extending down the sids of the cooler. The lid will be securely tapered shut prior to shipment. Examples of the required sample documentation from are contained in Appendix F of the QAPP.

SCHEDULING

The sequence of RI sampling events to take place at Pagel's Pit Landfill are as shown on Attachment 1-A of the QAPP.

SAMPLING TEAM MEMBERS

Soil samples and rock cores will be collected by the geologist logging the borehole. The geologist on the drill rig will also act as site safety officer.

Permeabilities will be measured by a team of two people consisting of a hydrogeologist and field technician.

Air sampling will be performed by an industrial hygienist.

The sampling team for stream sediment sampling will be composed of two people. The team will consist of the project hydrogeologist and field technician who are familiar with site conditions and sampling locations.

Leachate sampling will be performed by a hydrogeologist and field technician who are familiar with site conditions. Site (Pagel's Pit Landfill) personnel will also assist during collection of leachate samples.

The first and second rounds of groundwater and samples will be collected by two sampling teams. The sampling teams will consist of a hydrogeologist and a field technician familiar with site conditions. An additional person will be responsible for sample documentation, sample preparation and shipment. The number of sampling teams will be reduced to one for subsequent groundwater sampling rounds.

Surface water samples will be collected by two people familiar with site conditions and sampling locations.

Efforts will be made to use the same personnel throughout the course of the infield work, to optimize familiarity with site conditions.

RJK/jpl/BAW
[adf-50-8]

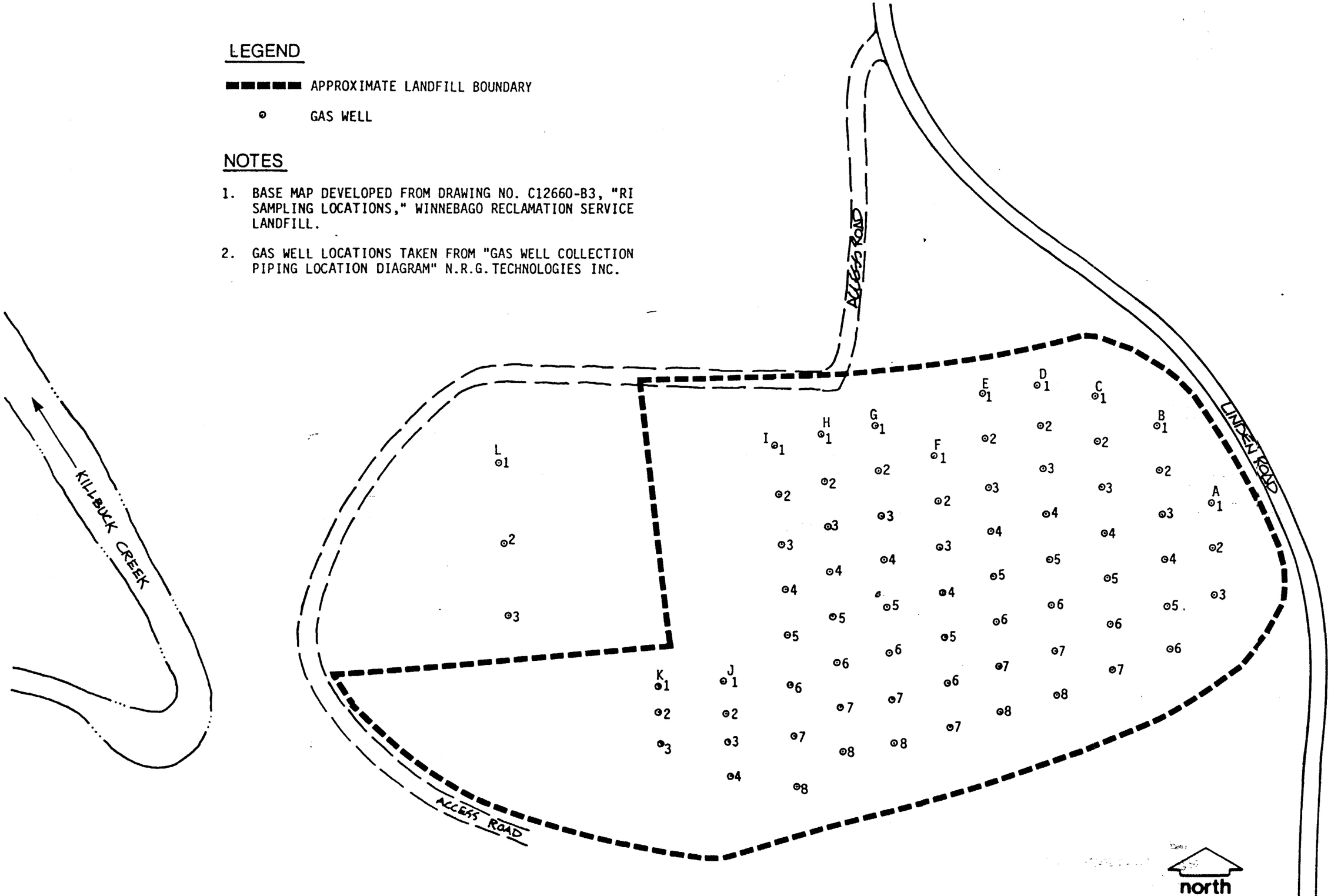
LEGEND

APPROXIMATE LANDFILL BOUNDARY

GAS WELL

NOTES

1. BASE MAP DEVELOPED FROM DRAWING NO. C12660-B3, "RI SAMPLING LOCATIONS," WINNEBAGO RECLAMATION SERVICE LANDFILL.
2. GAS WELL LOCATIONS TAKEN FROM "GAS WELL COLLECTION PIPING LOCATION DIAGRAM" N.R.G. TECHNOLOGIES INC.



WARZYN

LEACHATE SAMPLING LOCATIONS

WINNEBAGO RECLAMATION SERVICE LANDFILL
PARTS OF SECTION 36, T.43N, R.1E, SECTION 31, T.43N, R2E
WINNEBAGO COUNTY, ILLINOIS

1" = 200'

1 OF 1

12660-B5

WARZYN

TABLE 1

MONITORING WELL STATUS
WINNEBAGO RECLAMATION LANDFILL

Well	Top of Casing Elevation	Ground Elevation	Total Depth	Status[1]
B1	772.96	770.55	51.70	F
B2	792.34	790.14	73.50	F
B3	744.78	742.14	40.00	F
B4	757.58	755.06	37.50	F
B5	749.50	747.05	35.00	F
B6S	754.00	751.55	52.00	F
B6D	754.14	751.55	100.00	F
B7	751.75	748.84	33.50	F
B8	749.97	747.15	35.00	F
B9	758.33	755.11	42.50	F
B10	744.17	741.77	40.00	F
B10A	743.78	742.10	61.80	F
B11	760.57	757.58	47.30	F
B11A	758.92	757.10	75.40	F
B12	760.35	758.22	48.90	F
B13	739.28	736.43	32.50	F
B14	716.87	713.93	15.00	F
B15	744.47	741.80	40.00	F
B15R	743.70	742.00	43.20	F
B16	762.81	760.54	45.40	F
B16A	762.58	760.60	69.90	F
G101				F
G102				F
G107	739.45	737.60	36.20	F
G108	751.13	749.20	44.20	F
G109	760.60	758.90	51.70	F
G109A	760.90	758.90	79.50	F
G110	747.90	745.20	43.10	F
G111	740.59	738.80	36.20	F
MW101	799.20	796.92	100.00	F
MW102	760.64	759.24	53.90	F
MW103	751.10	748.36	59.50	F
MW104	756.58	754.28	135.00	F
MW105	752.89	750.65	76.00	F
MW106	725.85	724.65	60.00	F
MW107	749.92	747.90	150.00	F
P1	727.65	725.30	35.00	F
P3	725.59	723.32	20.00	NF[2]
P4	724.67	721.89	40.00	NF[2]
P5	722.85	721.13	60.00	NF[2]
P6	739.53	736.96	50.00	F
P7	728.75	727.13	30.00	F
P8	748.12	745.72		F
P9	748.60	745.80		F

[1] Status of wells as of May 7, 1986 - F=Functional, NF=Non-functional.
[2] Wells P3, P4 and P5 were removed for construction of the new landfill cell.

[cac-79-11]

C

APPENDIX C-1

Field Measurement of pH

APPENDIX C-1

FIELD MEASUREMENT OF pH

Method: Electrometric

Reference: EPA 1979, Page 150.1

Sensitivity: 0.1 pH unit

Optimum Range: 1-12 pH units

Sample Handling: Determine on-site or within 6 hours.

Reagents and Apparatus:

1. pH meter (Orion Model 211 Mini pH meter).
2. Combination electrodes
3. Beakers or plastic cups.
4. pH buffer solutions, pH 4, 7, and 10.
5. Deionized water in squirt bottle.
6. All glassware soap and water washed, followed by two hot water rinses and two deionized water rinses.

Calibration:

1. Place electrode in pH7 buffer solution.
2. After allowing several minutes for meter to stabilize, turn calibration dial until a reading of 7.00 is obtained.
3. Rinse electrode with deionized water and place in pH4 or pH10 buffer solution.
4. Wait several minutes and then turn slope adjustment dial until a reading of 4.00 or 10.00 is obtained.
5. Rinse electrode with deionized water and place in pH7 buffer. If meter reading is not 7.00, follow Steps 2-5 again.

Procedure:

1. Calibrate meter using calibration procedure.
2. Pour the sample into a clean beaker or plastic cup.

3. Rinse electrode with deionized water between samples. Recheck calibration with pH7 buffer solution after every 5 samples.

4. Immerse electrode in solution allowing several minutes for meter to stabilize. Make sure the white KCl junction on side of electrode is in the solution. The level of electrode solution should be one inch above sample to be measured.

Notes:

1. When calibrating the meter, use pH buffers 7 and 4 for samples with pH $\leq$ 8, and buffers 7 and 10 for samples with pH $\geq$ 8. If meter will not read pH4 or 10, something may be wrong with the electrode. Return it to the lab with a note.

2. pH is a temperature dependent analysis. Therefore, temperatures of buffers and samples should be within about 2°C. For refrigerated or cool samples, use refrigerated buffers to calibrate meter.

3. Weak organic and inorganic salts and oil and grease are interferences in pH measurements. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, followed by 10% HCl. Then recalibrate meter.

4. When not in use, the electrode should be stored in pH4 buffer.

5. Before going into the field:

   a) Check batteries;
   b) Do a quick calibration at pH7 and 4 to check electrode;
   c) Obtain fresh solutions.

6. Following field measurements:

   a) Report any problems;
   b) Compare with previous data;
   c) Clean all dirt off of meter and inside case;
   d) Make sure electrode is stored in pH4 buffer.

[cmj-12-13]

APPENDIX C-2

Instruction Manaul Orion Model 211
pH Meter

INSTRUCTION MANUAL

model 211
digital pH meter

ORION RESEARCH

contents

introduction 3

instrument description 3

instrument set-up 4

- support rod
- power source
- meter check-out
- connecting electrodes

measurement procedures 6

- general measurement technique
- pH measurement
  - single-buffer standardization
  - two-buffer standardization

battery replacement 8

recorder output 8

repair and service 8

accessories 9

specifications 10

notice of compliance 11

repair/service

For information on repair or replacement of this instrument, contact Orion Research toll-free. Ask for Customer Service.

ORION RESEARCH INCORPORATED
Customer Service
840 Memorial Drive
Cambridge, Massachusetts 02139 U.S.A.
800-225-1480 (Continental U.S.)
617-864-5400 (Massachusetts, Alaska, Hawaii, Canada)
Telex: 921466

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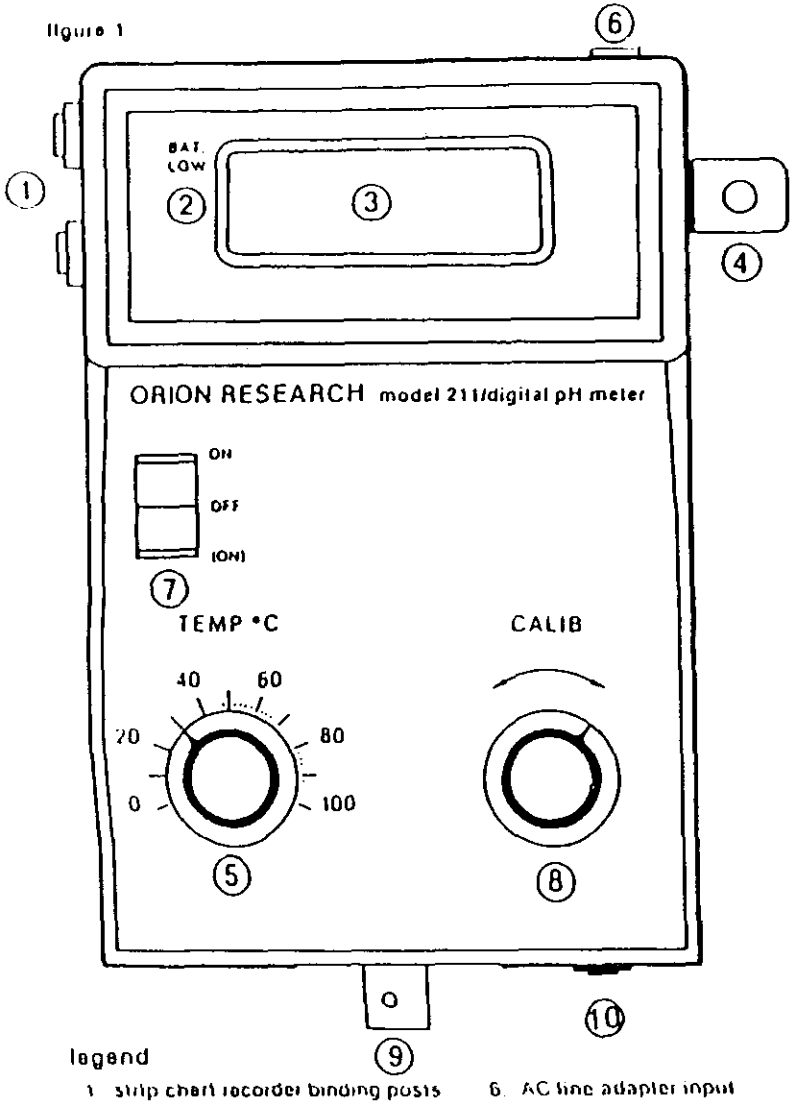
Form IM211/3860
Printed in U.S.A.

figure 1



legend

1. strip chart recorder binding posts
2. BAT LOW
3. LC display
4. support rod clip
5. temperature indicator control
6. AC line adapter input
7. function control
8. calibration control
9. electrode connector
10. slope control

introduction

The Model 211 is a battery- or line-operated (110/220 V AC adapter) digital pH meter for field or laboratory use. The meter is complete with strip chart recorder binding posts and is supplied with an unbreakable, gel-filled combination pH electrode, one packet of pH 7 buffer powder, one bottle for pH 7 buffer, one bottle for distilled water, support rod, electrode holder, AC adapter, six 1.5 V batteries, shorting plug, and carrying case.

instrument description

See figure 1.

1. strip chart recorder binding posts: black post is low (ground) and red post is high input side of recorder. See page 8.
2. BAT LOW: an arrow pointing towards BAT LOW appears on the display when battery requires replacement.
3. LC display: pH display over the range of 0 - 14 with ± .01 pH units resolution
4. support rod clip: holds steel rod used to mount electrode holder.
5. temperature indicator control (TEMP °C): compensates for variation in electrode slope or temperature changes. Used in two-buffer calibration
6. AC line adapter input: jack used to insert AC line adapter. With AC line adapter operational, the internal battery is bypassed
7. function control: rocker switch with three positions - ON, OFF and (ON). Depress (ON) for a momentary reading. The switch will return to OFF when released.
8. calibration control (CALIB): used to calibrate the meter with buffers of known pH.
9. electrode connector: accepts BNC connector from pH electrode.
10. slope control: screwdriver adjustment used to set second buffer in two-buffer calibration.

instrument set-up

support rod

1. Insert steel support rod into the hole in the support rod clip on side of the meter.
2. Mount electrode holder on the rod by pinching to compress the spring. Release to hold in place.

power source

The Model 211 operates on six nonrechargeable 1.5 volt batteries or on 110 or 220 ± 20% V with an approved AC adapter (specify voltage when ordering). Low battery is indicated by the BAT LOW indicator on the display.

NOTE Batteries are not rechargeable - use of line adapter whenever possible will prevent the unit's batteries from being discharged. If battery operation is desired, follow installation instructions under battery replacement.

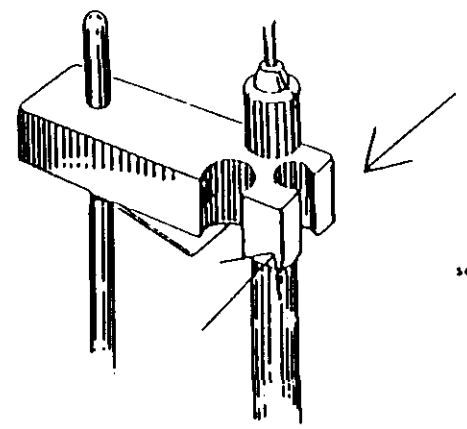
meter check-out

1. Install six AA batteries in the meter. Orient the (+) and (-) battery terminals to match the orientation shown in the battery compartment.
2. Depress ON button on the front panel. If the BAT. LOW indicator on the front display lights up, the batteries must be replaced.
3. If battery mode is not to be used, disregard steps 1 and 2. Insert pin end of appropriate AC line adapter into the meter, and the other end into the appropriate grounded AC line receptacle.
4. Attach BNC shorting plug to BNC input on the bottom side of the meter. Depress ON button on the front panel. Turn CALIB knob so display reads a steady 7.00. If this cannot be done consult ORION Technical Service.
5. Remove the shorting plug. Successful completion of steps 1-4 show the [illegible] is ready for use.

connecting electrode

1. Insert the BNC connector into the electrode jack on the bottom panel of the meter. Turn connector clockwise until it seats firmly.
2. Mount electrode in the electrode holder by spreading the electrode clip open and sliding the electrode into the holder so that the clip closes on electrode cap. See figure 2.
3. Follow measurement procedures to use the meter to measure pH.
4. Disconnnect electrode by turning connector counterclockwise until released from pin.

figure 2



squeeze as shown to insert electrode

measurement procedures

general measurement technique

temperature: All samples and buffers should be at the same temperature, as small variations in temperature can cause errors in measurement. The slope of the pH electrode, the potential of the reference electrode, and the pH of the buffer are temperature-dependent.

cleaning electrodes: Electrode should be rinsed and shaken between measurements to remove drops and to prevent solution carryover.

stirring: Stir measured solutions moderately to obtain good contact between the glass bulb and the solution. Insert electrode to a depth of about 3 cm.

pH measurements

single-buffer standardization
(where maximum precision is not required)

NOTE For maximum accuracy it is recommended that a two-buffer calibration be performed once at the beginning of each day (see page 7). This procedure ensures the correct setting of the slope control. Subsequent measurements during the day may be made using a single point calibration.

1. Place the electrode in a buffer solution whose pH is near the expected pH of the sample. Insert electrode to a depth of about 3 cm and stir moderately.
2. Set the temperature indicator control to the temperature of the buffer.
3. Set the function control to ON and allow the buffer reading to stabilize. Adjust the CALIB so that the display indicates the pH of the buffer at the solution temperature. See Table 1.
4. Remove the electrode from the buffer solution and rinse by stirring moderately in distilled water. Shake off excess drops of water.
5. Place electrode in the sample to a depth of about 3 cm and stir moderately. Set the function control to ON and allow the reading to stabilize. Record the steady pH reading.

two-buffer standardization
(where maximum precision is required)

1. Select two buffers to bracket the expected pH of the sample, with one buffer having a pH of 7.
2. Place the electrode in the pH 7 buffer to a depth of about 3 cm and stir moderately. Set the temperature indicator control to the temperature of the buffer. Set the function control to ON and allow the reading to stabilize. Turn CALIB until the display indicates the pH of the buffer at the solution temperature. See table 1.
3. Remove electrode from the first buffer and rinse by stirring moderately in distilled water. Shake off excess drops of water.
4. Place the electrode in the second buffer to a depth of about 3 cm and stir moderately. Set the function control to ON and adjust the slope control until the pH at the solution temperature is displayed. See Table 1.
5. Remove the electrode and rinse by stirring moderately in distilled water. Shake off excess drops of water.
6. Place the electrode in the sample to a depth of about 3 cm and stir moderately. Set the function control to ON and allow the reading to stabilize. Record the steady pH reading.

TABLE 1

TEMP (°C)	pH 7.00 Buffer	pH 4.01 Buffer	pH 10.01 Buffer
5	7.08	4.00	10.25
10	7.06	4.00	10.18
15	7.03	4.00	10.12
20	7.01	4.00	10.06
25	7.00	4.01	10.01
30	6.98	4.02	9.97
35	6.98	4.02	9.93
40	6.97	4.03	9.89
50	6.97	4.06	9.83
60	6.98	4.09	--

battery replacement

To replace the batteries, remove the panel on the back of the meter. Be sure to observe the polarity marking when inserting new batteries.

recorder output

The red and black binding posts at the side of the meter provide an output for strip chart recording of absolute mV independent of function mode. For recorders with input impedance of 100 Kilohms or greater, the output is fixed to about 100 mV/pH. pH 14.00 output is 1.40 V. Lower impedance recorders may be used but full scale output is reduced.

1. Connect the lead from the high (input side of the recorder) to the red binding post and the lead from the low (ground) side to the black binding post.
2. Proceed according to directions in the strip chart recorder instruction manual.

repair and service

ORION warranty covers failures due to manufacturer's workmanship or material defect from the date of purchase by the user. User should return the warranty card to ORION and retain proof of purchase. Warranty is void if product has been abused, misused, or repairs attempted by unauthorized persons.

Warranties herein are for products sold/installed for use only in the United States and Canada. For ORION products purchased for use in all other countries consult local in country, authorized ORION sales agent/distributor for product warranty information.

A Return Authorization Number must be obtained from ORION Laboratory Products Customer Service before returning any product for in-warranty repair, replacement or credit.

"No Lemon" Instrument Warranty

The instrument is covered by the ORION "No Lemon" warranty. If the instrument fails within twelve months from date of purchase for any reason other than abuse, the purchaser may elect to have it repaired or replaced at no charge. This warranty covers the original or replacement/repaired meter from date of original meter purchase; the warranty is not extended beyond the buyer's original warranty date.

accessories

815600	Ross™ epoxy body, bulb guard combination pH electrode
9104BN	Laboratory grade combination pH electrode (BNC connector)
910600	GX-series epoxy body, gel-filled combination electrode (BNC connector)
912600	GX-series epoxy body, gel-filled flask combination electrode (BNC connector)
913600	GX-series epoxy body, gel-filled flat surface combination pH electrode (BNC connector)
915600	RX-series refillable, epoxy body combination pH electrode (BNC connector)
9162BN	Combination pH electrode with rugged bulb (BNC connector)
9163BN	Combination pH electrode with needle shape (BNC connector)
910004	pH 4 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910007	pH 7 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910009	pH 9 buffer packets, box of 25 packets, each packet making 200 ml of buffer
910104	pH 4.01 buffer, 475 ml bottle
910107	pH 7.00 buffer, 475 ml bottle
910110	pH 10.01 buffer, 475 ml bottle
970899	Dissolved oxygen electrode
910002	Electrode holder
020030	Shorting plug
020120	110V AC line adapter
020121	220V AC line adapter

specifications

package contents	model 211 digital pH meter, with model 910600 gel-filled unbreakable combination pH electrode, support rod, electrode holder, bottles for pH 7 buffer and distilled water, one packet pH 7 buffer powder, AC adapter, six 1.5 V batteries, and carrying case
range	0 to 14 pH
resolution	± .01 pH
temperature compensation	manual (0 to 100°C)
isopotential point	pH 7 (fixed)
power requirement	six 1.5 V batteries; battery life: 3000 ten second intermittent measurements when line adapter is not used. line adapter: 110 or 220 V ± 20%, 50/60 Hz
dimensions	14 cm high x 9 cm wide x 4.5 cm deep
weight	0.4 kg

specifications subject to change without notice

notice of compliance

The Model 211 may generate radio frequency energy and if not installed and used properly, that is, in strict accordance with the manufacturer's instructions, may cause interference to radio and television reception. It has been type tested and found to comply with the limits for a Class B computing device in accordance with specifications in Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference in a residential installation. However, there is no guarantee that interference will not occur in a particular installation. If the Model 211 does cause interference to radio or television reception, which can be determined by turning the unit off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- reorient the receiving antenna
- relocate the Model 211 with respect to the receiver
- move the Model 211 away from the receiver
- plug the Model 211 into a different outlet so that the meter and receiver are on different branch circuits

If necessary, the user should consult the dealer or an experienced radio/television technician for additional suggestions. The user may find the following booklet prepared by the Federal Communications Commission helpful:

"How to Identify and Resolve Radio-TV Interference Problems"

This booklet is available from the U.S. Government Printing Office, Washington, DC 20402, Stock No. 004-000-00345-4.

APPENDIX C-3

Field Measurement of Specific Conductance and Temperature

APPENCIX C-3

FIELD MEASUREMENT OF SPECIFIC CONDUCTANCE AND TEMPERATURE

Method: Specific Conductance, umhos @ 25°C

Reference: EPA 1979, Page 120.1, Standard Methods, 15th edition, pp 70-73

Detection Limit: 1 umho/cm @ 25°C

Optimum Range: 0.1 - 100,000 umhos/cm

Sample Handling: Determine on-site or within 24 hours

Reagents and Apparatus:

1. Conductivity meter (YSI) and electrodes.
2. Deionized water in squirt bottle.
3. Standard potassium chloride solution, 0.0100 N.

Procedure:

YSI Conductivity Meter

1. With mode switch at off position, check meter zero. If not zeroed, use meter screw and adjust to zero.
2. Plug probe into jack on side of meter.
3. Turn mode switch to red line, and turn red line knob until needle aligns with red line on dial. Change batteries if cannot be aligned.
4. Totally immerse probe in sample. Do not allow the probe to touch the sample container.
5. Turn mode switch to appropriate conductivity scale, X100, X10, or X1. Use a scale that will give a mid-range output on the meter.
6. Wait for needle to stabilize (about 15 sec.) and record conductivity multiplying by scale setting.
7. While gently agitating the probe, take sample temperature (°C) and record.
8. Rinse probe with deionized water.
9. Record specific conductivity (1st column) and temperature on F.O.S. sheet.

Notes:

1. Calculate conductivity using following formula:

$$G_{25} = \frac{G_T}{[1 + 0.02\ (T-25)]}$$

G_{25} = Conductivity at 25°C, umhos/cm

T = Temperature of sample, °C

G_T = Conductivity of sample at temperature T, umhos/cm

2. Report results for the standard solution with each data set.

   Results are used to check the accuracy of instrument calibration. If results deviate more than ± 5% from the expected value (1413 umho/cm at 25° C), the instrument is to be replaced.

3. Record on field sheet which meter and probe were used. Meter should be wiped clean as necessary.

4. After returning to lab, compare results with previous data. Report problems to lab personnel.

Reagent Preparation:

1. Stock Potassium Chloride Solution, 1.00 N: Dissolve 74.555 g. K Cl in Milli-Q water and dilute to 1,000 ml. in a volumetric flask.

2. Standard Potassium Chloride Solution, 0.0100N: Dilute to 10.0 mls. of stock solution to 1,000 mls. with Milli-Q water using a volumetric pipet and flask.

APPENDIX C-4

Operating Instructions YSI Model 33 Conductivity Meter

TABLE OF CONTENTS

	Page
GENERAL DESCRIPTION	2
SPECIFICATIONS	2
OPERATION PROCEDURE	4
1 Setup	4
2 Temperature	5
3 Salinity	5
4 Conductance	5
5 Error	6
CIRCUIT DESCRIPTION MAINTENANCE AND CALIBRATION	9
1 Description	9
2 Maintenance	9
3 Calibration	9
PROBE	12
1 Description of YSI 3300 Conductivity/Temperature Probe	12
2 Maintenance	12
3 Probe Use	14
4 Cell Calibration & Standard Solutions	14
YSI MODEL 33 AND 33M USED WITH YSI 51A 54 AND 57 OXYGEN METERS	16
WARRANTY	21

GENERAL DESCRIPTION

The YSI Model 33 and 33M S-C-T Meters are portable, battery powered, transistorized instruments designed to accurately measure salinity, conductivity and temperature. They use a probe consisting of a rugged, plastic conductivity cell and a precision YSI thermistor temperature sensor combined in a single unit.

Conductivity with the Model 33 is expressed as micromhos/centimeter (μmhos/cm), with the 33M, it's millisiemens/meter (mS/m). These are measurements of the electrical conductance the sample would show if measured between opposite faces of a 1cm cube. (Conversion information: 1 μmho/cm = 0.1 mS/m.) Salinity is the number of grams of salt/kilogram of sample (‰ = parts per thousand). This measurement assumes the sample contains a "standard" sea water salt mixture. The sample temperature is measured in degrees Celsius.

Salinity measurements are manually temperature compensated by direct dial. Conductivity measurements are not temperature compensated, however, a temperature function is provided on the instrument to aid with calculation of corrections. Also, when just temperature and conductivity are known it is possible to calculate salinity, and when only temperature and salinity are known it is possible to calculate conductivity.

SPECIFICATIONS

Model 33 Conductivity

Ranges	0-500, 0-5,000, 0-50,000 μmhos/cm with YSI 3300 Series Probes (Note: The "μmho" designations on the meter are a shorthand form for "μmho/cm".)
Accuracy	±2.5% max. error at 500, 5,000 and 50,000 plus probe ±3.0% max. error at 250, 2,500 and 25,000 plus probe See Error Section

Readability	2.5 μmhos/cm on 500 μmho/cm range 25 μmhos/cm on 5,000 μmho/cm range 250 μmhos/cm on 50,000 μmho/cm range
Temperature Compensation	None

Model 33M Conductivity

Ranges	0-50, 0-500, 0-5,000 mS/m with YSI 3300 Series Probes
Accuracy	±2.5% max. error at 50, 500 and 5,000 plus probe ±3.0% max. error at 25, 250 and 2,500 plus probe See Error Section
Readability	0.25 mS/m on 50 mS/m range 2.5 mS/m on 500 mS/m range 25.0 mS/m on 5,000 mS/m range
Temperature Compensation	None

Salinity

Range	0-40‰ in temperature range of -2 to +45°C
Accuracy	Above 4°C: ±0.9‰ at 40‰ and ±0.7‰ at 20‰ plus conductivity probe Below 4°C: ±1.1‰ at 40‰ and ±0.9‰ at 20‰ plus conductivity probe See Error Section
Readability	0.2‰ on 0-40‰ range
Temperature Compensation	Manual by direct dial from -2 to +45°C

Temperature	
Range	-2 to +50°C
Accuracy	±0.1°C at -2°C, ±0.6°C at 45°C plus probe See Error Section
Readability	±0.15°C at -2°C to ±0.37°C at 45°C
Power Supply	Two D-size alkaline batteries. Eveready E95 or equivalent provide approximately 200 hrs. of operation
Probe	YSI 3300 Series Conductivity/Temperature Probe Nominal Probe Constant K = 5/cm
Accuracy	±2% of reading for conductivity and salinity Error of ±0.1°C at 0°C and ±0.3°C at 40°C
Instrument	
Ambient Range	Satisfactory operation -5 to +45°C. A maximum error of ±0.1% of the reading per °C change in instrument temperature can occur. This error is negligible if the instrument is readjusted to redline for each reading

OPERATION PROCEDURE

1. **Setup**
   (a) Adjust meter zero (if necessary) by turning the bakelite screw on the meter face so that the meter needle coincides with the zero on the conductivity scale.
   (b) Calibrate the meter by turning the MODE control to REDLINE and adjusting the REDLINE control so the meter

needle lines up with the redline on the meter face. If this cannot be accomplished, replace the batteries.
   (c) Plug the probe into the probe jack on the side of the instrument.
   (d) Put the probe in the solution to be measured. (See Probe Use.)

2. **Temperature**
   Set the MODE control to TEMPERATURE. Read the temperature on the bottom scale of the meter in degrees Celsius. Allow time for the probe temperature to come to equilibrium with that of the water before reading.

3. **Salinity**
   (a) Transfer the temperature reading from Step 2 to the °C scale on the instrument.
   (b) Switch the MODE control to the SALINITY position and read salinity on the red 0-40 ‰ meter range.
   (c) Depress the CELL TEST button. The meter reading should fall less than 2%. If greater, the probe is fouled and the measurement is in error. Clean the probe and re-measure.

4. **Conductivity on Model 33** (Model 33M data are in parentheses.)
   (a) Switch the MODE control to the X100 scale. If the reading is below 50 on the 0-500 range (5.0 on the 0-50 range) switch to the X10 scale. If the reading is still below 50 (5.0), switch to the X1 scale. Read the meter scale and multiply the reading appropriately. The answer is expressed in µmhos/cm (mS/m). Measurements are not temperature compensated.

Example	Meter Reading	247 (24.7)
	Scale	X10
	Answer	2470 µmhos/cm (247.0 mS/m)

(b) When measuring on the X100 and X10 scales, depress the CELL TEST button. The meter reading should fall less than 2%. If greater, the probe is fouled and the measurement is in error. Clean the probe and re measure.

NOTE: The CELL TEST does not function on the X1 scale.

5. Error

The maximum error in a reading can be calculated by using the graphs in the following sections.

(1) Temperature

The temperature scale is designed to give the minimum salinity error when the temperature readings are used to compensate salinity measurements.

Figure 1 shows total error for probe and instrument versus °C meter reading.

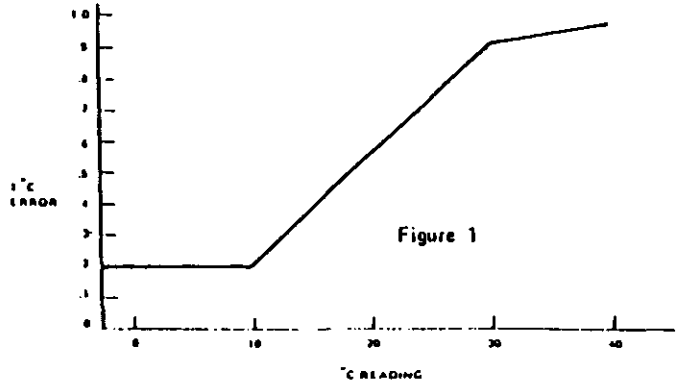


Figure 1

Example: Meter Reading 15°C

Total Error 0.4°C

Accuracy 15°C ± 0.4°C for probe and instrument combined

(2) Conductivity on Model 33 (Model 33M data are in parentheses.)

Figure 2 shows the worst case conductivity error as a function of the conductivity reading for the probe and instrument combined.

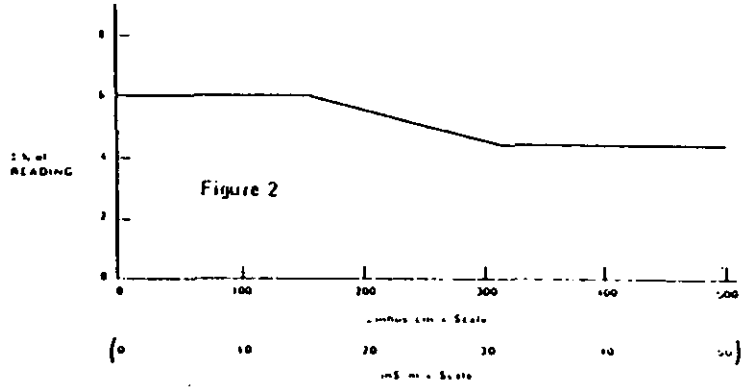


Figure 2

Example: Meter Reading 360 µmhos/cm (36 mS/m)

Scale X10

% Reading Error ± 4.5%

Accuracy 3600 ± 162 µmhos/cm (360 ± 16.2 mS/m) for probe and instrument

(3) Salinity

The salinity readings are a function of temperature and conductivity, therefore the accuracy is a function of both. The temperature scale and temperature control have been designed to minimize the temperature error contribution to the salinity error. The error shown in Figure 3 is the total of the temperature and conductivity probe, the temperature scale and the salinity scale error.

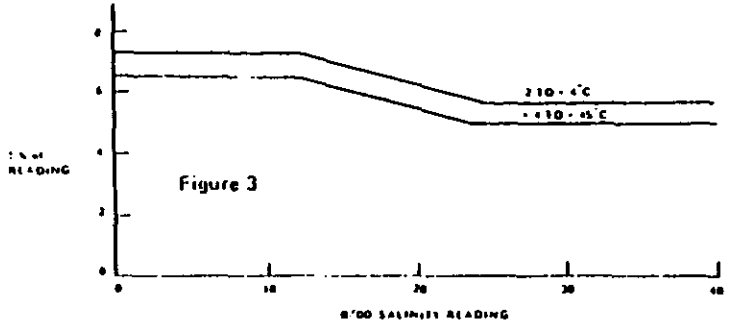
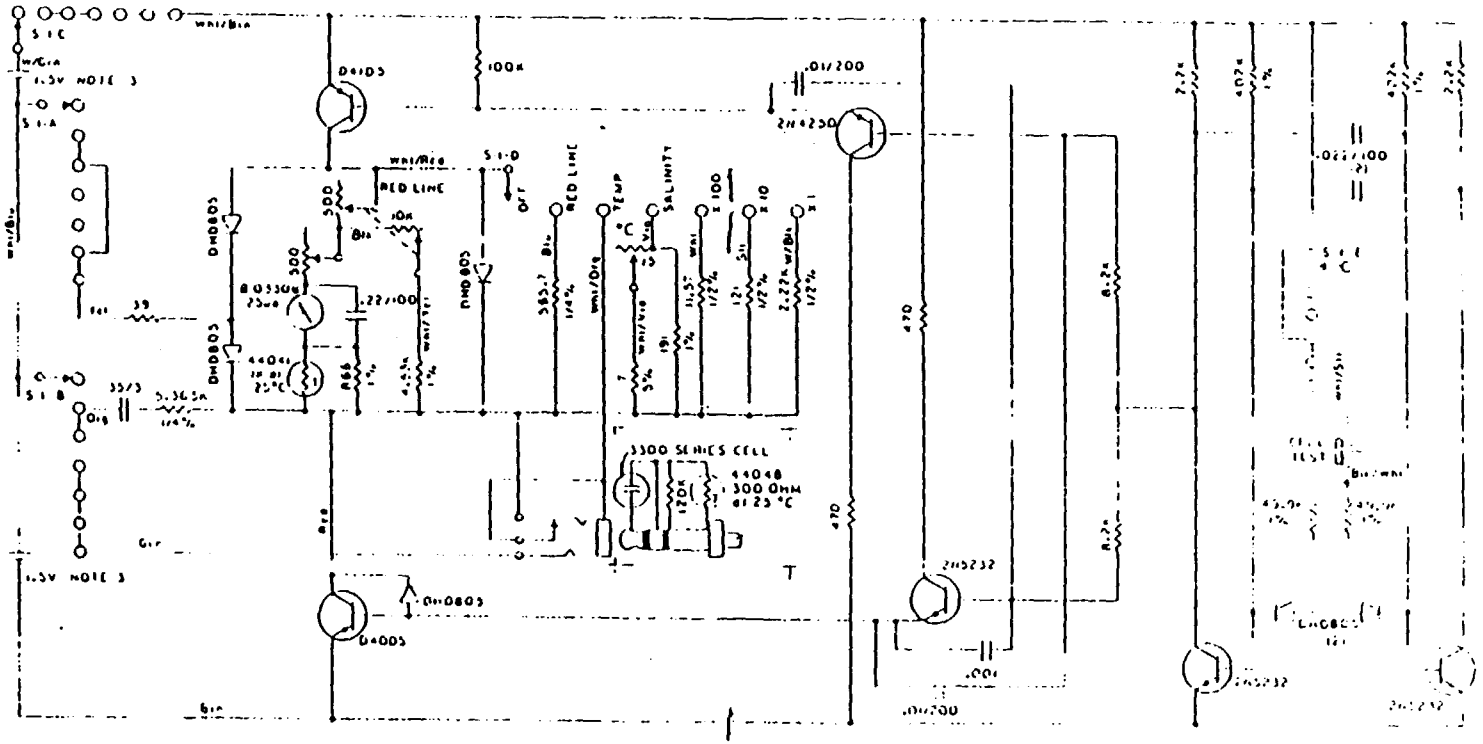


Figure 3

Example: Meter Reading. 10 0/00. @ 10°C

% of Reading Error	6 5%
Accuracy	10 ‰ ± 0 65 ‰ for all errors, combined worst case.



YSI MODEL 33 AND 33M B 03321-E

NOTE

1. Resistance values in ohms ± 1.000 resistors are ±0.10% unless otherwise specified.
2. The values shown on the schematic may differ from those in the instrument, if so other value can be used for replacement purposes.
3. Battery is "D" size alkaline unit. Exceeds 1.5 ohm equal.

CIRCUIT DESCRIPTION, MAINTENANCE AND CALIBRATION

1. Description

The circuit is composed of two parts, a multivibrator and switching transistors. The multivibrator produces a square waveform voltage. The square wave is applied to two switching transistors. They alternately apply two batteries of opposite polarity to the probe thus providing AC power which minimizes polarization effects. The meter is in series with one battery and measures the current from it. The current from the battery is proportional to the conductance of the cell. Salinity is measured in a special range conductivity circuit which includes a user-adjusted temperature compensator. In the temperature redline and X1 positions the multivibrator operates at 100 Hz. In the salinity, X100 and X10 positions the multivibrator operates at 600 Hz and in these ranges pushing the CELL TEST button drops the frequency to 100 Hz allowing the operator to judge the degree of probe polarization.

2. Maintenance

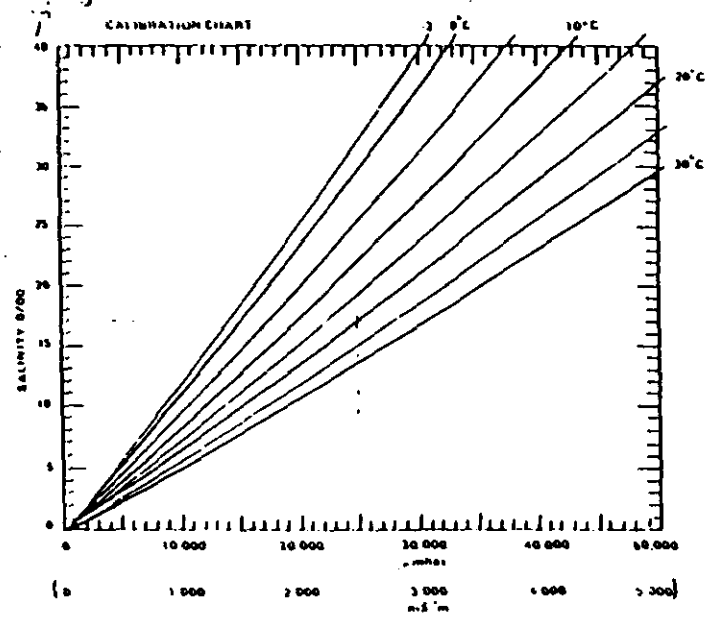
The only maintenance required is battery replacement. Two "D" size alkaline flashlight cells, such as Eveready E95 or equivalent, will provide 200 hrs. of operation. Accuracy will not be maintained if zinc-carbon "D" cells are used. Battery replacement is indicated when the redline adjustment cannot be accomplished.

Replace batteries every six months to reduce the danger of corrosion due to leaky batteries. To replace batteries, remove the six screws from the rear plate. The battery holders are color coded. The Positive (+ button) end must go on red.

3. Calibration of Model 33 (Model 33M data are in parentheses.)

It is possible for the temperature knob to become loose or slip from its normal position. In an emergency the dial can be re-positioned. It must be emphasized that this is an emergency procedure only, and that the instrument should be returned to the factory for proper recalibration at the earliest opportunity.

(a) Read the temperature and conductivity of the solution. Determine the salinity of the solution by running a line vertically on the graph from this conductance value until it intersects the appropriate °C line (interpolate as required for temperature between the given °C lines). From this intersection extend a



line horizontally to the edge of the graph. This determines the salinity for this sample.
Example: 25,000 µmhos/cm and 20°C gives a salinity of 17
(Example: 2,500 mS/m and 20°C gives a salinity of 17.)

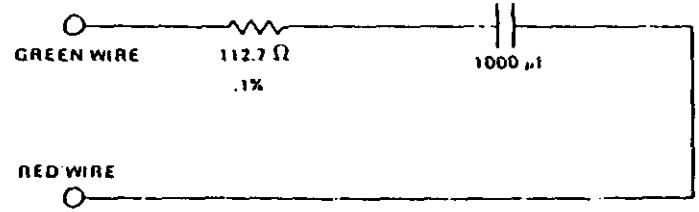
(b) Remove the °C knob, switch to SALINITY, and turn the control shaft until the meter needle indicates the salinity value determined in Step (a). In the example given, the value is 17.

(c) Switch to TEMPERATURE. (Note: This temperature reading must be the same as Step (a), if not, begin again at Step (a).) Place the knob on the control shaft (without turning the control shaft) with the knob pointer at the same temperature as the meter reading and tighten both set screws securely.

At earliest opportunity recalibrate using the following procedure or return the instrument to factory for service.

(a) Set the instrument for a salinity measurement as normal.
(b) Substitute a 1000 µf capacitor and 112.7 ohm 0.1% tolerance resistor for the probe.

Connect the resistor and capacitor between the green wire and red wire on the jack connections inside the instrument.



(c) Turn the temperature dial until the meter reads redline

Now install the temperature knob with the arrow at 25°C This is a temporary calibration only Return the instrument to the factory for proper recalibration

PROBE

1 Description of YSI 3300 Series Conductivity/Temperature Probe

The YSI 3300 Series Conductivity Probes are designed for field use, embodying construction and design for rugged, accurate service

Each probe features a built-in cell constant of 5 0 (500 0/M) ±2%, a precision YSI thermistor temperature sensor of ±0 1°C accuracy at 0°C and ±0 3°C at 40°C and a low capacitance cable assembly terminating in a three thermnal 0 25" dia phone type connector.

The 3310 has a 10 ft cable and the 3311 is a 50 ft version Other lengths are available on special order

The probe has a rigid P V C body platinized pure nickel electrodes, and a durable cable providing resistance to a wide range of water-borne substances

2 Maintenance

(a) Cleaning

When the cell test indicates low readings the probable cause is dirty electrodes Hard water deposits oils and organic matter are the most likely contaminants

For convenient normal cleaning soak the electrodes for 5 minutes with a locally available bathroom tile cleaning preparation such as Dow Chemical Bathroom Cleaner Horizon Industries Rally Tile, Porcelain and Chrome Cleaner Johnson Wax Envy Instant Cleaner or Lysol Brand Basin Tub Tile Cleaner

For stronger cleaning a 5 minute soak in a solution made of 10 parts distilled water, 10 parts isopropyl alcohol and 1 part HCl can be used

Always rinse the probe after cleaning and before storage

CAUTION Do not touch the electrodes inside the probe Platinum black is soft and can be scraped off

If cleaning does not restore the probe performance, re platinizing is required.

(b) Re Platinizing

Equipment Required —

(1) YSI #3140 Platinizing Solution, 2 fl oz (3% platinum chloride dissolved in 0 025% lead acetate solution)

(2) YSI Model 33 or 33M S-C-T Meter

(3) 50 ml glass breaker or equivalent bottle

(4) Distilled water

Procedure —

(1) Clean the probe as in Section (a) — either method

(2) Place the cell in the beaker and add sufficient YSI #3140 solution to cover the electrodes Do not cover the top of the probe

(3) Plug the probe into the Model 33 or 33M, switch to the X100 scale to platinize the electrode Move the probe slightly to obtain the highest meter reading and continue platinizing for the approximate time shown below

Meter Reading		Time
µmhos/cm	mS/m	(minutes)
30,000	3,000	5
25,000	2,500	6
20,000	2,000	8
15,000	1,500	11
10,000	1,000	16

(4) After the elapsed time remove the probe and rinse in fresh water.

(5) Return the solution to its container. 2 oz. of solution should be sufficient for 50 treatments.

3. Probe Use

(a) Obstructions near the probe can disturb readings. At least two inches of clearance must be allowed from non-metallic underwater objects. Metallic objects such as piers or weights should be kept at least 6 inches from the probe.

(b) Weights are attached to the cable of the YSI 3310 and 3311 Probes. The YSI 3327 Weights are supplied in pairs with a total weight of 4 ounces per pair. Should it become necessary to add more weight to overcome water currents, we suggest *limiting the total weight to two pounds (8 pairs). For weights* in excess of two pounds use an independent suspension cable. In either case, weights must be kept at least 6 inches away from the probe.

(c) Gentle agitation by raising and lowering the probe several *times during a measurement insures flow of specimen solu*tion through the probe and improves the time response of the temperature sensor.

4. Cell Calibration & Standard Solutions

The YSI #3300 Series Cells are calibrated to absolute accuracy of ± 1.5% based on a standard solution. Since the literature on conductivity does not indicate a consistently accepted standardization method, we have chosen the 0.01 demal KCl solution method as determined by Jones and Bradshaw in 1937 as our standard. Recent textbooks, as well as the ASTM standards, concur with this choice. The solution is prepared by diluting 0.745 grams of pure dry KCl with distilled water until the solution is 1 kilogram. The table below shows the values of conductivity this solution would have if the distilled water were non-conductive. However, since even high purity distilled water is slightly conductive, the measured conductivity will be higher by an amount equal to the water's conductivity.

Temperature °C	Conductivity µmhos/cm	Conductivity mS/m
15	1141.5	114.2
16	1167.5	116.8
17	1193.6	119.4
18	1219.9	122.0
19	1246.4	124.6
20	1273.0	127.3
21	1299.7	130.0
22	1326.6	132.7
23	1353.6	135.4
24	1380.8	138.1
25	1408.1	140.8
26	1436.5	143.7
27	1463.2	146.3
28	1490.9	149.1
29	1518.7	151.9
30	1546.7	154.7

The operator may use the standard solution and the table to check accuracy of a cell's constant or to determine an unknown constant. The formula is shown below.

$$K = \frac{R(C_1 + C_2)}{10^6} \quad \text{or} \quad \frac{R(S_1 + S_2)}{10^3}$$

where: K = Cell constant

R = Measured resistance in Ω

C_1 = Conductivity in µmhos/cm

C_2 = Conductivity in µmhos/cm of the distilled water used to make solution

S_1 = Conductivity in mS/m
S_2 = Conductivity in mS/m of the distilled water used to make the solution

B, C_1 and C_2, or S_1 and S_2, must either be determined at the same temperature or corrected to the same temperature to make the equation valid

Note: For further information on conductivity and the above standard information, refer to ASTM Standards Part 23 — Standard Methods of Test for Electrical Conductivity, or Water and Industrial Waste Water — ASTM Designation D1125 64

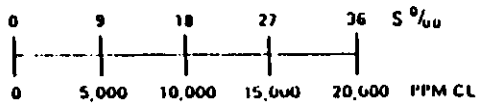
YSI MODEL 33 AND 33M USED WITH YSI 51A, 54 and 57 OXYGEN METERS

If the salinity measurement is to be used for salinity correction on the 51A the reading should be converted to Chlorosity The formula is

$$\text{PPM Chlorosity} = \frac{\text{Salinity } ‰ - 0.03}{1.8} \times 10^3$$

For these instruments the 0.03 can be neglected so the equation simplifies to

$$\text{PPM Cl} = \frac{\text{SS } ‰ \times 10^3}{1.8}$$



For salinity correction when using the Model 57 use the salinity reading direct from the Model 33 or 33M No conversion is necessary

Model 33 and 33M salinity readings taken in conjunction with Model 54 dissolved oxygen readings can be used to correct the Model 54 for salinity and to make post-measurement salinity corrections to dissolved oxygen data Correction tables are available from the factory

WARRANTY

All YSI products carry a one year unconditional warranty on workmanship and parts exclusive of batteries Damage through accident, misuse, or tampering will be repaired at a nominal charge

If you are experiencing difficulty with any YSI product, it may be returned to an authorized YSI dealer for repair, even if the warranty has expired If you need factory assistance for any reason contact

Service Department
Yellow Springs Instrument Co , Inc
P O Box 279
Yellow Springs, Ohio U S A
Phone (513) 767-7241

APPENDIX C-5

Calibration and Maintenance of Photovac TIP

APPENDIX C-5

CALIBRATION AND MAINTENANCE OF PHOTOVAC TIP

CALIBRATION

Two basic calibration operations must be performed. These are adjustment of zero and the adjustment of span.

The zero adjustment is the easiest. Under conditions where accuracy is very important and sensitivity is less important, it may be sufficient to zero the instrument using outdoor air. In other cases, office area or indoor may prove to be clean enough for zeroing purposes. When rigorously done, a source of "Zero Air" or "Ultra Zero Air" is necessary. These are high purity grades of compressed air available in bottled form. The bottle is fitted with a regulator and can be connected directly to the TIP's input fitting. A very low rate of flow should be used with pressure applied never exceeding 1 psi (6 kPa.).

Having adjusted zero (this is best done with the "Span" control at maximum), we will now turn to span calibration. To assess a situation where there is a high ionizable loading in the air consisting of a mixture of many components, it must be recognized that any reading obtained will be a composite of the various components. With photoionization, response factors vary greatly from compound to compound. This makes the reading on TIP dependent upon both concentration and nature of the mixture involved. TIP, in this case, works as a scoping tool; the user can move around the contaminated area seeking "hot spots".

HNu calibration gas (benzene) will be used to calibrate the TIP. The TIP probe will be inserted into the gas cylinder feeder base and the gas released. The TIP will then be adjusted to the gas concentration (generally 52 ppm).

MAINTENANCE

Routine maintenance requirements for TIP are minimal. All that is required is to assure the batteries remain close to full charge (during periods of non-use) and to assure that the inlet frit-filter is kept clear of debris.

The frit is a sintered, stainless type and must be periodused to calibrate the TIP. The TIP probe will be inserted into the gas cylinder feeder base and the gas released. The TIP will then be adjusted to the gas concentration (generally 52 ppm).

The frit is a sintered, stainless type and must be periodically replaced to assure free-flow of air to the detector.

Replacement of the frit is indicated when the inlet flow falls below 140 mL/min.

Referring to the TIP pictorial diagram, remove the four cover mounting screws holding the detector cover in place. Remove any inlet probe that may have been installed. Make sure the unit is switched "Off". Lift the detector cover straight off the front of TIP with a twisting motion to overcome friction against the seal. Take the cover and place it upside down against a soft, but firm, surface such as a block of wood, so that the inlet fitting will not be damaged. From the inside of the fitting, press out the filter with a tool such as a 1/16th inch hex screwdriver.

Turn the cover right side up and position the new filter squarely in the inlet fitting. Press it into position with the same tool.

Make sure that the black PID seal is in place in its recess in the TIP detector and slip the detector cover into position, twisting it over the seal. Replace the four cover mounting screws.

Further maintenance operations that can be performed by the user involve the cleaning of the ion chamber and the lamp window, replacement of the lamp and replacement of the battery pack.

The ion chamber is reached by removing the detector cover (as previously described), unplugging the yellow collector wire from the printed circuit board of the UHF driver, releasing the red repeller wire at its attachment point on the PID (loosen the small screw and pull gently free) and finally, unscrewing the PID from the lamp holder by grasping gently but firmly the body

of the PID and rotating counterclockwise. The lamp will pop up on a spring and may be lifted out for cleaning/replacement. The interior of the ion chamber contains a very delicate wire mesh and must not be touched with any solid object. The lamp window may be cleaned with a cotton swab dipped in methanol and the interior of the ion chamber may be blown free of dust using a gentle compressed air jet. The lamp (or its replacement) is simply put back into the lamp holder and the PID screwed back into place being very careful to avoid "cross threading". The two wires are replaced as before. It is vital to assure that the PID seal is replaced in its seat before putting the cover back onto the detector.

If the pump and LEDs stay off for longer than a minute or so on a fully charged TIP, the detector lamp driving circuit may need adjustment. Remove the detector cover after which the pump and LEDs should come on as a result of ambient light hitting the photo resistor on the exposed circuit UHF driver circuit board.

Locate the ceramic trimming capacitor on the UHF driver; it has a screwdriver adjustment slot on the top of it. Be sure TIP is switched off, and make a pencil mark on the trimmer capacitor to indicate its original position. Turn the trimmer adjustment slightly (five degrees or so) in one direction or the other, then replace the detector cover and turn TIP "On". Repeat this procedure until the lamp starts. After TIP has run for two minutes or so, turn it off and set the trimmer back to its original position, or very near it, replace the detector cover, and use TIP.

If the lamp will not start regardless of the trimmer capacitor setting, the lamp likely needs replacing. Set the trimmer to its original position and replace the lamp as previously described. Lamp replacement is also indicated if, with fully charged batteries, TIP response drops drastically from one day to the next. Normally, a slight ozone smell will be present at the TIP vent. A failed lamp will not produce ozone.

[cac-65-18]

APPENDIX C-6

Calibration and Maintenance of HNu Photoionizer

APPENDIX C-6

CALIBRATION AND MAINTENANCE
OF HNU PHOTOIONIZATION DETECTOR

(Extracted from Manufacturer's Instruction Manual)

To zero the instrument, turn the function switch to the standby position and rotate the zero photentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counterclockwise rotation yields a downscale deflection. Note: no zero gas is needed, since this is an electronic zero adjustment (see below). If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted if necessary. Wait 15 or 20 seconds to ensure that the zero reading is stable. If necessary, readjust the zero.

The instrument is now ready for calibration or measurement by switching the function switch to the proper measurement range. The instrument is supplied calibrated to read directly in ppm (v/v) 0-20, 0-200, 0-2000 of benzene with the span position set at 9.8. For additional sensitivity, the span potentiometer is turned counterclockwise (smaller numbers) to increase the gain. By changing the span setting from 10.0 to 1.0, the sensitivity is increased approximately ten fold. Then, the 0-20, 0-200, 0-2000 ppm scales become 0-2, 0-20, and 0-200 ppm full scale, respectively.

The span control is also utilized to make the instrument scale read directly in ppm of the compound being measured. E.G., it is adjusted to match the value of a calibration gas to that same reading on the instrument scale. The span control can be utilized to calibrate nearly any compound measured by photoionization to be direct reading on the 0-20 ppm range. For example, gain settings of 4.5 or 8.9, respectively, will provide direct reading capability (0-20, 0-200 ppm) for vinyl chloride and trichloroethylene, respectively.

Benzene will be used for calibration. Place the HNU probe top into the calibration gas cylinder discharge hose and open the gas valve. Place the scale selector on 0-200 and adjust to the calibration gas concentration (generally 52 ppm) by turning the potentiometer. The instrument is now calibrated. Recheck and adjust the zero if necessary.

APPENDIX C-7

Calibration and Maintenance of
Organic Vapor Analyzer (OVA)

Extracted from Manufacturers
Instruction Manual

damaging the batteries. When finished, move the battery charger switch to OFF and disconnect from the Side Pack Assembly.

The following are special instructions relative to batteries which have been allowed to completely discharge.

It has been established that the above battery recharging procedures may not be sufficient when the operator of the instrument has inadvertently left the INSTR Switch ON for a period of time without recharging and allowed the battery to completely discharge.

When this happens and the above procedures fail to recharge the battery, the following should be accomplished:

1) Remove the battery from the instrument case.
2) Connect to any variable DC power supply.
3) Apply 40 volts at 1/2 amp maximum.
4) Observe the meter on the power supply frequently and as soon as the battery begins to draw current, reduce the voltage on the power supply at a slow rate until the meter reads approximately 15 volts. NOTE: The time required to reach the 15 volt reading will depend on degree of discharge.
5) Repeat steps a), b), c), and d) above to continue charging.

2.7.2 DC CHARGER

a) The optional DC charger is designed to both charge the battery and to provide power for operating the instrument from a 12 volt DC source, such as vehicle power.
b) Connect the DC charger cord to the connector on the battery cover of the Side Pack Assembly. Plug the line cord into the vehicle cigarette lighter or other power source connection.
c) In mobile applications, the DC charger is used to supply vehicle power to the instrument. Therefore, it may be left connected at all times.

2.8 CHARCOAL FILTERING

When it is desired to preferentially remove the heavier hydrocarbons, such as those associated with automobile exhaust, gasoline, etc., simply remove the pickup fixture from the end of the probe and install the optional charcoal filter assembly.

This same charcoal filter assembly can be installed directly into the Readout Assembly by using the adapter provided.

2.9 MOISTURE FILTERING

Filtering of moisture in the sample is not normally required. However, when moving in and out of buildings in cold weather, excessive condensation can form in the lines and detector chamber. In this case, the charcoal filter adapter can be filled with a desiccant such as "Drierite" which will filter out the moisture contained in the sample.

SECTION 3

SUMMARIZED OPERATING PROCEDURES

3.1 GENERAL

The procedures presented in this section are intended for use by personnel generally familiar with the operation of the instrument. Section 2 presents the comprehensive detailed operating procedures.

It is assumed that, prior to start up the positions of all switches and valves are in shut down configuration as described in paragraph 3.3.

3.2 START UP

a) Move PUMP Switch to ON and check battery condition by moving the INSTR Switch to the BATT position.
b) Move INSTR Switch to ON and allow five (5) minutes for warm-up.
c) Set Alarm Level Adjust Knob on back of Readout Assembly to desired level.
d) Set CALIBRATE Switch to X10 position, use CALIBRATE Knob and set meter to read 0.
e) Move PUMP Switch to ON position then place instrument panel in vertical position and check SAMPLE FLOW RATE indication.
f) Open the H2 TANK VALVE and the H2 SUPPLY VALVE.
g) Depress Igniter Button until burner lights. Do not depress Igniter Button for more than six (6) seconds. (If burner does not ignite, let instrument run for several minutes and again attempt ignition.)
h) Use CALIBRATE Knob to "zero" out ambient background. For maximum sensitivity below 10 ppm, set CALIBRATE Switch to X1 and readjust zero on meter. To avoid false flame-out alarm indication, set meter to 1 ppm with CALIBRATE Knob and make differential readings from there.

3.3 SHUT DOWN

a) Close the H2 SUPPLY VALVE and the H2 TANK VALVE.
b) Move the INSTR Switch and PUMP Switch to OFF.
c) Instrument is now in shut down configuration.

SECTION 4

CALIBRATION

4.1 GENERAL

The OVA is capable of responding to nearly all organic compounds. For precise analyses it will be necessary to calibrate the instrument with the specific compound of interest. This is especially true for materials containing elements other than carbon and hydrogen.

The instrument is factory calibrated to a methane in air standard. However, it can be easily and rapidly calibrated to a variety of organic compounds. A GAS SELECT control is incorporated on the instrument panel which is used to set the electronic gain to a particular organic compound.

Internal electronic adjustments are provided to calibrate and align the electronic circuits. There are four (4) such adjustments all located on the electronics board. One adjustment potentiometer, R-38, is used to set the power supply voltage and is a one-time factory adjustment. The remaining three adjustments, R-31, R-32 and R-33 are used for setting the electronic amplifier gain for each of the three (3) calibrate ranges. Access to the adjustments is accomplished by removing the instrument from its case. Figure 4-1 indicates the location of the adjustments.

4.2 ELECTRONIC ADJUSTMENTS

Primary calibration of this instrument is accomplished at the factory using methane in air sample gases.

4.2.1 GAIN ADJUSTMENT

a) Place instrument in normal operation with CALIBRATE Switch set to X10 and GAS SELECT control set to 300.
b) Use the CALIBRATE ADJUST (zero) Knob and adjust the meter reading to zero.
c) Introduce a methane sample of a known concentration (near 100 ppm) and adjust trimpot R-32 on circuit board (see Figure 4-1 for location) so that meter reads equivalent to the known sample.
d) This sets the instrument gain for methane with the panel mounted gain adjustment (GAS SELECT) set at a reference number of 300.
e) Turn off H2 SUPPLY VALVE to put out flame.

4.2.2 BIAS ADJUSTMENT

a) Leave CALIBRATE Switch on X10 position and use CALIBRATE ADJUST (zero) Knob to adjust meter reading to 4 ppm.
b) Place CALIBRATE Switch in X1 position and, using trimpot R-31 on circuit board, adjust meter reading to 4 ppm. (See Figure 4-1)
c) Move CALIBRATE Switch to X10 position again. Use CALIBRATE ADJUST (zero) Knob to adjust meter to a reading of 40 ppm.
d) Move CALIBRATE Switch to X100 position and use trimpot R-33 on circuit board to adjust meter reading to 40 ppm.
e) Move CALIBRATE Switch to X10 position and use CALIBRATE ADJUST (zero) Knob to adjust meter reading to zero.
f) Unit is now balanced from range to range, calibrated to methane, and ready to be placed in normal service.

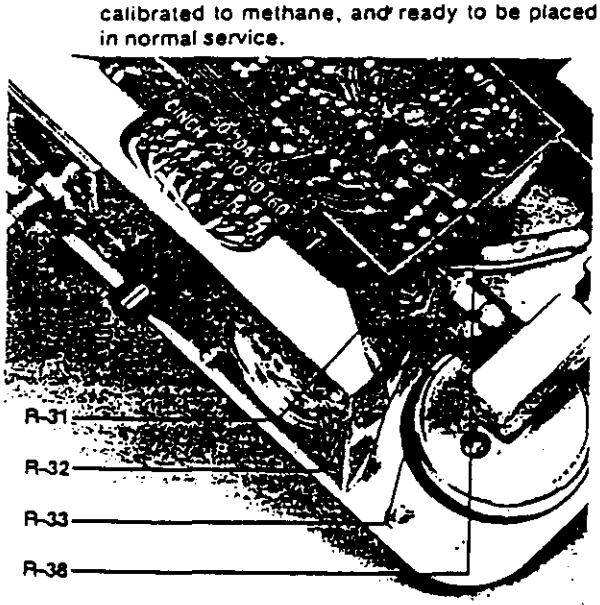


FIGURE 4-1. LOCATION OF ELECTRONIC ADJUSTMENTS
(Model OVA-118 shown; location typical to OVA-128)

4.3 CALIBRATION TO OTHER ORGANIC VAPORS

4.3.1 SETTING GAS SELECT CONTROL (Span)

Primary calibration of the instrument is accomplished using a known mixture of a specific organic vapor compound. After the instrument is in operation and the "normal background" is "zeroed out", draw a sample of the calibration gas into the instrument. The GAS SELECT Knob on the panel is then used to shift the readout meter indication to correspond to the concentration of the calibration gas mixture.

The instrument is then calibrated for the vapor mixture being used. After this adjustment, the setting on the "digidial" is read and recorded for that particular organic vapor compound. This exercise can be performed for a large variety of compounds and when desiring to read a particular compound the GAS SELECT control is turned to the predetermined setting for the compound. Calibration on any one range automatically calibrates the other two ranges.

4.3.2 USING EMPIRICAL DATA

Relative response data may be obtained, which can then be used to estimate concentrations of various vapors. With the instrument calibrated to methane, obtain the concentration reading for a calibration sample of the test vapor. The relative response, in percent, for that test vapor would then be the concentration read/concentration of the calibrated sample X 100.

4.3.3 PREPARATION OF CALIBRATION STANDARDS

4.3.3.1 COMMERCIAL SAMPLES

Commercially available standard samples offer the most convenient and reliable calibration standards and are recommended for the most precise analyses. Always remember to obtain the cylinder with the desired sample and the "balance as air". Sample should be drawn from the cylinder into a collapsed sample bag, then drawn from the bag by the instrument to prevent a pressure or vacuum at the sample inlet.

4.3.3.2 PURE GASEOUS SAMPLES

Obtain a large collapsible sample bag, preferably polyethylene such as a 40 gallon trash can liner. Insert a tube into the bag opening and tie shut around the tube. The tubing should have a shut-off valve or plug and be suitable for connecting the OVA input tube. Determine the volume of the bag by appropriate means (i.e., wet-test meter, dimensions of the bag, etc.). Forty gallon polyethylene bags provide a volume of approximately 140-160 liters. For gas samples, flush a 10 cc hypodermic syringe with the compound to be tested and then inject a 10 cc sample through the wall of the air-filled bag. Immediately after withdrawing the needle, cover the hole with a piece of plastic tape. Allow a few minutes for the sample to completely diffuse throughout the bag. Agitation will ensure complete diffusion. Connect the outlet tube to the OVA and take a reading. To verify repeatability of sampling technique, disconnect the bag and inject a second sample of the gas into the bag without emptying. Since only 2 or 3 liters will have been removed, the overall volume change will be small and the instrument reading should now be twice that of the

original. The concentration in ppm (V/V) will be equal to e sample size in cc divided by the volume of the bag in ters times 1000. For example, a 10 cc gas sample when laced in a 160 liter bag will provide a sample of 63 ppm, i.e., 10 X 1000/160 equals 63 ppm.

.3.3.3 GASEOUS AND LIQUID SAMPLES (Alternate Method)

Obtain a five (5) gallon glass bottle and determine its volume by measuring the volume of water needed to fill t (use of a 1000 ml graduated cylinder, obtainable from scientific supply houses, is convenient). Another approach is to weigh the empty bottle, fill it with water and weigh again. The difference between the two values is he weight of water. By multiplying the weight of water n pounds by 0.455, you obtain the volume of the bottle in liters. Empty the water out and allow the bottle to dry. Place a one-foot piece of plastic tubing in the flask to aid in mixing the vapors uniformly with the air. The volume of such a bottle should be about 20 liters, which is 20,000 ml. If the volume were 20,000 ml, then a 2 ml sample of a gas placed in the bottle would be equivalent to 200 ml per 2 million ml or 100 ppm (V/V). Use of a gas tight syringe, readable in 0.01 ml, allows the preparation of mixtures in the 1 - 2 ppm range, which are sufficient for the quantitative estimation of concentrations. A rubber stopper is loosely fitted to the top of the bottle and the needle of the syringe placed inside the jug neck and the stopper squeezed against the needle to decrease leakage during sample introduction. Inject the sample into the bottle and withdraw the needle without removing the stopper. Put the stopper in tight and shake the bottle for a few minutes with sufficient vigor that the plastic tubing in the bottle moves around to ensure good mixture of the vapors with the air.

For liquid samples, use of the following equation will allow the calculation of the number of microliters of organic liquid needed to be placed into the bottle to make 100 ppm (V/V) of vapor.

V1 equals V2 X Mw/244D

- V1 - Volume of liquid in microliters needed to make an air mixture of 100 ppm (V/V)
- V2 - Volume of bottle in liters
- Mw - Molecular weight of substance
- D - Density of substance

This procedure has the advantage that you can see when all of the organic liquid has vaporized and the volume can be determined readily.

For liquid samples, an alternate procedure involves the use of a diffusion dilution device such as that described by Desty, Geach and Goldup in "Gas Chromatography", R.P.W. Scott, ed., Academic Press, New York, 1961.

4.4 THEORY

Theoretical background and empirical data related to the Century Organic Vapor Analyzer is presented in 4.4.1 and 4.4.2.

4.4.1 HYDROCARBONS

In general, a hydrogen flame ionization detector is more sensitive for hydrocarbons than any other class of organic compounds. The response of the OVA varies from compound to compound, but gives excellent repeatable results with all types of hydrocarbons; i.e., saturated hydrocarbons (alkanes), unsaturated hydrocarbons (alkenes and alkynes) and aromatic hydrocarbons.

The typical relative response of various hydrocarbons to methane is as follows:

Compound	*Relative Response (percent)*
Methane	100 (reference)
Propane	64
N-butane	61
N-pentane	100
Ethylene	85
Acetylene	200
Benzene	150
Toluene	120
Ethane	90

4.4.2 OTHER ORGANIC COMPOUNDS

Compounds containing oxygen, such as alcohols, ethers, aldehydes, carbolic acid and esters give a somewhat lower response than that observed for hydrocarbons. This is particularly noticeable with those compounds having a high ratio of oxygen to carbon such as found in the lower members of each series which have only one, two or three carbons. With compounds containing higher numbers of carbons, the effect of the oxygen is diminished to such an extent that the response is similar to that of the corresponding hydrocarbons.

Nitrogen-containing compounds (i.e., amines, amides and nitriles) respond in a manner similar to that observed for oxygenated materials. Halogenated compounds also show a lower relative response as compared with hydrocarbons. Materials containing no hydrogen, such as carbon tetrachloride, give the lowest response; the presence of hydrogen in the compounds results in higher relative responses. Thus, $CHCl_3$ gives a much higher response than does CCl_4. As in the other cases, when the carbon to halogen ratio is 5:1 or greater, the response will be similar to that observed for simple hydrocarbons.

The typical relative response of various compounds to methane is as follows:

Methane	100 (calibration sample)
Ketones	
Acetone	60
Methyl ethyl ketone	80
Methyl isobutyl ketone	100
Alcohols	
Methyl alcohol	15
Ethyl	25
Isopropyl	65

Halogen compounds

Carbon tetrachloride	10
Chloroform	65
Trichloroethylene	70
Vinyl chloride	35

The OVA has negligible response to carbon monoxide and carbon dioxide which evidently, due to their structure, do not product appreciable ions in the detector flame. Thus, other organic materials may be analyzed in the presence of CO and CO_2.

SECTION 5

SAFETY CONSIDERATIONS

5.1 GENERAL

The Models OVA-108, OVA-128 and OVA-138 have been tested and certified by Factory Mutual Research Corporation (FM) as intrinsically safe for use in Class I, Division 1, Groups A, B, C & D hazardous atmospheres. Similar foreign certifications have been obtained, including BASEEFA and Cerchar approval for Group IIC, Temperature Class T4 on the Models OVA-108, OVA-128 and OVA-138, and equivalent approval from the Japanese Ministry of Labor for the Model OVA-128. Special restrictions must be strictly adhered to, to ensure the certification is not invalidated by actions of operating or service personnel.

All flame ionization hydrocarbon detectors are potentially hazardous since they burn hydrogen (H2) or H2 mixtures in the detector cell. Mixtures of H2 and air are flammable over a wide range of concentrations whether an inert gas such as nitrogen (N2) is present or not. Therefore, the recommended precautions and procedures should be followed for maximum safety. Safety considerations was a major factor in the design of the Organic Vapor Analyzer (OVA).

All connectors are of the permanent type as opposed to quick disconnect. To protect against external ignition of flammable gas mixtures, the flame detection chamber has porous metal flame arrestors on the sample input and the exhaust ports as well as on the H2 inlet connector. The standard battery pack and other circuits are internally current limited to an intrinsically safe level.

5.2 OPERATING, SERVICING AND MODIFYING

It is imperative that operation and service procedures described in this manual be carefully followed in order to maintain the intrinsic safety which is built into the OVA. No modification to the instrument is permissible. Therefore, component replacement must be accomplished with the same type parts.

5.3 ELECTRICAL PROTECTION

The 12V battery power supply circuit is current limited to an intrinsically safe level. Fuses are not utilized and all current limiting resistors and other components which are critical to the safety certification are encapsulated to prevent inadvertent replacement with components of the wrong value or specification. Under no circumstances should the encapsulation be removed.

5.4 FUEL SUPPLY & TANK

The OVA fuel tank has a volume of 75 to 85 cc which, when filled to the maximum rated pressure of 2300 PSIG, holds approximately 5/8 cubic foot of gas. The fuel used in the OVA is pure hydrogen which can be readily purchased in a highly pure form at nominal cost. The H2 tanks used in the instrument are made from stainless steel, proof-tested to 6,000 PSIG and 100% production tested to 4,000 PSIG.

5.5 H2 FLOW RESTRICTORS

Hydrogen gas gains heat when expanding and, therefore, should not be rapidly released from a high pressure tank to a low pressure environment. Flow restrictors are incorporated in the H2 refill fitting and H2 is restricted on the output side of the tank by the low flow rate control system. In addition, a special flow restrictor is incorporated in the FILL/BLEED valve of the hydrogen filling hose assembly. These precautions limit the flow rate of the H2 to prevent ignition due to self-heat from expansion.

5.6 DETECTOR CHAMBER

The OVA has a small flame ionization chamber cavity with sintered metal flame arrestors on both the input and output ports. The chamber is ruggedly constructed of teflon such that even if highly explosive mixtures of H2 and air are inadvertently created in the chamber and ignited, the chamber would NOT rupture.

5.7 H2 FILLING AND EMPTYING OPERATIONS

Precautions should be taken during H2 filling or H2 tank emptying operations to ensure that there are no sources of ignition in the immediate area. Since the instrument tank at 2300 PSIG holds only 5/8 cu. ft. of H2, the total quantity, if released to the atmosphere, would be quickly diluted to a non-flammable level. There is, however, the possibility of generating flammable mixtures in the immediate vicinity of the instrument during the filling or emptying operations if normal care is not exercised.

5.8 VENTING

The OVA case is vented to eliminate the possibility of trapping an explosive mixture of H2 and air inside the case.

SECTION 6

MAINTENANCE

6.1 GENERAL

This section describes the routine maintenance schedule recommended and provides procedures for trouble shooting malfunctions or failures in the instrument.

Appendix "A" to this manual contains the assembly drawings and associated parts list for the Side Pack Assembly and two major subassemblies; the Electronic Component Assembly and the Cylinder Assembly. These drawings and parts lists may be used for locating and identifying components. Also included in Appendix "A" is a schematic wiring diagram showing interconnecting wiring between major electronic assemblies and typical signal levels at selected points on the certified instruments. The enclosed drawings and parts lists are subject to change without notice and part replacement on any certified instrument should be verified to comply with the "no modifications permitted" requirement.

CAUTION

Maintenance personnel should be thoroughly familiar with instrument operation before performing maintenance. It is essential that all portions of this manual relating to safety of operation, servicing and maintenance, including Section 5, be thoroughly understood. There should be no potential igniters or flame in the area when filling, emptying or purging the hydrogen system and the instrument should be turned off.

Extreme care should be exercised to ensure that required parts replacement is accomplished with the same parts specified by Century. This is especially necessary on the Models OVA-108, OVA-128 and OVA-138 in order that their certification for use in hazardous atmospheres be maintained. No modifications are permitted. Disassemble instrument only in a non-hazardous atmosphere.

6.2 ROUTINE MAINTENANCE

Note that Figure 6-1 is a flow diagram of the basic gas handling system.

6.2.1 FILTERS

6.2.1.1 PRIMARY FILTER

This filter is located behind the sample inlet connector (Fitting Assembly) on the Side Pack Assembly and is removed for cleaning by using a thin wall socket to unscrew the Fitting Assembly. The filter cup, "O" ring and loading spring will then come out as shown in the Side Pack Assembly drawing in Appendix "A". The porous stainless filter cup can then be cleaned by blowing out or washing in a solvent. If a solvent is used, care should be taken to ensure that all solvent is removed by blowing out or heating the filter. Reassemble in reverse order ensuring that the "O" ring seal on the Fitting Assembly is intact.

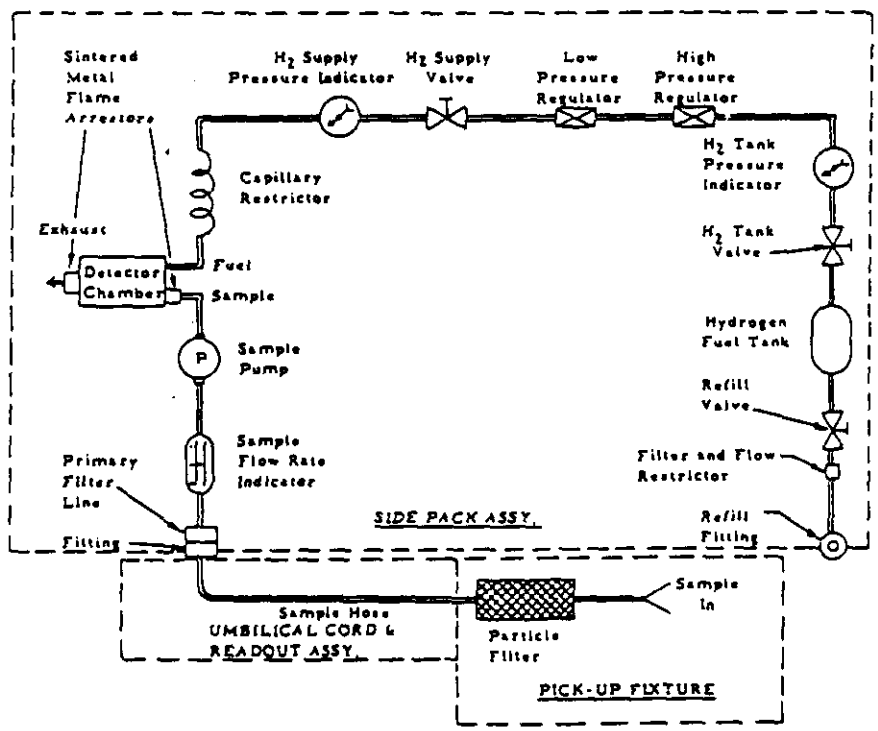


FIGURE 6-1. Flow Diagram - Gas Handling System

6.2.1.2 PARTICLE FILTERS

A particle filter is located in each pickup fixture. One of these filters must be in the sample line whenever the instrument is in use. The Models OVA-88 and OVA-138 use a disposable cellulose filter which should be changed as often as required. The Models OVA-98, OVA-108, OVA-118 and OVA-128 use a porous metal filter which can be replaced or cleaned using the cleaning procedure in paragraph 6.2.1.1.

6.2.1.3 MIXER/BURNER ASSEMBLY FILTER

Another porous metal particle filter is incorporated in the Mixer/Burner Assembly which screws into the Preamp Assembly. See Side Pack Assembly drawing. This filter is used as the sample mixer and inlet flame arrestor in the chamber. This filter should not become contaminated under normal conditions but can be cleaned or the assembly replaced if necessary.

Access to this filter for output surface cleaning is gained by simply unscrewing the exhaust port from the Preamp Assembly without removing the instrument from the case. The OVA-108, OVA-128 and OVA-138 instruments require removal of the safety cover prior to unscrewing the exhaust port. The Filter Assembly can now be seen on the side of the chamber (Preamp Assembly) and can be scrapped or cleaned with a small wire brush.

If filter replacement is required, install a new or factory rebuilt Mixer/Burner Assembly. In several OVA models, this requires removal of the Preamp Assembly.

6.2.1.4 EXHAUST FLAME ARRESTOR

A porous metal flame arrestor is located in the exhaust port of the detector chamber (Preamp Assembly). See Side Pack Assembly drawing. It acts as a particle filter on the chamber output and restricts foreign matter from entering the chamber. This filter may be cleaned, if required, by removing the exhaust port from the Preamp Assembly. The exhaust port is removed from the bottom of the case without case removal. Note that the filter is captive to the exhaust port on the Models OVA-108, OVA-128 and OVA-138. Clean the filter with a solvent or detergent but ensure that it is dry and any solvent completely baked out at 120°F before reinstalling.

6.2.2 PICKUP FIXTURES

The pickup fixtures should be periodically cleaned with an air hose and/or detergent water to eliminate foreign particle matter. If a solvent is used, the fixture should be subsequently cleaned with detergent and baked out at 120°F to eliminate any residual hydrocarbons from the solvent.

6.2.3 SEAL MAINTENANCE - CYLINDER ASSEMBLY

6.2.3.1 H2 TANK, H2 SUPPLY AND REFILL VALVES

After some time, the teflon washers under each valve packing nut can "cold flow" (move with pressure) and allow hydrogen to leak. Leakage can be determined by using Leak-Tec, Snoop or a soap solution around the valve stems. This leakage can usually be stopped by tightening the compression nut (adapter) as outlined below. See Side Pack Assembly and Cylinder Assembly drawings.

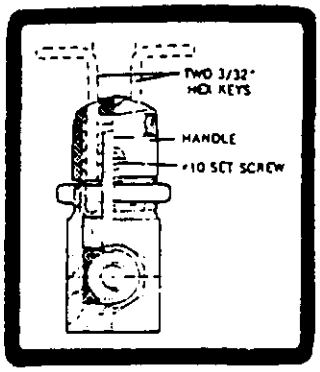
1) Remove instrument from the case by unlocking the four (4) 1/4 turn fasteners on the panel and removing the exhaust safety cover (if included), exhaust port and refill cap nut. Be sure refill valve is closed before removing refill cap nut.
2) Remove the valve knob screw and knob.
3) Loosen the panel nut with a 3/4" wrench.
4) The valve compression nut is located just under the panel. Tighten the compression nut—usually not more than 1/4 turn.

This compression is against soft material and only a small amount of force is necessary to sufficiently compress the teflon washers. If, after tightening, leakage still occurs, it would be advisable to replace the two teflon washers, as follows:

1) Drain hydrogen system slowly and to the extent necessary to work on the leaking valve(s). Observe safety precautions (see Section 5). There should be no potential igniters in the area.
2) Disconnect the capillary tube from the manifold at low pressure gauge (H2 Supply Pressure).
3) Remove all three (3) knob screws and knobs.
4) Remove the three (3) panel nuts and washers.
5) Carefully remove the tank assembly from the panel. NOTE: If OVA has GC Option installed, the GC valve assembly must be loosened or removed in order to remove the tank assembly from the panel.
6) Remove the compression nut on the valve that is not sealing properly. Remove the stem by unscrewing it from the valve body. Observe the sandwich of metal and teflon washers and note their order.
7) Visually check the Kel-F seat on the stem for cracks or foreign material. Wipe clean, if necessary, with a lint free cloth (no solvents or oils) and replace if damaged.
8) Remove the washers and replace the teflon washers (the factory procedure is a light wipe of hydrocarbon free silicone grease).
9) Replace the stem assembly in the valve body and tighten lightly.
10) Push the washers down into the compression area in the same order as noted upon removal. Replace the compression nut and tighten snugly.
11) Close the low pressure valve and fill the tank assembly. Check valves for leaks. Tighten again, if necessary, and reassemble the unit.

6.2.3.2 REFILLER VALVE PACKING ADJUSTMENT

Adjustment for the valve on the refiller can be made by loosening the set screw with a 3/32" hex key, so that the handle turns freely on the stem. Insert two (2) 3/32" hex keys through the holes provided in the handle and turn until they engage the holes in the packing adjuster. Then tighten the packing by turning the handle.



6.2.4 AIR SAMPLING SYSTEM MAINTENANCE

6.2.4.1 GENERAL

A potential problem associated with the OVA instrument is that leaks can develop in the air sample pumping system. These leaks can result in either dilution or loss of sample, causing low reading of vapor concentration and slow response time.

6.2.4.2 TESTING FOR LEAKS

The OVA's a re equipped with a flow gauge, which provides a method to check for air leaks. Assemble the pickup probe selected for use to the readout assembly and then position the sidepack vertically so the flow gauge may be observed. Cover the end of the pickup probe with your finger and observe that the ball in the flow gauge goes to the bottom, indicating no air flow (if ball has slight chatter while on bottom, this is acceptable). Cover the center of the chamber exhaust port with your thumb and again observe the ball going to the bottom. Another simple check is to expose the pickup probe to cigarette smoke or a light vapor (butane) and observe that the meter responds in approximately 1.5 - 2.0 seconds. It should be noted that slow meter response may also indicate a restriction in the air sampling system.

6.2.4.3 LEAK ISOLATION

Failure of the ball to go to the bottom when the inlet is blocked indicates a leak in the system between the probe and the pump inlet or the inlet check valve. To isolate the problem, remove parts, one at a time, and again block off the air inlet. Remove the pickup probe(s) and cover the air inlet at the Readout Assembly. If the ball goes to the bottom, check that the "readout to probe" seal washer is in place and replace the probes, holding them back against this seal while tightening the nut. Recheck, and if leakage is still present, it is probably in the probe (pickup fixture), which should be repaired or replaced.

If leakage is indicated as being past the readout handle when the connection to the sidepack is tight, disconnect the sample line at the fitting on the sidepack and cover this inlet with your finger. If the flow gauge ball goes to the bottom, the problem should be a leak in the umbilical cord/Readout Assembly, which should be investigated and repaired. There is also the possibility of a leaking check valve in the pump which would not show up on this test. If the leakage is not found in the umbilical cord, it is most likely in the pump check valve which should be repaired or replaced.

If the ball does not go to the bottom, the leak will be either in the flow gauge or it's connecting tubing. Visually check that the tubing is connected and if so, the flow gauge should be repaired or replaced. Check the "O" ring installation in the sample inlet connector (Fitting Assembly).

As an alternate approach, leaks on the inlet side of the pump can be detected by using alcohol on a "Q" Tip and lightly swabbing the connections one at a time or by directing organic vapor or smoke at the potential leakage points and observing the meter response or audible alarm.

Leaks (beyond the pump) are easier to locate, as any of the commercially available leak detection solutions can be used. Cover the exhaust port, which will place the exhaust system under pressure, and check each connection, one at a time. Replace the teflon tubing or retape the threaded connections with teflon joint tape. Check the igniter and Mixer/Burner Assembly where they screw into the detector, the high voltage terminal screw on the side of the Mixer/Burner and exhaust port itself. If after these checks, the flow gauge ball still will not go to the bottom with the exhaust blocked, the problem is likely a leaking exhaust check valve in the pump, which should be repaired or replaced.

6.2.5 CONTAMINATION CONTROL AND MAINTENANCE

6.2.5.1 GENERAL

On occasion, the background reading of the OVA may be relatively high under normal ambient conditions. Ambient background readings will vary somewhat depending on the geographical location where the instrument is being used. However, the background reading normally should be in the range of 3 to 5 ppm as methane. The acceptable background reading consists of 1 to 1-1/2 ppm of methane which is present in the normal air environment. In addition to the measurement of a normal methane background, there will normally be 2 to 4 ppm of equivalent methane background caused by acceptable levels of contamination in the hydrogen fuel and/or hydrogen fuel handling system resulting in a total equivalent methane reading of 3 to 5 ppm in clean air.

If the background reading goes above 5 ppm to 6 or 7 ppm, this is normally still acceptable since any measurement is additive to that background reading, i.e., 2 ppm on top of 5 or 2 ppm on top of 7 provides the same differential reading, however, the lower background is obviously desirable.

The background reading on the linear OVA's is zeroed out or nulled out—even though in reality the background still exists. The background reading on the

linear OVA's is measured by zeroing the meter with the flame out and noting the meter indication after the flame is on. However, on the logarithmic scaled OVA's the background reading is observed on the meter at all times. This is considered desirable since it assures the operator that the instrument is, in fact, operating properly. The background reading on the OVA's serves as a low level calibration point since it does represent the measurement of ambient levels of methane in the air, which are extremely stable and predictable any place in the world.

The cause for a high background reading is usually associated with contamination in the hydrogen fuel system. This will, of course, cause a background reading since this is the function of the basic detector "to measure contamination entering the detector chamber". In addition, contamination present in the hydrogen will many times leave a small unobservable deposit on the burner face which can continue to generate a background reading when the detector is in operation and the burner assembly is heated.

Another possible cause of contamination is the mixer/burner assembly when the contamination is trapped in the porous bronze sample filter. This is not a common problem and usually only happens when an unusually high level of contaminant is drawn into the assembly. Another possible cause of high background reading is contamination someplace in the air sample line to the detector. This is also uncommon but can be the source of the problem.

NOTE

OVA's that include the Chromatograph Option installed can also have an indication of high background related to saturation or contamination of the activated charcoal filter, which is in the line during chromatograph analysis, or of the column which is in the hydrogen line at all times.

6.2.5.2 ANALYSIS AND CORRECTION

Prior to analyzing the problem, the OVA should be checked for proper electronic operation. Check logarithmic instruments for proper high and low calibration points and for proper gas selector operation (see Section 4). On logarithmic OVA's, check Gas Selector by turning to 500 and observing the flame-out alarm comes on as the needle goes below 1 ppm. It should be ensured that the instrument is calibrated to methane as referenced.

If, after checking that the OVA is properly calibrated, the background is still higher than normal for ambient conditions, the following procedure should be followed to isolate the cause of the problem.

1) Let the OVA run for a period of time (15 to 30 minutes) and see if the background level decreases as a function of time. The background could go down and stay down as a result of clearing line contamination which is removable simply by the normal flow of air through the sample line.
2) Take a reading in a known, relatively clean air environment. Normally, outside air environment is clean enough to assess by comparison whether the background reading is internal to the instrument or is present in the laboratory, office or location where the instrument is being used.
3) If the OVA includes the Gas Chromatograph Option, depress the sample inject valve so that the activated charcoal is in the line and observe whether the background reading goes down and stays steady after the elution of the air peak. The reading should always go down or stay the same but never be a higher background reading with the sample valve depressed, since the charcoal filter will take out any trace elements of organic vapors in the air heavier than a C_2. If another activated charcoal filter is available, this may be attached to the end of the probe to scrub the air so that a clean air sample would be going to the detector. The external activated charcoal can be used on any instrument, with or without chromatograph, for providing a clean air sample to assess background level.
4) If background still stays up and cannot be reduced by any of the previous steps, the safety cover (if included) and the exhaust port on the detector chamber (Preamp Assembly) on the bottom of the case should be removed and the Mixer/Burner Assembly scraped or brushed with a small wire brush. (Reference paragraph 6.2.1.3.) This will remove any small quantities of contamination that are on the Mixer/Burner Assembly which could be the source of the background vapor. After cleaning the face of the burner and tube, replace the exhaust port and safety cover (if included) and reignite the OVA. If contamination on the burner face was the cause, the problem should be immediately resolved and the ambient background will drop to an acceptable level.
5) If the background is still present, place your finger over the inlet of the probe so as to reduce the flow of air to the detector chamber. Reduced flow rate may be observed either on the sample flow gauge or can normally be observed by the sound of the pump motor.
6) If the background drops immediately in response to the reduced flow of air to the chamber, this is an indication that the contamination is in the air sample line. Therefore, the various parts of the sample flow line such as pickup probes, umbilical cord to the instrument, etc., should be investigated by the process of elimination to see if the contamination can be isolated.
7) Serious contamination in the air sample line is very uncommon. However, if very large doses of very heavy compounds are sampled, there is a possibility of a residual contamination which would eventually clear itself out but may take a considerable period of time. A typical cause for the high background from the sample line is a

contaminated Mixer/Burner Assembly. See paragraph (4) above for cleaning procedure. If heavy contamination of the Mixer/Burner is still indicated by a high background, replace the Mixer/Burner Assembly. In several OVA *models, this will require removal of the Preamp* Assembly. The old Mixer/Burner Assembly should be either discarded or returned to the factory for cleaning and rebuilding.

8) In the event there is contamination in the pump or other internal parts of the sample flow lines *which cannot be removed, the sample flow* components would have to be disassembled and cleaned. This is normally a factory type operation. However, the components such as the pump can be replaced in the field along with any contaminated tubing in the sample lines.

9) *High background readings on OVA's which in*clude the Gas Chromatograph Option can be caused by other sources of contamination. If the charcoal in the charcoal filter mounted on the panel of the instrument is contaminated or saturated, contaminated air would be supplied to *the detector and raise the ambient level* background. To check for this, the charcoal filter cartridge can be removed from the panel and either a bypass tube put between the two connectors or the charcoal can be removed from the charcoal cartridge and the cartridge refilled with clean activated charcoal. This would determine if the charcoal was the source of the background reading. It is possible that an apparent high background reading could be due to contamination in the column that is on the instrument. This background could be caused by compounds that are slowly eluting from a column which has become contaminated. The easiest way to check for column contamination is to replace the column with a known clean column or a short empty piece of column tubing and see if the high background reading drops.

10) If all the above steps do not correct the high background problem, the cause will normally be contamination in the hydrogen fuel system.

Contamination in the hydrogen fuel system is usually the direct result of contamination in the hydrogen gas sed or *contamination introduced during the filling* peration. Filling hose contamination can be caused by storing the hose in a contaminated area.

To remove contamination from the hydrogen fuel ystem, it should be purged with hydrogen. Effective urging of the hydrogen system is accomplished by *disconnecting the capillary tube fitting which attaches* on to the manifold block which has the low pressure auge (H2 Supply Pressure Gauge and H2 Supply alve). This disconnects the capillary tubing from the ydrogen line so that hydrogen may be purged at a reasonable rate from the tank assembly through the *egulators, gauges and valves. After disconnecting the* apillary, the hydrogen tank can be filled in the normal manner. The tank valve and H2 supply valve can then be opened which will bleed the hydrogen from the tank through the H2 fuel system purging out the contamination which is in vapor form. There is the possibility that contamination has been introduced into the hydrogen *fuel system which is not readily purged out by the* hydrogen gas but this is unlikely. After purging with clean hydrogen, approximately two or three times, the capillary tube should be reconnected and the background again checked. Five or ten minutes should be allowed before assessing the background reading, *since contaminated hydrogen may still have been trap*ped in the capillary tube.

If another tank assembly in a clean instrument is available, the fuel system from the clean instrument can be connected to the contaminated instrument to absolutely verify that it is or is not in the hydrogen fuel supply system. The interconnection should be made to the capillary tube of the contaminated instrument.

6.2.6 FUSE REPLACEMENT

This paragraph applies only to the standard (non-certified) OVA's. There are two (2) overload fuses incorporated in the Battery Pack Assembly, one is a 3AG-1 AMP Slo-Blo in the power line to the pump and igniter and the other a 3AG-1/4 AMP in the power line to the electronics. Both fuses follow the current limiting resistors which provide primary short circuit protection. However, in the event of an excessive overload, the fuses will open and prevent overheating of the current limiting resistors. It should be pointed out that the 1 AMP Slo-Blo fuse will blow in approximately 8 to 12 seconds if the igniter switch is kept depressed. Normal ignition should take place in not more than 6 seconds. Therefore, do not depress igniter button for more than 6 seconds. If ignition does not occur, wait 1 to 2 minutes and try again. If the required 1 AMP Slo-Blo fuse cannot be readily obtained, replace temporarily with a 3 AMP-3 AG standard fuse.

6.3 TROUBLE SHOOTING

Table 6-1 presents a summary of recommended field trouble shooting procedures. If necessary, the instrument can be easily removed from the case by unlocking the four (4) 1/4 turn fasteners on the panel face and removing the refill cap and exhaust port. The battery pack is removed by taking out the four (4) screws on the panel and disconnecting the power connector at the battery pack.

6.4 FACTORY MAINTENANCE

To ensure continuous trouble-free operation, Century recommends a periodic factory maintenance, overhaul *and recalibration. The recommended schedule is every* six (6) to nine (9) months. This maintenance program includes replacement of plastic seals and parts as required, pump overhaul, motor check, new batteries, sample line cleaning, H2 leak check, recalibration, replacement of plastic hose as required, and detailed *examination of the unit for any other required* maintenance and repair.

The recommended procedure for maintenance and repair beyond the scope of this manual is to send the complete instrument or subassembly to the Century factory for repairs. The assemblies will be handled expeditiously for rapid turn-around.

6.5 FIELD MAINTENANCE

Although not recommended, where field maintenance beyond that described herein is considered essential, the assembly drawings, parts lists and schematics in Appendix "A" will be of assistance.

6.6 RECOMMENDED SPARES

Century does not recommend that spares be maintained for its instruments. However, if the instrument is to be used in a remote area or spares are desired for other reasons, the following list should be used as a guide.

RECOMMENDED SPARES

Item	Description	Part No.	Recommended Quantity					
			Standard			Approved		
			88	98	118	108	128	138
1	Igniter	510027-1	2	2	2			
2	Igniter	510461-1				2	2	2
3	Pump Valve	510067-3 (10/pkg.)	1	1	1	1	1	1
4	Pump Diaphragm (Buna-N)	510091-1	1					1
5	Pump Diaphragm (Teflon)	510063-1		1	1	1	1	
6	Cup, Filter (3/8 OD, SS)	510318-1 (5/pkg.)	1	1	1	1	1	1
7	Mixer/Burner Assy	510557-2	1					
8	Mixer/Burner Assy	510557-1		1	1			
9	Mixer/Burner Assy	510513-1				1	1	1
10	Wafer, Teflon, H_2 Valve	510160-1 (10/pkg.)	1	1	1	1	1	1
11	Washer, Brass, H_2 Valve	510160-2 (10/pkg.)	1	1	1	1	1	1
12	Exhaust Port Assy	510425-1	1	1	1			
13	Exhaust Port Assy	510530-1				1	1	1
14	Battery Pack Assy	510070-1	1	1	1			
15	Battery Pack Assy	510542-1				1	1	1
16	Sample Line Assy	510316-1		1	1	1	1	1
17	Particle Filters	510114-1	1					1
18	Particle Filters	510116-1		1	1	1	1	

NOTE: Unit quantity is each unless otherwise noted.

TABLE 6-1

TROUBLE	TROUBLE SHOOTING PROCEDURE	REMEDY
1) *Low sample flow rate on flow indicator.* Nominally 2 units on flow gauge. (See also 6 below and refer to paragraph 6.2.4)	a) *Check primary filter in sidepack and particle filters in the pickup assembly.*	*Replace or clean filter if clogged. (See paragraph 6.2.1)*
	b) Determine assembly containing restriction by process of elimination, i.e., remove probe, remove Readout Assembly, remove primary filter, etc.	Investigate the assembly containing this restriction to determine cause of blockage. Clean or replace as required.
	c) If the restriction is in the Side Pack Assembly, further isolate by disconnecting the sample flow tubing at various points, i.e., pump output, chamber input, etc. *Note: The inherent restrictions due to length of sample line, flame arrestors, etc., must be taken into account when trouble shooting.*	If in the detector chamber, remove and clean or replace porous metal flame arrestors. If pump is found to be the problem, remove and clean or replace.
2) H2 flame will not light. (See also 8 below)	a) Check sample flow rate (see 1 above).	If sample flow rate is low, follow procedure 1 above.
	b) Check igniter by removing the chamber exhaust port and observing the glow when the IGNITE Button is depressed.	If igniter does not light up, replace the plug. If igniter still does not light, check the battery and wiring.
	c) Check for rated H2 Supply Pressure. (Listed on calibration plate on pump bracket.)	If low, remove battery pack and adjust to proper level by turning the allen wrench adjustment on the low pressure regulator cap.
	d) *Check H2 flow rate by observing the PSI* decrease in pressure on the H2 Tank Pressure gauge. The flow rate should be about 130 PSI decrease in pressure per hour. (Approximately 12 cc/min. at detector.) *On instruments with GC Option, disconnect column and measure H2 flow rate with a bubble meter.*	*The normal cause for H2 flow restriction would be a* blocked or partially blocked capillary tube. If flow rate is marginally low, attempt to compensate by increasing the H2 Supply Pressure by one-half or one PSI. If flow rate cannot be compensated for, replace capillary tubing.
	e) *Check all H2 plumbing joints for leaks using* soap bubble solution. Also, shut off all valves and note pressure decay on H2 tank gauge. It should be less than 350 PSIG per hour.	*Repair leaking joint.*
	f) Check to see if H2 supply system is frozen up by taking unit into a warm area.	If there is moisture in the H2 supply system and the unit must be operated in subfreezing temperatures, purge *the H2 system with dry H2 and ensure the H2 gas used* is dry.
	check battery level by connecting to charger	If in red zone, battery may not have enough power to light flame (could with clean battery, NOVA)

	g) Remove exhaust port and check for contamination. (See Figure 6-2.)	If the chamber is dirty, clean with ethyl alcohol and dry by running pump for approximately 15 minutes. If H2 fuel jet is misaligned, ensure the porous metal flame arrestor is properly seated.
	h) Check spacing between collecting electrode and burner tip. Spacing should be 0.1 to 0.15 inches.	Adjust by screwing Mixer/Burner Assembly in or out. This spacing problem should only occur after reassembling a Mixer/Burner Assembly to a Preamp Assembly.
3) H2 flame lights but will not stay lighted	a) Follow procedures 2 (a), (c), (d), (e), (g) and (h) above. Also refer to 5 below.	
4) Flame-out alarm will not go on when H2 flame is out	a) Check instrument calibration setting and GAS SELECT control setting. Refer to paragraphs 2.3.1.2 and 2.3.2.	Readjust as required to proper setting. Note that on linear OVA's the flame-out alarm is actuated when the meter reading goes below zero. On logarithmic OVA's, the alarm is actuated when the signal level goes below 1 ppm methane or equivalent.
	b) Remove exhaust port and check for leakage current path in chamber (probably moisture or dirt in chamber).	Clean contamination and/or moisture from the chamber using a swab and alcohol, dry chamber by running pump for approximately 15 minutes.
	c) If above procedures do not resolve the problem, the probable cause is a malfunction in the preamp or power board assemblies.	Return preamp chamber or power board assembly to the factory for repair.
	d) Check volume control knob is turned up.	Adjust for desired volume.
5) False flame-out alarm. (Applies to linear OVA's)	a) Flame-out alarm is actuated on linear instruments when signal goes below electronic zero (eventhough flame is still on). This can be due to inaccurate initial setting, drift or a decrease in ambient concentration. Verify if this is the problem by zeroing meter with flame out and reigniting. (See paragraph 2.3.2)	When using the X1 range, adjust meter to 1 ppm rather than zero. See paragraph 2.3.2. Be sure instrument has been zeroed to "lowest expected ambient background level".
6) Slow response time, i.e., time to obtain response after sample is applied to input. (Refer to paragraph 6.2.4)	a) Check to ensure that probe is firmly seated on the rubber seal in the readout assembly.	Reseat by holding the probe firmly against the rubber seal and then lock in position with the knurled locking nut.
	b) Check sample flow rate per procedure 1 above.	See 1 above.

7) Slow recovery time, i.e., too long a time for the reading to get back to ambient after exposure to a high concentration of organic vapor.	a) This problem is normally caused by contamination in the sample input line, requiring pumping for a long period to get the system clean of vapors again. Charcoal in the lines would be the *worst type of contamination. Isolate through* the process of elimination. (See 1 (b)).	Clean or replace contaminated sample line or assembly as required.
	b) Check flame chamber for contamination.	Clean as required.
8) Ambient background reading in clean environment is too high. (Refer to paragraph 6.2.5)	a) An ambient background reading can be caused by hydrocarbons in the H2 fuel supply system. Place finger over sample probe tube restricting sample flow and if meter indication does not go down *significantly the contamination is probably* in the H2 fuel.	Use a higher grade of hydrocarbon free hydrogen. Check for contaminated fittings on filling nose assembly.
	b) An ambient background reading can be caused by a residue of sample, building up on the face of the sample inlet filter. If the test in 8 (a) above produces a large drop in reading, this is usually the cause.	Remove the exhaust port (it is not necessary to remove instrument from case), use small wire brush from the tool kit or a knife blade and lightly scrub surface of sample inlet filter.
	c) *An ambient background reading can also be* caused by hydrocarbon contamination in the sample input system. The most likely cause would be a contaminant absorbed or condensed in the sample line. Note: It should be emphasized that running the instrument tends to keep down the buildup of background vapors. Therefore, run the unit whenever possible and store it with the carrying case open in clean air.	*Clean and/or replace the sample input lines. Normally* the lines will clear up with sufficient running.
9) Pump will not run	a) Check 1 AMP Slo-Blo fuse on the battery pack cover. *NOTE:* Certified OVA's do not have fuses.	Replace fuse. IMPORTANT: Note that fuse is a Slo-Blo *type. If fuse continues to blow when igniter switch is* closed, check igniter for short circuit. If igniter is not the *problem, there is a short in the wiring or pump motor.* Return OVA to factory or authorized repair facility.
10) No power to electronics but pump runs	a) Check 1/4 AMP fuse on the battery pack cover. NOTE: Certified OVA's do not have fuses.	Replace fuse. If fuse continues to blow, there is a short in the electronics assembly. Return OVA to factory or authorized repair facility.
11) No power to pump or electronics.	a) Place battery on charger and see if power is then available. Recharge in a non-hazardous area only.	If power is available, battery pack is dead or open. Recharge battery pack. If still defective, replace battery pack. Reference paragraph 2.7.

APPENDIX C-8

Calibration and Maintenance of
GPK Gas Meter

Extracted from Manufacturers
Instruction Manual

OPERATING INSTRUCTIONS
Model GPK or GK
COMBINED OXYGEN/COMBUSTIBLES INDICATOR

INSTRUCTIONS 23-9081
OPERATION AND MAINTENANCE
J-W MODEL GPK AND GK OXYGEN/
COMBUSTIBLE GAS INDICATOR

7.0 CALIBRATION (COMBUSTIBLES SECTION)

The standard calibration using methane has been selected so that most commonly encountered gases and vapors as listed below and the more volatile lacquer and paint thinners, all indicate quite accurately or within reasonable error on the side of safety.

Gases	Vapors	
Natural Gas	Pentane	Benzene
Methane	Hexane	Ethyl Ether
Ethane	Heptane	Acetone
Propane	Methyl Alcohol	Ethyl Acetate
Butane	Ethyl Alcohol	
Carbon Monoxide		
Acetylene		

7.1 Checking Combustibles Calibration Using Known Gas Sample (See Figure 12)

It is often necessary, and even mandatory, to periodically check the instrument against a known, standard blend of calibration gas.

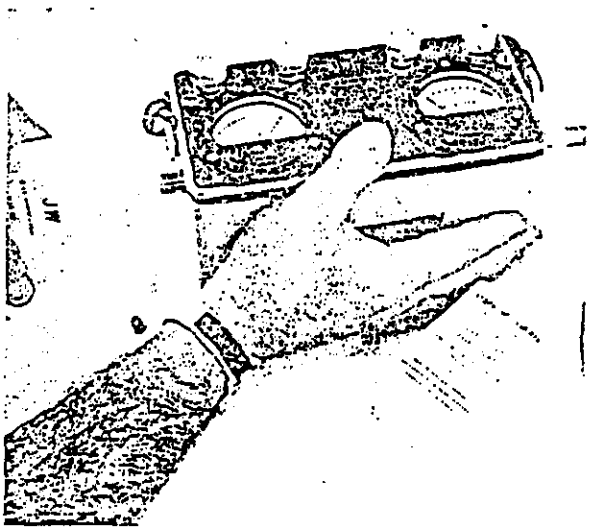


Figure 12 [illegible]

The J-W 550-075 Gas Indicator Test Kit provides a convenient method for testing the instrument's response to a known gas concentration. Methane is used as a calibration gas because indicator response to methane is more critical than response to most other gases. If the indicator has normal response to methane it will respond normally to most other combustible gases and vapors. Contained in the Kit are gas-filled ampoules and a plastic squeeze bottle which will produce a known 1.1% by volume methane/air mixture, when one ampoule (brown color coded end) is shattered in the bottle. Use the following procedure to calibrate the instrument.

(a) Remove the screw spout and squeeze plastic bottle several times to purge residual gas from previous test.

(b) Drop one glass ampoule into the plastic squeeze bottle and replace the screw spout. Make certain that red end cap is firmly in place.

(c) Shake the bottle until the ampoule is shattered by the breakers in the bottle. With the instrument in normal operation but without *sample probe and hose attached, remove* the red end cap, grasp the bottle firmly in the left hand and force the nozzle into the sample inlet fitting on the left side of the instrument. Inject gas sample while keeping pressure on the bottle.

Do not depend upon the pump or use of the aspirator bulb to draw sample through the instrument as this will result in leakage and unintentional dilution.

(d) Observe that the combustible meter pointer deflects upscale. Record the maximum meter reading indicated. An acceptable reading is approximately .25 L.E.L. on the combustible meter scale. .20 L.E.L. or less is unacceptable and is cause for recalibration.

NOTE: If low reading is obtained the first time, repeat the test to insure that no dilution occurred.

(e) Refer to Figure 13a for location of the calibration adjustment potentiometer, which is accessible through the small hole in back of the case. Use a small screwdriver to turn the slotted shaft *clockwise* to *increase* reading. This is a 25 turn potentiometer, so several turns may be required to bring the meter reading to .25 L.E.L. while simultaneously squeezing the plastic bottle. After readjusting the calibration potentiometer, repeat 7.1 (c) again as a final check.

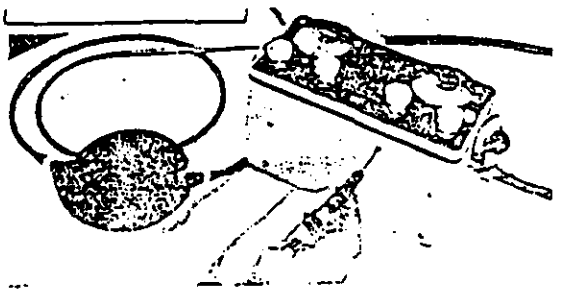


Figure 13a Adjusting Calibration Potentiometer

7.3 Testing Oxygen Cell for Calibration and Sensitivity

Operation of the instrument is normal if the meter can be set to read 21% on atmospheric air. A convenient test to prove response is to set the calibration to 21% and then blow slowly into the indicator inlet. As the lungs become exhausted the readings will go lower, a reduction to 15% being normal. With effort, the reading can be reduced as low as 10%.

To check performance near 0% oxygen, admit a small steady stream of oxygen free gas to inlet, such as nitrogen or natural gas. A cell in good condition will give a zero reading. A reading of 1% or more indicates need for reactivation.

8.0 MAINTENANCE SCHEDULE FOR MODELS GK AND GPK

DAILY:

The instrument should be checked for response before each day's operation as follows:

1. Check battery voltage reserve by observing whether voltage can be adjusted to at least 0.7 on the combustibles scale; then reset to green index arrow.
2. Check oxygen cell on fresh air and observe whether meter can be set to 21% O_2.
3. Check combustibles response by placing a few drops of gasoline or lighter fluid on a cloth, and proceeding as in Section 5.1 Step (e). Observe that combustibles pointer deflects upscale (if a slight explosion is heard, no harm will be done). Avoid excessive use of fluid as it will be difficult to clear the instrument.
4. Test leak tightness of sample inlet system by closing off the inlet fitting with finger and listen for pump slowdown to occur (GPK). With Model GK, check for internal leaks by squeezing the aspirator bulb, placing the finger over the inlet fitting, and releasing the bulb. If the bulb springs back quickly, an internal leak is present that should be corrected before using the instrument.
5. Replace cotton filter in probe chamber if working in an extremely dirty environment.

WEEKLY: Test instrument response to a known concentration of methane in air as outlined in Section 7.1.

MONTHLY (or as required):
Check combustibles reaction chamber for accumulations of dirt or foreign material that may restrict free movement of gas sample.

EVERY SIX MONTHS (or as required):
Maintain Oxygen Cell as outlined in Section 8.1.1 Step (a) through (e).

8.1 Oxygen Cell-Maintenance (Refer to Figures 14 and 15)

The 514-010 Oxygen Cell requires periodic maintenance and reactivation.

The 514-011 Reactivation Kit is supplied for this purpose. There are two levels of cell maintenance, which are described below.

Both operations can be carried out with one kit, as an adequate supply of electrolyte solution (KCl) is provided to fill the cell at least twice.

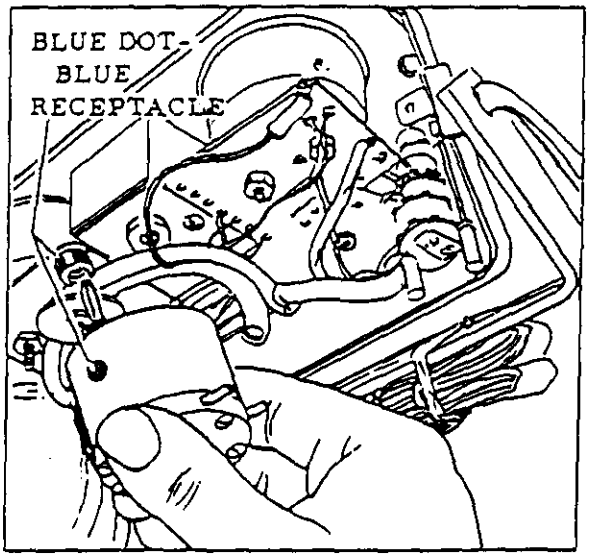


Figure 14: Correct method of connecting Oxygen Cell (note position of blue dot).

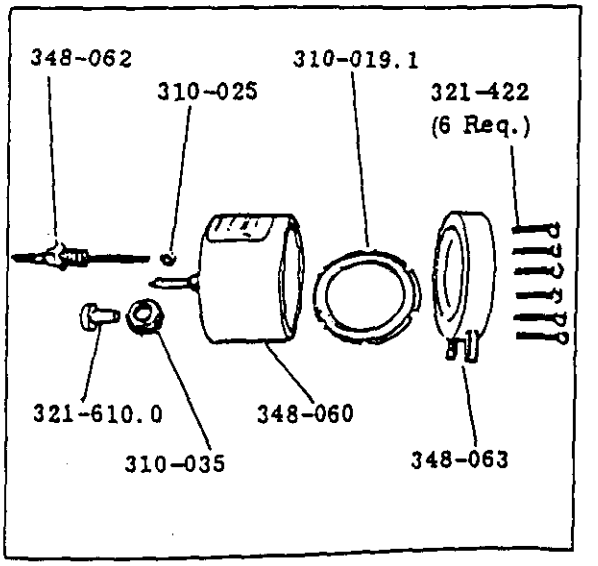


Figure 15. Parts identification for 514-010 Oxygen Cell.

8.1.1 Maintenance

The Oxygen Cell will usually operate up to 6 months without attention, but occasionally within this period, cell output may drop appreciably or change suddenly with sampling or sudden shock. To correct this condition, proceed with the following:

(a) Remove top panel from instrument case by loosening the two captive hold-down screws.

Unplug Oxygen Cell from the printed Circuit Board and disconnect the plastic tubing from the cell inlet and outlet nipples.

(b) Remove the nylon screw and seal ring on the electrical plug side of the cell. Refer to Figure 15 and unscrew the zinc electrode (banana plug nearest the blue dot on the side of cell) and remove it together with its O-Ring seal.

(c) Drain all the solution from the cell and completely fill with fresh KCl solution through either opening.

(d) Replace O-Ring on the zinc electrode, screw the electrode into the cell and tighten, using a small wrench. Install and retighten nylon screw with seal.

(e) Allow a few minutes for the cell to stabilize before attempting to make a reading.

NOTE: DO NOT WASH OR WIPE ELECTRODE AT ANY TIME.

8.1.2 Reactivation of Oxygen Cell (Refer to Figures 15, 16, 17)

In the event oxygen cell fails to function properly after changing solution as outlined in Section 8.1.1 or if it has been six months since solution was last changed, the cell may require reactivation. Reactivation Kit 514-011 is all that is required to completely reactivate the oxygen cell. The Kit contains a cap gasket with Teflon membrane, zinc electrode with O-ring, nylon screw with seal, KCl solution, nylon pad and brush.

To reactivate the cell, proceed as follows:

1. Remove nylon screw and seal and unscrew and remove zinc electrode and O-ring. Discard the zinc electrode, the screw, the O-ring and the seal.
2. Drain solution from cell.
3. Remove cap and gasket with membrane by removing the six screws which hold the cap in place. Discard the gasket with membrane.
4. Wash the cell thoroughly with cold, clean water. Use the brush to remove any white substance that may remain on the gold electrode. DO NOT USE ANY SOAP OR DETERGENT. If the white substance cannot be removed from sive nylon pad can be used to remove it, but the gold electrode should be handled very carefully and only slight pressure applied in cleaning with the nylon pad. Wash again with brush and water.
5. Put one drop of KCl solution on top of the gold electrode and put new gasket with membrane in place. The side of the gasket with the Teflon membrane should face the gold electrode.
6. Replace the cap, observing the direction of the sampling nipples. Nipples should point away from the label on the cell body. Tighten all screws until the cap shoulder is flush with the cell body.

The cell is now dry charged and can be stored as long as desirable.

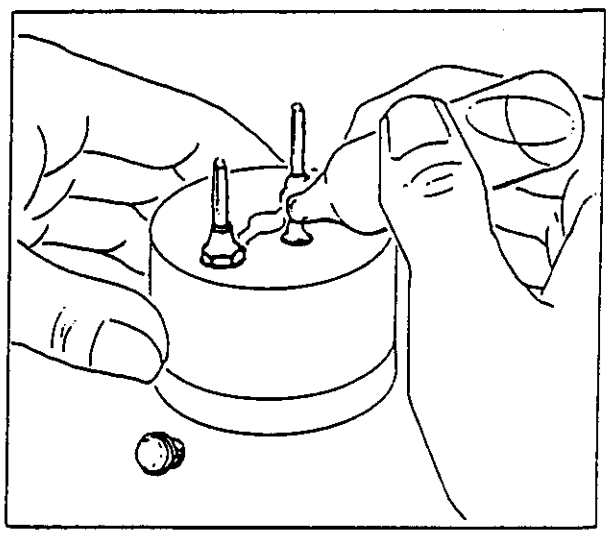
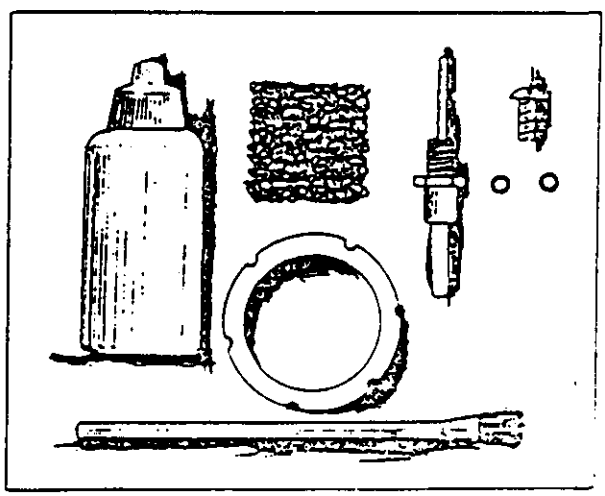


Figure 16: Activating cell with electrolyte (KCl solution).



To activate the cell:

1. Fill the cell with KCl solution, through either of the openings for the nylon screw or for the zinc electrode.
2. Check to see if the O-ring is still in the zinc electrode opening; if it is, remove it.
3. Be sure the new O-ring is in place on the zinc electrode, screw zinc electrode into cell and tighten with a small wrench.
4. Plug fill hole with nylon screw and new seal ring (310-035). The cell is now fully activated.

8.2 Oxygen Cell Contamination by Foreign Liquids

If any liquid has been accidently introduced into the sampling line and thus into the sampling chamber of the cell, it will be necessary to flush the system out with distilled water as soon as possible. To do this, use the following procedure:

(a) Disconnect the cell completely, remove, and pour distilled water into one sampling nipple until it comes out of the other. Drain water out and repeat this procedure several times.

(b) Insert the cell into the P.C. Board, observing plug color coding. Reconnect sample tubing, one to the inlet and the other directly to sample pump or aspirator bulb (bypass the combustibles reaction chamber).

(c) Turn "ON" the pump (Model GPK) and allow 5 minutes drying time for samoling chamber. For Model GK, stroke aspirator bulb about 50 times to insure cell chamber is thoroughly dried. The cell should now register normal output and is ready for use.

(d) Reconnect sample tubing so that outlet of O_2 cell goes to reaction chamber, and outlet of reaction chamber goes to pump inlet (or aspirator bulb).

8.3 Maintenance of the Combustibles Section

Filaments and Flame Arrestor (Refer to Figure 18)

There are two separate filament units: (1) the active filament, which is installed in the flame arrestor, and is the unit that actually comes in contact with the gas being tested; and (2) the reference filament that helps balance the electrical circuit.

The reference filament should rarely require replacement inasmuch as it operates at a very low voltage and never comes in contact with the gas. Replacement may be necessary if the indicator has been cropped or severly damaged, or if the instrument has been in frequent use over many years.

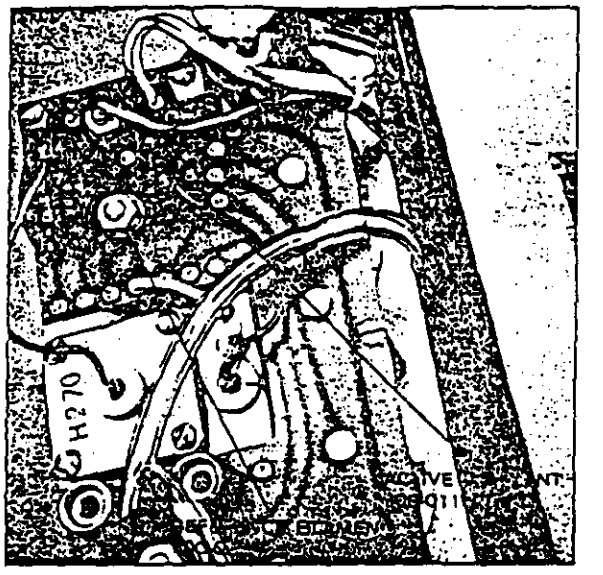


Figure 18: Active and reference filament locations.

The active filament should experience long life in ordinary service, probably on the order of a year or more, depending on frequency of use. The end of the useful life of the active filament is reached when it is no longer possible to bring the hand of the meter to "zero" with the zero adjustment. Another sign of trouble in the filament unit is when the meter pointer flies violently upscale with the instrument "ON," and zero adjustment is impossible. This usually means the active filament is burned out and must be replaced.

If the GK or GPK has been in use for a considerable period of time, or in moist or dusty conditions, it is frequently desirable to replace the filament flame arrestor assembly in combination. This is done by unplugging the O_2 cell, removing the four screws holding the brass plate to the underneath of the panel, and removing the filament lead from the subpanel terminal. When putting in a new filament flame arrestor assembly, make certain that the gasket is in place and compressed evenly by four screws.

When replacing the filament only, and reusing the flame arrestor, first remove and inspect the flame arrestor, checking for dust, corrosion or other damage. Replace gasket if it has deteriorated. Install new filament in place of old one, screwing it into the flame arrestor plate to compress the O-ring gasket as tightly as possible using the thumb and finger. When inserting filament be very careful not to touch or distort the platinum coil.

To replace the reference filament, first unsolder the black wire at subpanel, and unplug the O_2 cell. Then unscrew the knurled reference filament base from its cavity, using a pair of pliers. Install the new filament in the same position, tightening with pliers, and solder wire to subpanel.

NOTE: After replacement of either the active or reference filaments, perform the OPERATIONAL CHECK as outlined in Section 5.1, Steps (a) through (e).

Part numbers for filaments are:

800-007—Active Filament Flame Arrestor Assembly
800-011—Active Filament only (red wire)
800-022—Reference Filament only (black wire)
310-017—*Flame Arrestor Gasket only*

8.4 Battery Replacement and Charger Check (Refer to Figures 19, 20)

The most frequent form of maintenance will involve recharging the batteries as outlined in Section 5.3 and occasional burnishing of the silver contact buttons on the Printed Circuit Board with fine sandpaper (No. 400 or equivalent). The batteries can be recharged hundreds of times to render many years of useful service. Usually, the *most probable cause of "apparent" battery failure* can be traced to charger malfunction or contact problems. The batteries can only be charged with the instrument panel firmly in place. With the panel removed, the batteries can be inspected for deterioration by removing them from the storage compartment. When replacing batteries, be certain to reinstall them in the positions indicated on the side of the battery compartment.

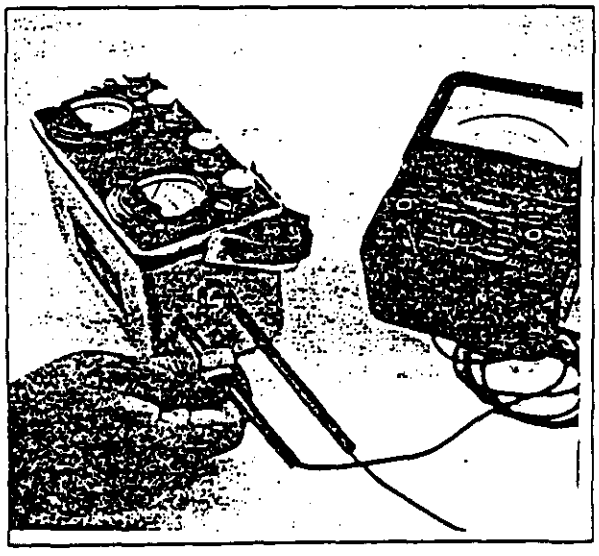


Figure 19: Checking power circuit for proper voltage (Section 8.4, Steps a and b)

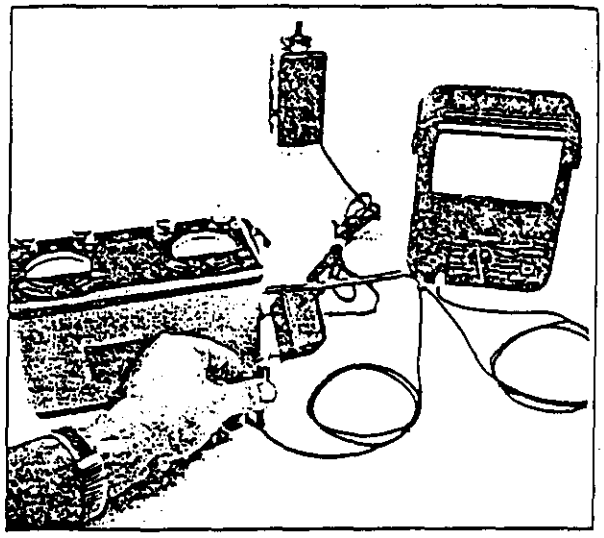


Figure 20: Measuring charge rate (Section 8.4, Step d).

NOTE: OBSERVE CORRECT POLARITY—INCORRECT BATTERY INSTALLATION CAN RESULT IN DAMAGE TO THE BATTERIES OR TO THE INSTRUMENT ITSELF. To insure that batteries are being recharged and that the Charger is functioning properly, perform the following check:

(a) Obtain a Multimeter and place the range selector on the 10 volt scale. Connect the meter test probes across the instrument charging jacks as illustrated in Figure 19. With the instrument *FUNCTION CONTROL in the "OFF" position*, the Multimeter should indicate 5 volts.

(b) Turn the FUNCTION CONTROL switch to the "ON" position and observe that battery voltage drops to 2.5 to 2.6 volts, then remove the meter probes. NOTE: *Failure to obtain 2.5 volts* indicates depleted batteries, open contact with subpanel buttons or open electrical connection. Correct malfunction before proceeding with the next step.

(c) Plug Charger line cord into 115 VAC outlet. Connect meter probes across charger pins and check output for approximately 5.5 volts (normal unloaded voltage).

(d) Now refer to Figure 20 and connect the Charger as shown with only the negative pin (—) connected to the black (—) charging jack. Place the Multimeter range selector to the 500 ma scale.

Connect the (+ red) positive meter probe to the remaining exposed pin on the Charger and the black (—) Multimeter probe to the red (+) charging jack of the GPK (GK). With Charger

connected to 115 VAC. Multimeter should indicate a charge rate of 120 to 150 ma (depending on battery state, line voltage, etc.) with the FUNCTION SWITCH in the "OFF" position. If Multimeter indicates "zero" ma, refer to Troubleshooting Section 8.9, Step 6.

(e) Now turn on the FUNCTION SWITCH to the "ON" position. Multimeter should indicate approximately 250 ma. (Never attempt to charge the batteries with the switch "ON," because the power consumption is greater than the Charger delivery rate.) If Multimeter indicates 300 ma or more, refer to Troubleshooting Section 8.9, Step 6.

NOTE: Failure to attain the above readings indicates a faulty Charger or Pump motor, open contact with subpanel buttons, or open electrical connection. If the correct indications are obtained, the circuit is normal and the batteries simply require recharging.

8.5 Testing Sample Flow Lines (Refer to Figure 17)

When installing the instrument panel in the bottom housing, take care not to pinch or obstruct the sample tubing. Always make certain that the sample tubing is placed in the cavity beside the battery compartment and arranged to prevent sharp bends or pinching when the panel is secured. For both Models GK and GPK, normal flow rate is approximately 2.5 liters per min. and response time to a combustible gas is approximately 5 seconds when short probe and 30" hose is used. Leaks or restrictions in the flow system will of course increase the response time.

A quick and simple check to determine the tightness of the sample system is to obstruct the flow at the probe tip or inlet fitting of the indicator. In the Model GPK the pump should slow or stop. If it does not, a leak in the pump or sample system is indicated. In the Model GK the aspirator bulb should be squeezed, released, and remain compressed for about 20 to 30 seconds. If the pump continues to operate normally or the bulb immediately springs back to its original form a leak exists which must be corrected before attempting to use the instrument.

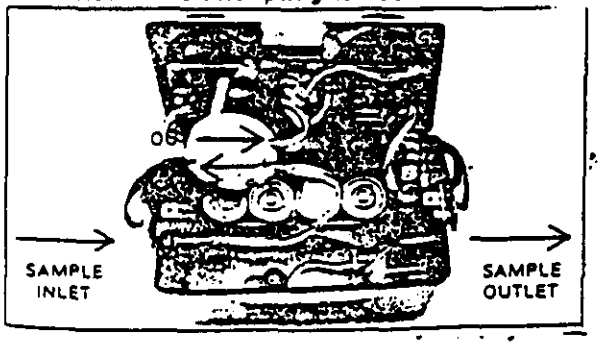


Figure 21 Sample tubing connections for all major components

8.6 GPK Sample Pump/Motor Replacement (Refer to Figure 18)

The GPK Sample Pump Assembly Code 342-626 is located adjacent to the outlet fitting in the Bottom housing.

To replace the Sample Pump Assembly, first remove the two screws from the outside of the lower case housing and release the pump assembly with mounting bracket. Mark the bracket "TOP" and "BOTTOM" for reference, then disconnect sample tubing. At the printed circuit board, unsolder the red and black leads to the pump motor and note the location for future reference.

With a 3/64" Allen wrench, loosen the set screw in the cam to free motor shaft, then remove the two screws from the motor mount.

Check valve action by sucking gently on the inlet tubing connection and blowing gently into the outlet tubing connection. The valves should resist either test and stay firmly closed. If the valves pass the above test, only the pump motor will require replacement. If not, the complete Sample Pump Assembly Code 342-626 must be overhauled or replaced.

Install new motor Code 04-4053 and secure to pump mounting bracket. Reinstall pump assembly and bracket, solder pump connections to printed circuit board and connect tubing as illustrated in Figure 22A

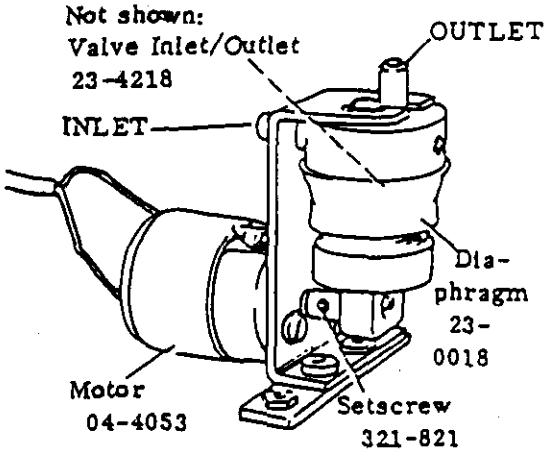


Figure 22: 342-626 Sample Pump with motor and mounting bracket. Inlet and Outlet tubing connections shown.

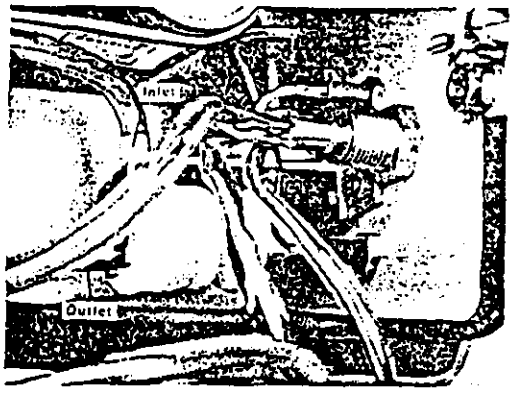


Figure 22A: Sample Pump Assy. properly installed

8.7 Meter Replacement

Meter trouble is usually indicated by erratic action, for instance, the indicator meter pointer will start to go up smoothly, pause momentarily at a reading, and then jump quickly over the the next few divisions of the scale. Another sign of trouble is for a meter to be almost or entirely inactive, and to "stick" or show a permanent displacement of the pointer.

If the meter is giving trouble it may be removed by taking off the meter nuts and the meter hold down screws. Field repair of meters is generally inadvisable unless a qualified instrument repair facility is available.

PLEASE NOTE FOLLOWING BEFORE REPLACING METER:

The zero, calibrate and voltage adjuster knobs all turn an arm which runs back and forth over a resistance coil. To keep the sliding contact of the coil clean, it is good practice to turn the knobs back and forth three or four swings of the meter pointer each day as the instrument is first put into service.

8.8 Trouble Shooting Guide

SEE CHART ON OPPOSITE PAGE.

9.0 ACCESSORIES FOR MODELS GK, GPK

9.1 Probes and Sampling Hoses

When Models GK and GPK are purchased complete with carrying case and accessories, a short aluminum probe with filter and 30" of Nylon lined hose are supplied as standard (Stock No. 550-015).

For special applications as noted below, longer probes in aluminum and fiberglass (nonconducting) and longer hose assemblies can be supplied. Various styles are available, and extension hoses can be provided for coupling between the hose and the instrument. The aluminum probe is generally used except where the non-conducting properties of fiberglass are required for the application. Nylon lined hose is particularly resistant to organic vapors, such as the aromatics like xylene.

The following hose and probe combinations with standard union fittings are available.

1. Fiberglass probe with dust filter chamber and 42" nylon lined hose (Stock No. 550-013). Used primarily by utility companies where an electrically non-conducting probe is desired and gases such as natural and manufactured gas are sampled.

2. Aluminum probe (long) with dust filter chamber and 42" nylon lined hose (Stock No. 550-014). Used in nearly all other applications. Probe of 3/8" diameter is very rugged and is perforated a few inches from the tip to prevent liquid from getting into the sampling hose should the probe be accidentally placed in liquid. The hose is unaffected by petroleum vapors.

3. Five foot hose. Stock No. 550-060 (nylon lined). Used as an extension hose. Can be used with either (1) or (2) above. Longer lengths of hose, complete with unions, are available in 5, 10, 15, 20, 25 and 50 foot lengths.

4. Dust filter chamber with fittings (Stock No. 550-025). Useful where just a filter chamber is required at the instrument inlet. Standard hose (550-060, or longer extensions) can be attached for remote sampling.

9.2 Sample Line Moisture Trap (See Figure 23)

#550-081 Sample Line Chamber. Although the cotton filter in the filter chamber of the probe tends to trap water or other liquids before they reach the instrument, it is normally a wise procedure to use a water trap if there is a continual problem with excessive moisture.

The 550-081 Sample Line Chamber is designed to act as a moisture trap when there is danger of drawing liquid or water into the instrument, or to help dry a sample when it is filled with calcium chloride or other drying agent. It can also be used as an additional dust filter trap where extremely dusty atmospheres are encountered.

The Sample Line Chamber comes complete with fittings, one end attaching to the GK or GPK sample inlet, and the other end to the sampling hose. The Chamber itself is a clear lucite tube. The capacity of the chamber is about 3 oz.

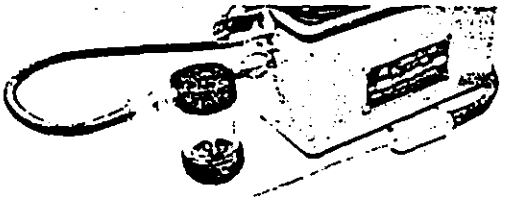


Figure 23: 550-081 Sample Line Chamber

APPENDIX C-9

AIR MONITORING SAMPLING EQUIPMENT AND ANALYSIS METHODS

A) Operating Manual for the HFS Air Sampling System
B) Mini-Buck Primary Gas Flow Calibrator
C) Quality Assurance Plan for the Analysis of Volatile Organics on Tenax/Tenax-charcoal Sorbent Tubes

A. OPERATING MANUAL FOR THE HFS AIR SAMPLING SYSTEM

Operating Manual
for the
HFS Air Sampling System

4.0 MAINTENANCE

4.1 BATTERY PACK REPLACEMENT (Refer to Figure 1)

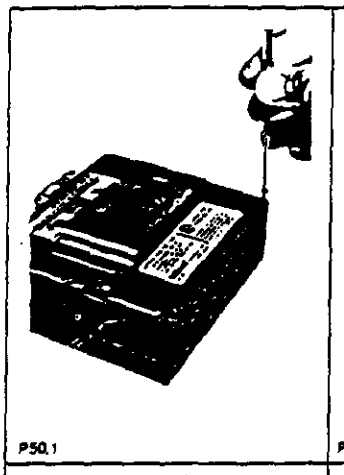
REMOVAL: Remove the two screws (19) which secure the Battery Pack (20) to the case front. Carefully slide the battery pack to the right out from under the belt clip (17) being careful not to cock it at an angle. Rails on the battery pack and the top of the belt clip guide the battery pack out of the unit.

4.1.2 BATTERY CHARGING

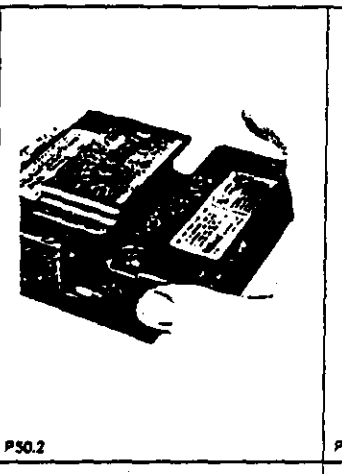
A proper battery maintenance program is essential to insure maximum battery life and performance. Specific charging and discharging procedures will vary with the users needs and/or applications. The following is a list of general recommendations which should help to provide efficient battery life and serviceability.

1. Do not short the battery connectors. This will result in irreversible damage to the battery pack.
2. Do not overcharge batteries. You should not charge battery packs at the high (standard) rate for more than 24 hours. Repeated overcharging will eventually lead to a deterioration in the performance level of the battery pack. If you must have the batteries on charge for more than 24 hours, we recommend that you switch the charger to the low (trickle) rate.
3. The long term storage of pumps will require some special handling. If pumps are not scheduled to be used for long periods of time (more than 2 months), it is recommended that the following procedure be followed on a periodic basis.
   a. Run pumps until shut down on low battery.
   b. Recharge batteries overnight (16 hours) and return pumps to storage.

REPLACEMENT: Place the pump on a soft, level surface. Slip the front edge of the battery pack (20) under the belt clip (17) and rock the battery pack so the rails engage with the slots on the case front. Push the battery pack to the left until it is properly located and reinstall screws (19).



P50.1



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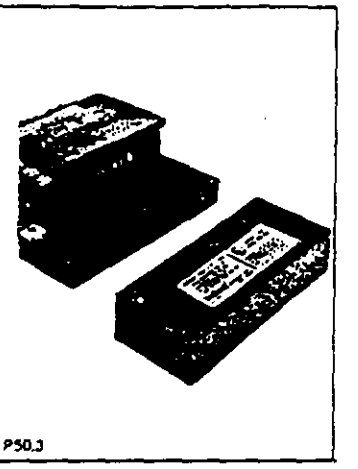
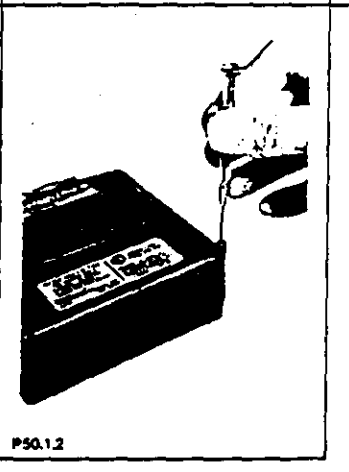


P50.3

P50.1.1



P50.1.2

P50.0 REMOVAL/INSTALLATION/ MAINTENANCE. This section is to provide the user with a basic knowledge of sampler upkeep and maintenance. When disassembling your pump, it is recommended that you work on a clean, level surface with a soft cloth underneath your sampler.

REPLACING THE BATTERY PACK / REMOVING THE BATTERY PACK

P50.1 Remove the (2) long screws from Battery Pack rear.

P50.2 Slide Battery Pack out slowly from under belt clip.

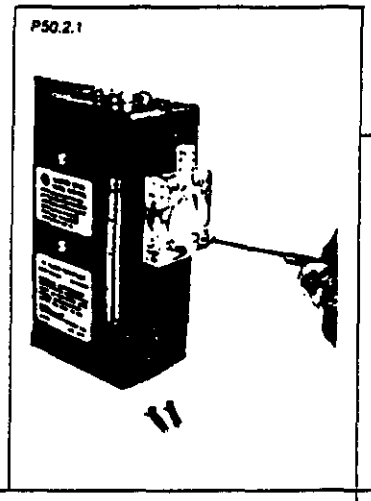
P50.3 Battery Pack removed. (Refer to section 4.0 of manual text on Battery Pack storage and maintenance.)

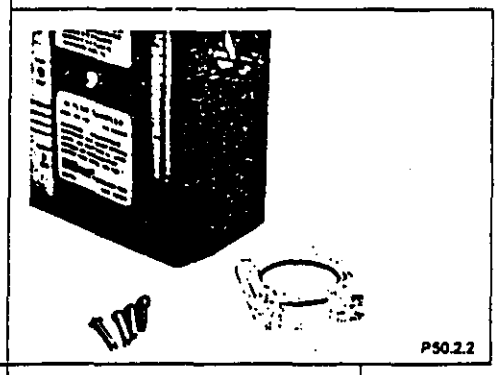
INSTALLING the BATTERY PACK

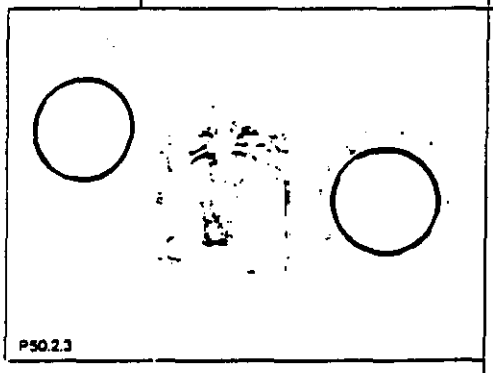
P50.1.1 Engage the rails of the Battery Pack.

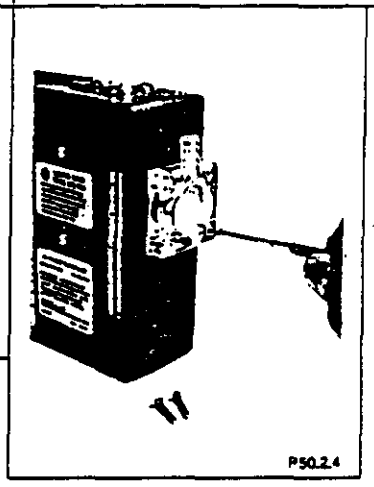
Gently slide Battery Pack under the belt clip until both jacks engage and the pack sits firmly in place.

P50.1.2 Reinstall the (2) long screws back into the Battery Pack rear. Battery Pack installation is now complete.









P50.2 FILTER REPLACEMENT — The filter and o-ring should be replaced when you see discoloration of the filter or a build-up of unknown particulate. Keeping up with this will insure accurate flows and samples as well as keeping the sampler operating efficiently.

P50.2.1 Remove (4) screws from the Filter Housing front located at the right side of the pump.

P50.2.2 Remove the filter housing front.

P50.2.3 Remove old filter and o-ring and replace with a new filter and o-ring using a Gillen Pump Filter Kit (PFK-100).

P50.2.4 Replace Filter Housing Front and reinstall (4) screws. Tighten screws until they stop turning. DO NOT OVERTIGHTEN. Filter replacement is now completed.

4.2 CHANGING THE PUMP FILTER (Refer to Figure 3). Under normal operating conditions the pump filter should be changed after approximately 250 hours of operation or sooner. Failure to change the filter as it becomes dirty will decrease the pump back pressure capability and performance envelope. Blow all dust and debris from around the filter housing. Remove the four screws (8) and the front filter housing (5). Remove and discard the filter membrane (6) and "O" ring (7). Wash the filter housing in mild soap and warm water solution and then blow it dry. Insert a new filter membrane and "O" ring. Insert the four screws (8) and cross tighten until the front filter housing is secured to the rear housing within the case.

LEAK TESTING (Refer to Figure 1) — Leakage test the system by removing screw (22) and installing the discharge air boss. Block the pump inlet boss and apply 20" of suction to the pressure fitting. No leakage should be observed. An alternate, which will pick up severe leakage is to turn the pump on, block the inlet boss, and observe that the pump cuts out on flow fault.

4.4 CALIBRATION

4.4.1 HIGH FLOW CALIBRATION (Refer to Figure 1, Table 1)

The constant flow feature of the sampler utilizes load sensing of the pump motor which, through a feedback loop, readjusts the motor speed so as to maintain flow constant. High Flow Calibration is accomplished by checking and adjusting the flow and Back Pressure compensation system (BCS) at two points, 1 LPM and 3 LPM. This insures that the calibration is accurate at all ranges in the high flow mode.

4.4.2 HIGH FLOW CALIBRATION AT 1 LPM

Set the pump to 1 LPM using the Flow Adjust Pot (9). Allow approximately 15 seconds to stabilize. Apply approximately 20" of back pressure by partially restricting the inlet to the pump and note the change in flow on the flowmeter or external flow calibrator. (The pump flowmeter is in the suction side of the pump. Consequently, the flowmeter will read higher as the pressure within the system decreases. An external flowmeter calibrator is not affected by this.) If the flow decreases (at 20" of back pressure) turn the Flow Adjust Pot (9) slightly counter clockwise which will cause the motor to speed up, increasing the flow until it is back to 1 LPM. If the flow increases (at 20" back pressure) turn the 1 LPM Flow Calibration Pot (10) slightly clockwise which will cause the motor to slow down, decreasing the flow until it is back to 1 LPM. Remove the back pressure. Reset the flow to approximately one liter if necessary. *NOTE:* IT IS NOT NECESSARY TO SET THE FLOW EXACTLY SINCE IT IS THE CHANGE IN FLOW WHICH IS OF IMPORTANCE. Repeat this procedure until the change is within ±5%.

4.4.3 HIGH FLOW CALIBRATION AT 3 LPM

Set the pump to 3 LPM using the Flow Adjust Pot (9). Allow approximately 15 seconds to stabilize. Apply approximately 10" of back pressure by partially restricting the inlet to the pump and note the change in flow on the flowmeter or external flow calibrator. (The pump flowmeter is in the suction side of the pump. Consequently, the flowmeter will read higher as the pressure within the system decreases. An external flowmeter calibrator is not affected by this.) If the flow changes with load (increases or decreases) note the AMOUNT OF CHANGE (differential). Remove the back pressure and adjust the 3 LPM Flow Calibration Pot (4) to increase or decrease flow ten times (10x) the differential noted.

Example:	*FLOW INCREASES.*	
	Flow at Set Point	3000 cc/min.
	Flow at 10" Back Pressure	3050 cc/min.
	Flow at Differential	+50 cc/min.
	10x	+500 cc/min.
	Calibration Set Point	3500 cc/min.
Example:	*FLOW DECREASES.*	
	Flow at Set Point	3000 cc/min.
	Flow at 10" Back Pressure	2950 cc/min.
	Flow at Differential	-50 cc/min.
	10x	-500 cc/min.
	Calibration Set Point	2500 cc/min.

Adjust Flow Control Pot (9) to bring flow back to 3 LPM. Reapply the back pressure and note change in flow if any. Repeat this procedure until the change is within ⅞5%.

4.4.4 LOW FLOW CALIBRATION 1 CC TO 500 CC/MIN

(HFS 113A U, HFS 113A UC, HFS 113A UT, HFS 113A UP, TIMING MODELS ONLY) (Refer to Figure 1 and Table 1.)

Low flow calibration consists of simply setting the differential pressure drop of the Low Flow Pressure Regulator. Once set, the regulator should not require frequent readjustment. Turn the pump on and adjust the Flow Control Pot (9) to 1 LPM. Remove the Protective Cap (23). With the Regulator Adjust Screwdriver, open the pressure regulator shut-off valve located under the cap (23) by turning it counter-clockwise approximately 5 turns (additional turns will have no effect). Connect a manometer or pressure gauge capable of reading up to 40" H_2O to the pump as shown in Figure 7.

NOTE: IF A MANOMETER IS USED, MAKE SURE THAT A WATER TRAP IS INSERTED BETWEEN THE MANOMETER AND THE PUMP INLET. THIS WILL PREVENT POTENTIAL DAMAGE CAUSED BY LIQUID ENTERING INTO THE PUMP ACCIDENTLY.

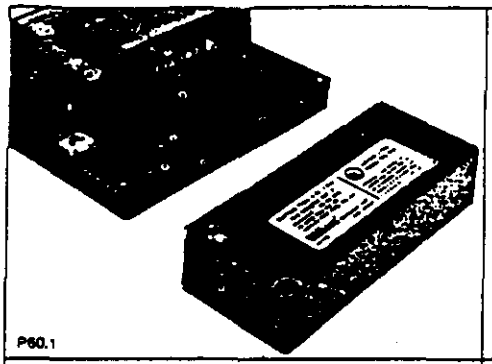
Attach a Variable Flow Controller tube holder as shown in Figure 7. Remove the Protective Cap (21) and completely block the air entering the tube holder. Adjust the pump suction pressure to 20" H_2O by turning the pressure regulator adjust screw located under Protective Cap (21) clockwise to increase the pressure or counter clockwise to decrease the pressure. With the Flow 0 to 200 cc/min. the pressure should not change more than 1½". Reinstall protective caps. This completes Low Flow calibration.

5.0 RECOMMENDED SPARE PARTS

Pump Filter Kit — PFK-100

5.1.0 PARTS LIST

(SEE PAGE #28 FOR FULL LISTING)



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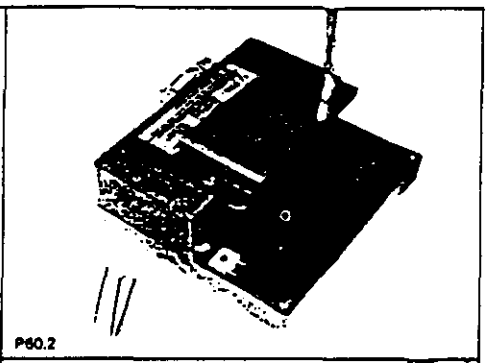

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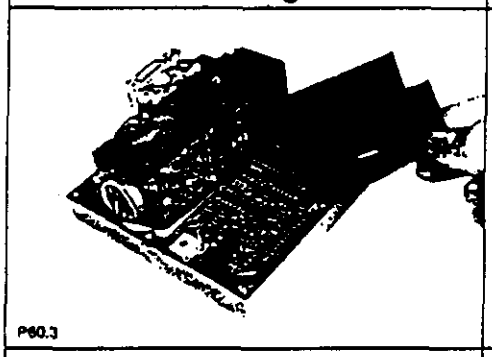

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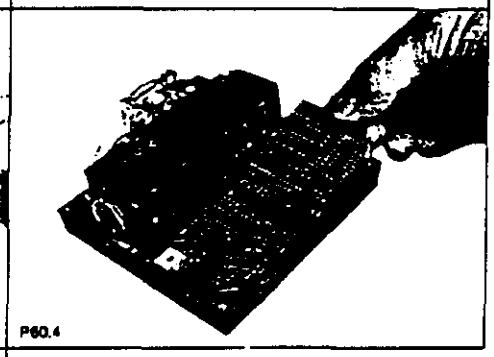

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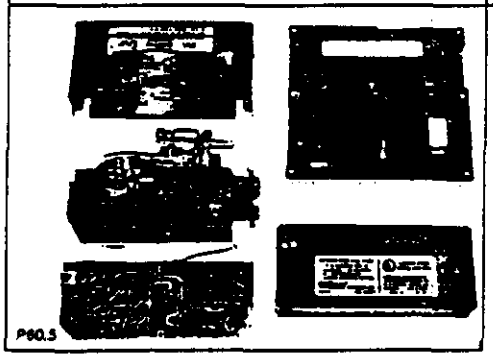

P60.5

P60.0 GENERAL DISASSEMBLY

P60.1 Remove Battery Pack (See Removal of Battery pack Section P50.0). (Photos)

P60.2 Remove (4) long screws from Case Rear. While holding the filter assembly, gently and slowly wiggle this cover upward.

P60.3 Remove protective foam from the electronic system and remove the (4) hold down screws from the Timer/-Control Board. NOTE: The fourth screw is located in the electrical jack.

P60.4 After hold down screws have been removed, lift gently and slide the Timer Board out of the Control Board.
NOTE: Handle these components gently and place on a soft surface.

The Pneumatic assembly will lift out of the case front along with the Control Board which is connected by a wire harness.

P60.5 These are your basic HFS assemblies.

NOTE: If you have problems with the Pneumatic Assembly or have damaged it with water or in a corrosive atmosphere, send the unit to the Gillan Repair Dept. or contact the factory for further servicing. (Please refer to Gillan Service Policy on Repair and Service Information.)

B. MINI-BUCK PRIMARY GAS FLOW CALIBRATOR

PRIMARY GAS FLOW CALIBRATOR

THE mini-BUCK CALIBRATOR®

Instruction Manual

FOR

Model M-5: Range: 5.000 to 5500 Cubic Centimeters/Minute

Model M-30 Range: 4.000 to 25.00 Liters/Minute

BY

A. P. BUCK, INC.
3139 South Orange Avenue
Orlando, Florida 32806
(305) 851-8910

INSTRUCTION MANUAL

TABLE OF CONTENTS

SECTION		PAGE
1	WARRANTY & LIMITATION OF LIABILITY	2
2	ANALYSIS OF AIR FLOW CALIBRATION	
	a. Physics of Measurements	3
	b. Accuracy of Gaseous Flow Measurements	4
3	PRINCIPAL OF OPERATION and FEATURES	5
4	START UP PROCEDURE	6 & 7
5	CALIBRATION OF AIR FLOW SOURCES WITH THE mini-BUCK	8 & 9
6	MAINTENANCE OF THE mini-BUCK CALIBRATOR	10 & 11
7	VERIFICATION OF THE CALIBRATOR	12
8	POWER SUPPLY	12
9	PRECAUTIONS/WARNINGS	13
10	PARTS LIST and SERVICE POLICY	14

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Section 2

Analysis Of Air Flow Calibration

a. Physics of Measurements:

At room temperature, all gases obey — to a very close approximation — the ideal gas equation $PV = nRT$, where P is the newtons per square meter, V is the volume in cubic meters, n is the number of moles of gas, R is the empirically determined gas constant with the approximate value 8.31 joules/mole degrees Kelvin and T is the temperature in degrees Kelvin.

The method of measuring flow rates in the mini-Buck Calibrator is the technique known as the soap film bubble test. A frictionless soap is suspended perpendicular to the air flow up a small bore tube. This film forms a complete seal across the tube. The effect causes the soap film to move along the tube at exactly the same rate as the air flow. Knowing a measured distance and tube bore size a volume can be calculated. Thus a flow rate can be determined by the movement of soap film across a fixed volume per unit of time. This technique is classified as a Primary Standard.[1]

By comparing the four variables of the Ideal Gas Law against this detection technique, the validity of measurement can be accessed. The volume (v) is known and fixed therefore cannot change or be considered variable. The mass (m) of the gas being measured is not changed in this technique as the soap film is simply suspended across the tube crossection and moves with the gas flow rate. Mass is not affected. This leaves pressure and temperature. These are expressed by Boyle's Law $P(1)V(1) = P(2)V(2)$ and Charles Law $V(2)/V(1) = T(2)/T(1)$. No pressure changes from the ambient during a test as the soap film is practically frictionless. Temperature has no influence in this type of flow measurements when all elements are at an ambient. This includes the Calibrating device, the flow of gas and room temperature. These conditions are the general circumstances in which tests are performed.

Conclusion: The detection method of measuring flow rates of gases over a fixed volume per given unit of time is for all practical purposes independent of all variables in the Ideal Gas Law. Thus, the mini-Buck Calibrator® serves as a primary standard calibration method.

[1]Lippman, Morton, "The Industrial Environment — its Evaluation and Control," [U.S. Department of Health, Education and Welfare], NIOSH, 1973, Ch 11, 101pp

b. Accuracy of Gaseous Flow Measurements

To properly evaluate the accuracy of flowing gases two parameters must be considered. First, the steadiness of the flow rate must be known. Practically every type of pump creates some *pulsing of the flowing gases. Second, fact is the rate of flow can drift up and down over some* range. Current battery powered personal air sampling pumps use various techniques to dampen *pulsing and special circuitry to monitor pump speed, thereby, attempting to generate constant* and steady flows.

The mini-Buck Calibrator® is an automation of the "Classic" technique of using a soap film seal to measure flow over a predetermined volume in a known time. A microprocessor, operating at 6 megahertz per second, detects the passing of the soap film seal over the established flow tube volume and automatically calculates the rate of flow. The typical apparatus, a 1,000 ml buret using a stop watch can be significantly reduced in size since the microprocessor can detect and measure the speed at 80 microsecond intervals. Compare this speed to a technician's response time. With good precision on a stopwatch he or she could be repeatable within 50,000 micro-second (0.05 seconds). Over 500 times slower than the microprocessor.

Consider this analysis: 1,000 cc buret NBS traceable; 0.01 second stopwatch crystal controlled *with clock accurate to +/- 15 sec per month; 1,000 cc/min steady flow source (constant flow* +/- 1 cc/min)

Example: Measure the flow rate using 1,000 cc buret by a skilled technician. All devices are at a constant room temperature.

Test	Time	Actual Flow Rate
1	*60.06 sec*	*999.0 cc/min*
2	60.00	1000
3	59.94	1001

Note: At 1,000 cc/min of flow +/- 0.06 is equal to 1 cc/min change or +/- 0.1% accuracy. Repeatability is strictly a function of the technicians skill.

Mathematically it would seem 0.1% accuracy could be obtained using this large volume and a skilled technician with a 1000 cc/min. steady source. Of course, if the volume is smaller than 1,000 cc or the flow is faster, the percent of accuracy is further reduced by this manual method.

To summarize, the accuracy of measuring air flow relies on quality measurement tools, NBS traceable buret, crystal control stopwatch, constant air flow source and, finally, the method of detection.

Section 3

Principle Of Operation And Features

A. Detection Principle:

The mini-Buck Calibrator® utilizes the principle of measuring the flow rate of gases over a fixed volume per unit of time. A Quartz controlled timer is the timing device and the fixed volume is located in the flow cell center tube. A microprocessor measures the time for a frictionless soap film to travel from the first sensor to the second sensor (infrared which detects the passage up the tube) and then calculates the volume per unit of time. The results are displayed in flow rate, cc/min. for the model M-5 and liters/min. for model M-30 on a four digit liquid crystal display. The decimal point floats to present the data in the proper range. The timer is capable of detecting a soap film at 80 microsecond intervals. This speed allows under steady flow conditions an accuracy of +/- 0.5% of any display reading. Repeatability is +/- 0.5% of any display. The unique flow cell can create a soap film over a range of 4 cc/min. to 5500 cc/min. for the M-5 and 4 to 25 LPM for M-30. The soap in both models is spillproof when properly filled.

B. Understanding the Display Results

A unique feature of the microcomputer software program is data display. On initiation, the mini-Buck display will present 0000's with no decimal point indicated. A series of ---- are displayed while a test is in progress. During sequential tests the previous reading is averaged with the current test and displayed. If the previous reading and current reading are different by more than +/- 5% a series of EEEE's will flash four times on the display and then present the actual last test result. The next flow readings will continue this averaging technique over again. Flows greater than 5500 cc/min. on M-5 and 25 LPM on M-30 will display EEEE's. The flashing numbers on the display after a test are for three seconds duration. The purpose is to allow sufficient time for soap to return to the bottom reservoir.

C. Battery saver

Another feature of the mini-Buck is the battery saver "automatic shut down." If a test is not conducted within approximately 7 minutes from turn on, the Calibrator will turn itself off. Continuous use of the batteries is rated at 3 hours. This can be extended to 6 hours by using the A/C charger.

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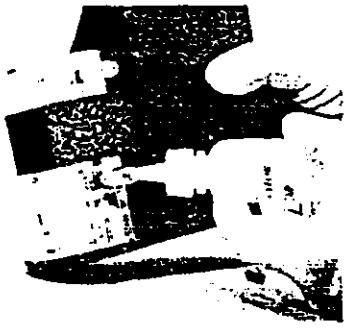
Section 4

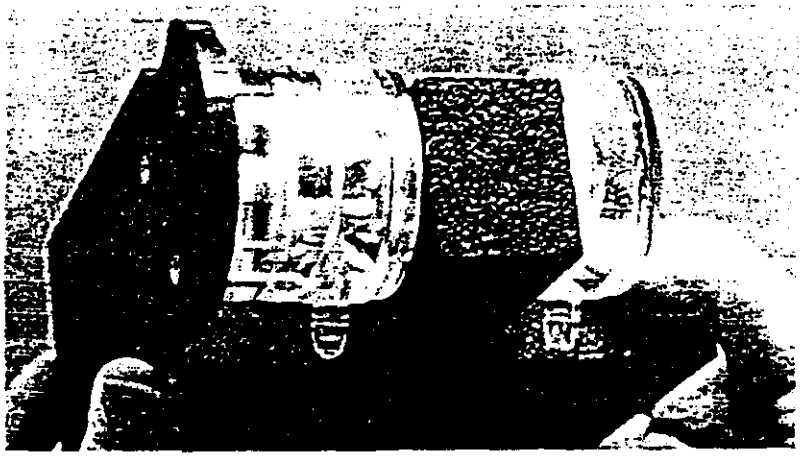
Start Up Procedure

A. Read the previous section on Principle of Operation if unfamiliar with device.

B. Add soap solution (Soap to bottom hose nipple)

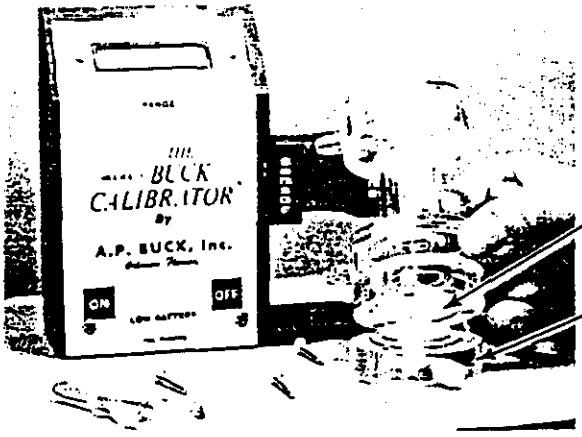


C. Check for the correct amount of soap by turning unit in such a position as to pour the soap out of the bottom nipple. If the chamber is overfilled, the excess will run out leaving the proper level.



D. Wet flow cell by connecting a pump flowing at 1000 to 2000 cc/min. for Model M-5 and 5 LPM for Model M-30. Initiate soap film up center tube by rapidly pressing white button in and releasing. Repeat this procedure until bubble doesn't break. The tube is now wet and tests at any range may be conducted. One minute warm up time is all that is required of the mini-Buck.

mini-BUCK CALIBRATOR® PACKAGE



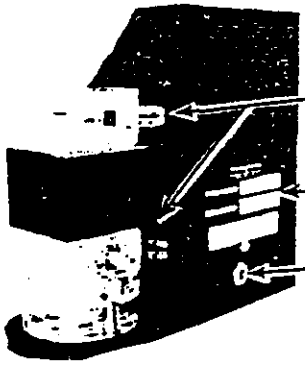
BASIC FLOW CELL

Diaphram in place for assembly on bottom of flow cell.

Flow cell base plate with plunger, lift plate and cam screw.

Screwdriver supplied fits all screws used in the mini-Buck.

REAR VIEW



Hose Nipples: Top Exit
Bottom Inlet

Location of Model Numbers and Serial Numbers.

A/C Adapter Jack

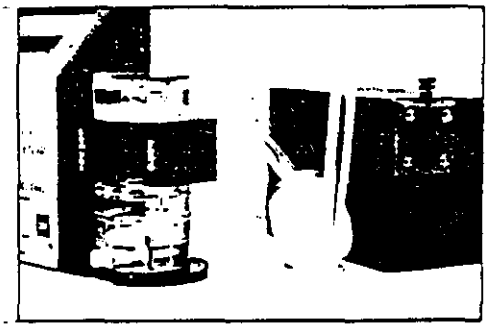
Calibration Of Air Flow Sources With The mini-Buck

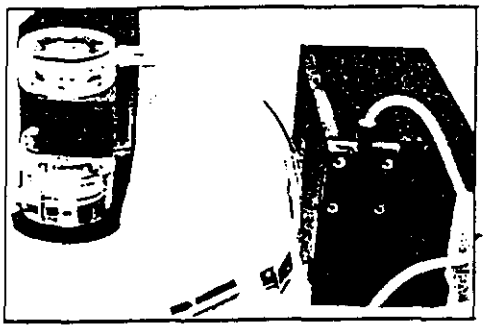
These instructions apply to both the Model M-5 and Model M-30. The range for each are:

Model M-5 ≈ 5.000 to 5500 cc/min.
Model M-30 ≈ 4.000 to 25.00 liters/min.

The instructions relate to industrial hygiene air sampling. It basically applies to any gases flow rate measurements.

1. Wet the flow cell as described in Section 4.
   Obtain proper hose adaptors for connecting tubing to flow cell.
2. Start pump and allow approximately 10 minutes to stabilize.
   Perform this step before connecting to flow cell as air flow dries center tube if bubbles are not initiated at intervals.





3. The top hose nipple of the flow cell is connected to the end of the sampling media (filter cassette, charcoal tube or bubbler).
4. Connect sampling median to pump.
5. Begin Calibration:
   Depress white plunger into cell and quickly release.
   Different flow rate can require quicker or slower release of the button.

6. Carefully observe the bubble passing through the sensor zone. Only a single straight bubble, perpendicular to the tube wall, is necessary for an accurate test. If several bubbles go up the tube at once it will not effect the test data because the first sensor will not reset until the final sensor has been tripped. A good technique is to watch the bubble pass properly across the sensor zone to ensure a good test has been conducted.

7. Observe the display. A number will be displayed. The decimal point will be appropriately placed. Read section 3-B on features concerning data display.

8. After a 3 second delay from the time the final sensor is tripped another test can be performed.

9. Repeat Step 5 through 8 for minimum of 3 tests.

Note:
If difficulty is encountered in making a clean single soap film rise up the center tube:

a. Check level of soap in flow cell (Section 4-C).

b. If soap is cloudy, (not clear), change the soap solution.

c. The diaphragm may need replacing at this time.

Section 6

Maintenance Of Calibrator

General Information:

Do not store flow cell for periods of one week or more with soap in cell. To store, remove bottom plate and diaphragm of flow cell and flush with tap water. Allow to air dry. The acrylic flow cell can be easily scratched. Wipe with cloth "only." Do not allow center tube, where sensors detect soap film, to be scratched or get dirty. NEVER clean with ACETONE. Use only soap and warm water. On stubborn dirt use alcohol as a last resort.

Procedure for Installing the Diaphragm

Dismantle the Flow Cell by:

Step 1. Loosen plunger cam screw, 3 to 4 turns.

Step 2. Lay unit on its left side, remove the 3 flow cell screws from the case bottom.

Step 3. Stand up carefully not to allow cell to fall from case. Remove flow cell from sensor block, pull base off and dispose all of soap (clean flow cell and allow to air dry).

Step 4. Put new diaphragm in recess of flow cell body (wetting, diaphragm with a small amount of soap to aid holding in place).

Step 5. Hole in lift plate must be to plunger side of bottom plate.

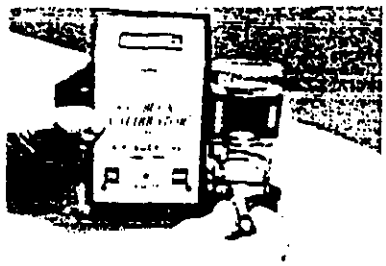
Step 6. Align holes in bottom plate and flow cell with plunger and nipples in proper orientation *then slip together. Avoid twisting bottom plate, as this may wrinkle diaphragm.*

Step 7. Holding flow cell and bottom plate together, slip flow cell into unit.

Step 8. Install the three bottom flow cell screws. Tighten the screws evenly. See step 2 of photographs.

Step 9. Lay unit on its back. Insert screwdriver (provided) into bottom of case and push diaphragm up until plunger may be pushed in, hold plunger and tighten plunger cam screw (DO NOT OVER TIGHTEN). Refill with soap.

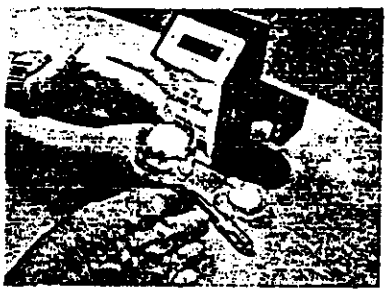
PROCEDURE FOR INSTALLING DIAPHRAGM



Step 1

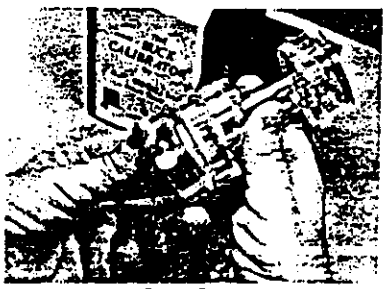
Step 2

Step 3

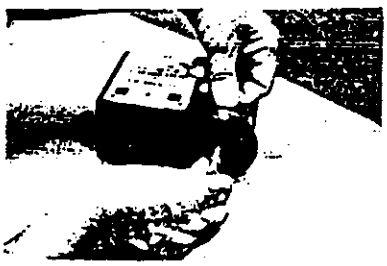


Step 4

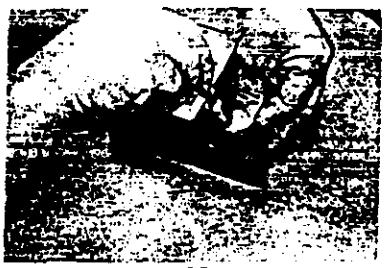
Step 5



Step 6



Step 9A



Step 9B

Section 7

Verification Of Calibration

The calibrator is factory calibrated using a standard traceable to National Bureau of Standards (N.B.S.). Attempts to verify calibrator against a glass one liter burette should be conducted at 1000 cc/min. for maximum accuracy. The calibrator is linear throughout the entire range.

Factory reverification of N.B.S. will be conducted as a service to any mini-BUCK owner. The charge for calibration including return postage is $80.00.

Section 8

Power For The mini-Buck Calibrator®

1. Battery power is 4 Nicad AA batteries.
2. Low battery light will indicate the power supply is too low for accurate results.
3. Charge battery pack for 16 hours minimum using the A/C adapter charger. Connect to 110 VAC 50 or 60 Hertz outlet. A fully charged battery pack will provide up to three hours of continuous operating time.
4. The mini-Buck Calibrator® may be operated with the AC adapter plugged in, thus *extending the operating time to twice the normal run time of three hours. However,* it is not designed for continuous AC adapter operation.
5. A special power saving feature is the Automatic Shut Off. After 7 minutes of non-use *the mini-Buck will shut off automatically.*

Section 9

Precautions/Warnings

1. Avoid the use of chemical solvents on flow cell or calibrator case and faceplate. Generally, soap and water will remove any dirt.
2. Never plug intake hose connector of flow cell while pulling gases through the cell. A reduced pressure within the cell will cause the diaphragm to be drawn to the top of flow cell and this will cause the diaphragm to rupture.
3. Never pressurize the flow cell at any time with more than 25 inches of water pressure.
4. Do not charge batteries for longer than 16 hours. Do not leave A/C adapter plugged into calibrator when not in use as this could damage the battery supply.
5. The mini-Buck Calibrator® soap is a special blend of soap to provide a clean film throughout the operating range and prevent exiting of soap in tubing. The use of any other soap is not recommended.
6. Black hose fitting covers help to reduce evaporation of soap in the flow cell when not in use.
7. Do not store flow cell for a period of one week or longer with soap. Clean and store dry (see section 6).

C. QUALITY ASSURANCE PLAN FOR THE ANALYSIS OF VOLATILE ORGANICS ON TENAX/TENAX-CHARCOAL SORBENT TUBES

The following Quality Assurance Plan describes the analysis method to be used by Radian in analyzing air and landfill gas samples collected at the Janesville Disposal Facilities. Within the plan, several options are to be specified by the user of the analytical service. These items are addressed below.

1) Sampling Procedures

   Sampling will be performed by Warzyn Engineering. Equipment used and method of sampling are given in the Sampling Plan.

2) Analysis of the Sampling Train

   Analyses may be performed separately on the individual Tenex and Tenex-charcoal tubes making up the sampling train or by a single analysis of the combined tubes. For this study, analyses will be performed on the combined tubes.

3) Target Compounds

   The target compounds are those listed in Table 4. The normal combination range of 25 ng to 450 ng will be used. The compound cis-1,2-dichloroethene has been added to the normal list of compounds in Table 4. This addition has been confirmed with the Mr. Jim Homolya of Radian Corporation (919-541-9100).

4) Tentatively Identified Compounds

   Warzyn requests tentative identification and estimated concentrations for the five highest concentration compounds not listed in Table 4.

5) Data Reporting

   All data are to be reported, including those for all blank samples. Data will not be blank corrected. Criteria for rejection of data will be followed as listed in the Quality Assurance Plan. Rejected data will be reported and noted as rejected.

RCW/jap/DWH
[jap-600-65aa]

QUALITY ASSURANCE PLAN

PREPARED FOR:

Warzyn Engineering, INC.

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JUNE 1987

2.0 TABLE OF CONTENTS

SECTION		PAGE(S)
1.0	TITLE	1
2.0	TABLE OF CONTENTS	2
3.0	LIST OF TABLES	3
4.0	INTRODUCTION	4
5.0	PROJECT DESCRIPTION	4
6.0	PROJECT ORGANIZATION AND RESPONSIBILITIES	4
7.0	QUALITY ASSURANCE OBJECTIVES	5
8.0	SAMPLING PROCEDURES	8
9.0	SAMPLE CUSTODY AND DOCUMENTATION	9
10.0	CALIBRATION PROCEDURE AND FREQUENCY	11
11.0	ANALYTICAL PROCEDURES	11
12.0	DATA REDUCTION, VALIDATION, AND REPORTING	16
13.0	INTERNAL QUALITY CONTROL CHECKS	17
14.0	PERFORMANCE AND SYSTEM AUDITS	20
15.0	SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS	21
16.0	CORRECTIVE ACTION	21
17.0	QUALITY ASSURANCE REPORTS TO MANAGEMENT	22
18.0	SAFETY	22

3.0 LIST OF TABLES

TABLE #	TITLE	SECTION	PAGE
1	Interlaboratory Tenax™ Performance Evaluation	7.0	7
2.	Analytical Conditions for Analysis of Sampling Cartridges	11.0	12
3.	Summary FC-43 Tune % Relative Abundance	11.0	13
4.	Calibration Compounds for Air Toxics Analysis	11.0	14
5.	Limits of Detection of Compounds of Interest	13.0	19

4.0 INTRODUCTION

The purpose of this document is to describe the Quality Assurance Program for air toxic analytical measurements performed by the Radian RTP Analytical Laboratory. This plan outlines the policies, organization, functional responsibilities, and general operating procedures designed to ensure that measurement efforts results in valid data, of known data quality. This plan is presented in the general format prescribed for Quality Assurance Project Plans the Environmental Protection Agency (EPA Document QAMS-005/80).

5.0 PROJECT DESCRIPTION

The objective of this Quality Assurance Plan is to describe the procedures which are implemented for the analysis of air toxics samples and to ensure that data acceptability criteria are imposed and obtained on a routine basis.

The aim of this Quality Assurance Plan is to provide Warzyn Engineering with timely delivery of data of the highest possible quality. Samples of air will be provided to Radian and analyzed for a given set of constituents which are of environmental concern. These analyses will be performed to support environmental sampling activities conducted by Warzyn Engineering.

6.0 PROJECT ORGANIZATION AND RESPONSIBILITY

In the Radian organization, Mr. James B. Homolya will serve as Program Manager for activities performed under contract to Warzyn Engineering, Inc. Mr. Homolya will supervise and administer all contractual requirements which will include monitoring Radian's budget and schedule of performance. He will: 1) keep Warzyn Engineering informed on all aspects of the program including expenditures, problems, and recommended solutions; 2) be available to Warzyn Engineering for action on any problem; 3) be available to the Project Director (Dr. D. E. Wagoner) for any action on any problem requiring additional management or technical support; and 4) keep Radian corporate management informed on all matters relating to the program.

Dr. Denny E. Wagoner, Project Director, will work directly with Mr. Homolya, the Program Manager, to schedule and direct the analytical requests during the project. Dr. Wagoner will be the technical manager with overall responsibility for ensuring the technical quality of the analytical programs performed for Warzyn Engineering in the Radian Perimeter Park Laboratory.

Dr. Joan T. Bursey, Senior Technical Advisor, is available to the Project Director for consultation on any technical problems arising from the analysis of samples and

interpretation of data for this program. Dr. Bursey also provides review of analytical data and review of data packages prior to delivery of the packages to the Project Director for final review and delivery to the client. Dr. Bursey also organizes and coordinates the Quality Assurance efforts on the Warzyn Engineering program.

Mr. Randall L. Porch, Supervisor of the Gas Chromatography/Mass Spectrometry Laboratory, is responsible for the scheduling and supervision of day-to-day analytical activities for the sample analysis portion of this program. Working under Mr. Porch's supervision are the operators of the GC/MS instrumentation, Mr. Mitchell Howell, Mr. James Carleton, Mr. Ron Harris, and Mr. David Kusel. Mr. Porch is responsible for the scheduling of activities in the GC/MS laboratory and for the delivery of quality analytical results in a timely manner. Mr. Porch supervises the analyses and provides an initial review of data and organization of the data packages. Mr. Tom Buedel works under Mr. Porch's supervision in the generation of components of the data packages and in the initial organization of the data package. Mr. Porch and the system operators in collaboration provide the primary level of quality control for the program: the analysts, under Mr. Porch's supervision, assure that quality criteria are tested and met prior to the initiation of any analyses in any given day.

Mr. James F. McGaughey, Operations Manager, is responsible for the coordination of effort in the preparation of sampling cartridges. The tubes are packed, conditioned, and tested for cleanliness under Mr. McGaughey's supervision.

Ms. Candace Blackley and Ms. Leslie Cooper work under Mr. McGaughey's supervision in the preparation and quality control for the sampling cartridges. Ms. Paula Crabtree, Sample Control Officer, is responsible for the shipping of prepared sampling cartridges to the field staff for sampling. Ms. Crabtree also logs in samples received, both into the laboratory Master Log and the computerized Sample Analysis and Management (SAM) System used in the Radian laboratories. Ms. Crabtree generates report forms through SAM which are included as part of the data package ultimately delivered to the client.

7.0 QUALITY ASSURANCE OBJECTIVES

The analysis of air samples is always subject to a matrix effect. Within the constraints of this matrix effect, various observations may be made. The level of moisture is always a primary consideration in analytical sampling of air, since the presence of water has a deleterious effect upon most analytical instrumentation. The effects which are encountered range from a minor distortion of chromatographic peaks because of water entering the analytical system to a

complete shutdown of the entire analytical system because of a vacuum surge resulting from the entrance of water into the instrument. The existence and extent of introduction of water into the analytical system is outside the control of the analyst. However, considering the matrix effects, the following Quality Assurance goals can be set for precision, accuracy, and completeness.

There is usually far more difficulty associated with producing truly replicate air samples than with producing replicate results. Precision is defined as the numerical measure of the reproducibility of an analytical measurement, as expressed by the coefficient of variation obtained when at least three replicate analytical measurements are compared. When replicate sampling cartridges are spiked, precision as expressed by coefficient of variation of replicate (at least three) analyses is typically in the range of 15-30% and the precision is compound-specific. For compounds such as toluene, chlorobenzene, ethylbenzene, hexane, heptane, the xylenes, precision data will tend toward the lower part of the range cited above. For other compounds such as chloromethane, bromomethane, chloroethane, vinyl chloride, the dichlorobenzenes, acetone, and 2-butanone, precision will tend toward the higher part of the range and may, at times, extend above the higher part of the range. A historical database is the best predictor of the range of precision which should be attainable in any given set of circumstances. When the effects of matrix are overlaid with the effects of compound-to-compound variation, the range of precision may be expected to be at least 15-40%, chiefly due to the presence of water and the effects which the water has upon the analytical determination.

Accuracy is defined as the correspondence of observed values to true values, usually related to spiked samples. Air toxic sampling and analysis programs to date have not been characterized by the use of externally prepared audit samples, so no historical database is available for prediction of accuracy or bias values. Two assessments of an expected value for accuracy can be given. In the analytical process, each sample is spiked with surrogate compounds, which are isotopically labeled or fluorinated analogs of compounds of interest. The compounds used as surrogates are d_{10}-ethylbenzene, perfluorobenzene, and perfluorotoluene. A typical range of recoveries for these compounds is 50-100%, depending upon the moisture which is introduced with the samples. Use of a laboratory or field blank where the moisture problems are not so severe as actual field samples will usually raise the lower part of the range to 75% or better. An additional measure of accuracy is provided by the analysis of audit samples which have been provided by EPA prior to the initiation of a sampling and analysis program. The results shown in Table 1 were obtained for analysis of triplicate spiked cartridges.

Table 1. Interlaboratory Tenax Performance Evaluation

Compound	Spiked Value	Observed Value #1	#2	#3
chloroform	108	111	100	100
1,1,1-trichloro-ethane	293	314	245	251
carbon tetra-chloride	233	216	236	204
benzene	384	348	328	312
1,2-dichloro-ethane	366	356	311	315
trichloroethene	320	285	262	265
1,2-dichloro-propane	168	159	147	135
toluene	441	409	391	317
tetrachloroethene	354	329	294	252
chlorobenzene	483	398	393	153
ethylbenzene	315	261	279	47
o-xylene	384	265	385	16
bromoform	420	266	371	35

Note that, in these audit samples, the percent difference observed between spiked values and observed values tends to increase with decreasing volatility of the compound of interest. The increase in this parameter reflects the decreasing efficiency of the desorption from Tenax. The values can still be quite reproducible, however, even with decreasing efficiency of desorption. Thus, in this series of audit samples, an average percent difference for all samples of -20.4 is observed, and the range of compounds studied is sufficient to consider 20% a reasonable range for the assay as a whole. However, this is an average range only, and specific values for specific compounds may range from -4% to -50% while the assay is performing with all analytical parameters in control. The accuracy expected for the assay is also compound-dependent, with the desorption efficiency of the more polar compounds being lower and more variable.

Completeness is a measure of the number of acceptable assays relative to the number of assays which can possibly be performed. The goal of 100% completeness can sometimes not be attained, however, because of instrument or power failures and other circumstances, so a goal of 90% for completeness is reasonable and ought to be attainable in most circumstances, and > 95% will be attainable in most circumstances.

8.0 SAMPLING PROCEDURES

Sampling is not an activity which will be conducted by Radian personnel in the Warzyn Engineering program. The extent of Radian involvement in sampling will be to supply Tenax® and Tenax®/charcoal tubes to Warzyn Engineering sampling teams. The procedures for preparation of these tubes and the quality control associated with the procedure are described below.

All tubes are prepared by packing and thermal desorption for a minimum of 24 hours at 185°C. After desorption, the tubes are removed from the oven, stored in individual containers, and blanked using gas chromatography with flame ionization detection with cryotrapping. At least 50% of the tubes are blanked individually. Tubes are then re-packaged and shipped to Warzyn Engineering with Chain of Custody forms, sample seals, and special instructions for handling of the tubes.

The assembly and packing of sorbent cartridges is carried out in an area which is free of volatile organic material, in a laboratory in which no organic solvents are handled or stored, and in which the laboratory air is charcoal-filtered. New Tenax® is Soxhlet-extracted for 24 hours with methanol (Burdick and Jackson, Pesticide Grade or equivalent), then dried for 6 hours in a vacuum oven at 50°C prior to use. Cartridges packed with Tenax® are thermally conditioned by flowing organic-free nitrogen at a rate of 30 mL/min through the resin while heating to 185°C for 24 hours. After thermal conditioning, the sorbent cartridges are stored for 24 hours at room temperature prior to monitoring the cartridges for residual organic contamination. If the cartridges have collected high concentrations of organic compounds in field use (microgram levels of organic materials), the sorbent will require Soxhlet extraction prior to use for another field application. Because of problems with possible charcoal contamination, charcoal is not reused. New charcoal is prepared as Tenax® is prepared.

Glass tubes and culture tubes for holding the tubes in shipment are cleaned with a non-ionic detergent in an ultrasonic bath, rinsed well with organic-free water, and dried at 100°C. Metal parts (fittings to cap the ends of the sampling tubes and to make connections to the sampling apparatus and the analytical instrumentation) are cleaned in an ultrasonic cleaner with a warm non-ionic detergent solution, rinsed with organic-free water, air dried, and heated in a muffle furnace for 2 hours at 400°C. Glass wool used in packing the sampling tubes is Soxhlet-extracted using pentane for 8-16 hours, and oven-dried at 110°C before use. The glass wool is checked before use to be sure that all residual pentane has been removed. Nitrogen gas used in the drying and quality control of sampling tubes is organic-free (Linde-Union Carbide, 99.999% pure, hydrocarbon-free, or

equivalent). Cryogenic traps are used to ensure purity.

In the preparation of the sampling cartridges, approximately 1.6 g Tenax® is weighed and packed in a sorbent tube and held in place with glass wool. The Tenax® tubes are stored and transported in culture tubes with Teflon®-lined screw caps, within a clean metal container which contains clean charcoal. The storage container is kept cold using ice or cold pack storage. Tenax®/charcoal tubes are packed with an approximately 3:1 volume ratio of Tenax® and charcoal. The cartridge is assembled so that the inlet side during sample collection is Tenax®, followed by a layer of charcoal at the outlet side of the sorbent cartridge. The sorbents are held in place in the cartridge by glass wool. End caps are placed on the assembled cartridges and the Tenax®/charcoal cartridges are packed and stored by the procedure used for Tenax® cartridges.

The following Quality Control measures are used to ensure that the sorbent cartridges are free from background contamination prior to sample collection. Gas chromatograph with flame ionization detection is calibrated using direct injection of propane standards. The chromatographic column is 1.8 m x 0.25 cm ID glass packed with 1% SP-1000 on Carbopack (60/80 mesh). Following assembly and leak-checking of the sampling cartridges, the cartridges are connected to a source of organic-free nitrogen. Nitrogen is passed through each sampling cartridge at a flow rate of at least 30 mL/min, while the cartridges are heated to 200°C. Every sampling cartridge receives the conditioning with nitrogen under heat; 50% of the cartridges are checked by flame ionization gas chromatography after the conditioning to ensure freedom from contamination. If in using this procedure, the background exceeds 10 ng, the cartridges must be recleaned and reanalyzed.

9.0 SAMPLE CUSTODY AND DOCUMENTATION

With every set of sampling tubes which leave the Radian laboratory, a set of Chain of Custody forms and sample seals is furnished to the field sampling personnel. Upon receipt of exposed sampling tubes at the Radian laboratories, the Chain of Custody forms are signed and dated by the Sample Custodian. The sampling tubes are then logged into the laboratory Master Log, with subsequent entry into the computerized laboratory Sample Analysis and Management system. After the sampling tubes have been logged into the Master Log, the tubes are relinquished to the Mass Spectrometry Laboratory Supervisor (or his designee), to be placed into the freezer especially designated for Tenax® tubes. When the samples are analyzed, the analyst is the one who breaks the Sample Seal to remove the tubes from the containers in which they have been shipped. The sample seal is affixed to the chromatogram generated from the sample in

the analyst's laboratory project notebook. This sample seal is subsequently duplicated for inclusion in the ultimate data package. After samples have been analyzed, the analyst obtains the initial quantitative data. The project notebook with all data therein is subsequently relinquished to the Data Control Officer, who generates the other information required for the complete data package which is delivered to the client. When a data package is complete, the package is reviewed by the Mass Spectrometry Laboratory Supervisor and by the Senior Technical Advisor, prior to delivery. The Project Director (Radian) also reviews the data package. The laboratory notebook is signed and dated by reviewing personnel, and the data sheets produced by the Sample Analysis and Management system are signed by a reviewer.
The documentation which is supplied in a data package includes:

1) a summary table prepared from the individual samples
2) a table which contains a summary of the surrogate recoveries for all of the samples and blanks analyzed in a given batch of samples
3) a description of the experimental procedure, which typically is not varied
4) a description of the analytical conditions, which are maintained as nearly constant as possible
5) tabulated tuning data for every day of analysis
6) a calculation of estimated limits of detection, which are calculated from one of the calibration samples which are injected from a static dilution bulb and thus represent the best possible conditions
7) a table which shows the response factor database(s), including the mean of all of the calibration runs, the standard deviation, and the coefficient of variation. In addition, all of the chromatograms and all of the hardcopies of the computer reports for each individual analysis of standards are reported
8) daily calibration checks are tabulated for comparison to the mean of the response factor database
9) chromatograms and computerized reports of the quantitative analysis for all samples and blanks are reported
10) mass spectra of the compounds of interest from each datafile (other than the calibration analyses) are reported
11) when additional compounds are identified, copies of the mass spectra and copies of the computerized library search are supplied. In addition, estimated concentrations are supplied for the other compounds identified
12) report forms printed by the Sample Analysis and Management system are supplied
13) Chain of Custody forms and copies of the sample seals are also supplied with the report.

Thus, the entire history of the sample through the Radian Perimeter Park laboratory is documented through the data package.

10.0 CALIBRATION PROCEDURE AND FREQUENCY

The GC/MS system is calibrated by gaseous injections from a static dilution bulb prepared according to EPA TO1. For the initial database, a three-point calibration is used, subject to acceptability of the coefficient of variation. If an acceptable coefficient of variation is not observed after three injections, additional injections are performed until an acceptable coefficient of variation is obtained and then the best three points are used for a response factor database. The calibration is checked daily, using a single injection from the static dilution bulb, and checked for acceptability. If the check of the calibration shows that the calibration database is still acceptable, samples are analyzed on that day. If the daily calibration check shows that the original database is not valid, a second check is made. If the second check is acceptable, samples are analyzed. If the second check is not acceptable, the calibration and generation of the response factor database is repeated. The frequency of calibration is thus a variable depending on the length of time before the daily check of the calibration fails to meet the specifications for acceptability. A calibration database has been observed to maintain acceptability for a week or longer, and recalibration has also been required after a single day. The determining factor is usually external parameters such as power failures or other types of instrument failure.

11.0 ANALYTICAL PROCEDURES

The procedures for sample analysis are adapted from EPA Method TO1 ("Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air," R. M. Riggin, EPA-600/4-84-041, April, 1984). Method TO1 as written allows considerable flexibility in instrument configuration and operating conditions; the procedures used by Radian are described below.

Field samples taken on Tenax® and/or Tenax®/charcoal are analyzed by thermal desorption into a gas chromatograph/mass spectrometer/data system. The actual cartridges used for sampling are glass, in the same configuration as the tubes used in the analysis of emissions samples taken by the Volatile Organic Sampling Train (VOST). (Sampling cartridges are analyzed either singly or as a Tenax®/charcoal pair according to client request. The analytical conditions are shown in Table 2.

Table 2. Analytical Conditions for Analysis of Sampling Cartridges

Instrument	Finnigan-MAT 4500
GC Column	DB-624 megabore fused silica capillary, 30m
Carrier Gas	Helium
Carrier Gas Flow Rate	2.2 mL/min
GC Program	5°C for 2 min, then 6°C/min to 200°C, hold for 10 min
Electron Energy	70 eV
Ionizer Temperature	150°C
Interface Oven Temperature	200°C
Injector Temperature	100°C
Manifold Temperature	100°C
Analytical Trap, Tekmar LSC2	5% Carbotrap®/25% Carbosieve S-III (w/w)
Cartridge Purge Time	10 minutes
Purge Gas	Helium
Cartridge Purge Gas Flow	40 mL/min
Analytical Trap Desorb Time	4 minutes
Analytical Trap Desorb Temperature	180°C
Trap Bakeout Time	15 minutes
Trap Bakeout Temperature	200°C

In the analytical procedure for the sample cartridges, Tenax® and Tenax®/charcoal tubes are heated to 180°C and purged with 40 mL/min of helium for 10 minutes, thus directing organic constituents through a heated 5 mL purge flask (90°C) onto an analytical trap composed of 75% Carbotrap®/25% Carbosieve S-III (w/w). A quantitation standard and surrogate compounds from a static dilution bulb are spiked through a side arm of the purge flask at the beginning of the purge cycle. The trap is then backflushed with helium for 4 minutes and the organic compounds directed to the head of a 30m DB-624 megabore fused silica capillary column with a helium flow rate of 2.2 mL/min. The megabore column is directly interfaced to a heated transfer line in the GC/MS interface oven.

In initiating analysis, the mass spectrometer is tuned using perfluorotributylamine to the criteria recommended by the instrument manufacturer. A mass spectrum and a tabular listing of the mass spectral data for the tuning compound are obtained for every day of analysis. After the initial day when the instrument is tuned, the tuning compound (perfluorotributylamine) is monitored daily prior to the initiation of any analysis to verify that the relative abundances of all of the key peaks are within 25% of the

values obtained when the initial tune of the instrument was performed. If the relative abundance of one of the key peaks of the tuning compound is more than 25% different from the value obtained for the previous day's tune, the instrument is retuned to conform with the previous day's relative abundances for key peaks. If the instrument cannot be retuned to match these criteria in a reasonable amount of time, the instrument is tuned and a complete calibration is performed. A representative table of tuning data for a series of samples is shown in Table 3.

Table 3. Summary FC-43 Tune % Relative Abundance

	% RA	
M/E	3/2/87	3/3/87
51	0.19	0.20
69	100.00	100.00
100	12.88	13.04
119	10.48	10.32
131	33.98	33.37
169	3.72	3.93
219	32.36	33.67
264	8.86	9.37
314	2.07	2.53

After the tune has been established, a three-point calibration is performed by gaseous injection from a static dilution bulb. The compounds shown in Table 4 are in gaseous solution in the heated bulb.

Table 4. Calibration Compounds for Air Toxics Analysis

trichlorofluoromethane
methylene chloride
1,1-dichloroethane
1,1,1-trichloroethane
benzene
trichloroethene
bromochloromethane
toluene
1,1,2-trichloroethane
dibromochloromethane
ethylbenzene
o-xylene
1,1,2,2-tetrachloroethane
p-dichlorobenzene
chloromethane
chloroethane
heptane
styrene
2-butanone

1,1-dichloroethane
t-1,2-dichloroethane
chloroform
carbon tetrachloride
1,2-dichloroethane
1,2-dichloropropane
t-1,3-dichloropropene
c-1,3-dichloropropene
tetrachloroethene
chlorobenzene
m-, p-xylene
bromoform
m-dichlorobenzene
o-dichlorobenzene
bromomethane
vinyl chloride
isopropylbenzene
acetone
hexane
c-1,2-dichloroethane

Surrogates:
d_{10}-ethylbenzene
perfluorobenzene
perfluorotoluene

Quantitation Standard:
d_6-benzene

The three-point calibration typically covers a range of approximately 25 ng to 450 ng, but the exact range is flexible according to client request. The method of relative response factors is used, with the coefficients of variation of the calibration check compounds over this concentration range required to have a value of 20% or less. If this coefficient of variation is not attained with the original three data points, additional calibration data points are added until an acceptable calibration curve is obtained. This initial calibration curve provides the original database prior to the initiation of sample analysis. The initial sample analysis is a blank, and the laboratory and field blanks are checked prior to the initiation of sample analyses. If contamination is encountered at this point, the client is advised of the extent and nature of the contamination and a joint decision is made as to whether or not to continue analysis. If no contamination is observed, sample analysis is initiated.

If sample analyses span a period of several days or weeks, the initiation of analysis for each day requires that an acceptable tune be demonstrated. After a tuning check, a

calibration check is performed. A calibration sample is analyzed, and quality criteria are imposed on this calibration check before sample analysis is initiated. The quality criteria for the daily calibration check are:
1) gaseous components (chloromethane, bromomethane, chloroethane, and vinyl chloride) must be observed. Close criteria for these compounds on a day-to-day basis cannot be imposed because of their lack of stability in the static dilution bulb. The stability of the gaseous components under the conditions of sample storage and delivery for the calibration sample tends to be variable. However, if the gases cannot be observed at all, the calibration standard must be re-prepared and the calibration process repeated.
2) the calibration check compounds (chloroform, carbon tetrachloride, trichloroethene, toluene, tetrachloroethene, chlorobenzene, o-xylene, and isopropylbenzene) are checked to verify that the response factors obtained for these compounds are within 20% of the value obtained for the mean of the database.
3) the other calibration compounds are checked to be sure that no more than 3 compounds other than the calibration check compounds have a value which is more than 70% different from the mean of the response factor database.
If these criteria cannot be met, the calibration sample is re-analyzed. If the criteria still cannot be met, the calibration standard is prepared and analyzed again and the analytical system is recalibrated, if necessary, prior to the initiation of sample analysis. If the acceptance criteria are met, sample analysis is initiated and the values for response factors obtained from the analysis of the daily calibration sample are used for quantitative calculations for the samples analyzed on that day.

Samples received at the Radian Perimeter Park laboratory are logged into the Master Log Book by the Sample Control Officer. Custody of the samples is then relinquished to the Supervisor of the Mass Spectrometry Laboratory, who places the samples in a limited access freezer which is reserved exclusively for Tenax® samples. The samples are stored in this freezer until analysis. When the instrument operator is ready to analyze the samples, one container of samples is removed from the freezer and allowed to come to room temperature (to prevent cracking of the glass tubes during analysis). Samples are then placed into the desorption apparatus as rapidly as possible, to minimize the handling time. The possibility for contamination of the samples in the mass spectrometry laboratory is extremely limited, since no bulk solvents are handled in this laboratory and sample exposure time is extremely limited.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

The steps of data review described above are always included for any data package delivered to a client. In addition, the contents of a data package include:

1) a summary data table containing all the values obtained for analytes in all of the blanks and samples analyzed
2) a summary of surrogate recoveries for all blanks and samples analyzed
3) a description of the experimental procedure
4) a summary of the analytical conditions
5) a summary of the tuning data obtained for every day that analysis of a given series of samples was performed
6) a summary of the estimated limits of detection, calculated by extrapolating the signal level for a given compound which is estimated to correspond to an interpretable mass spectrum to the number of nanograms required to produce that signal level
7) response factor database (the response factor database obtained in the initial calibration, as well as any response factor databases generated by subsequent recalibration, are tabulated and reported)
8) calibration standards (all calibration standards analyzed to generate response factor databases are reported, including chromatograms and raw data reports generated by the data system of the mass spectrometer)
9) daily calibration standards (all raw data associated with the analysis of daily calibration standards are reported)
10) total ion current chromatograms and quantitation reports generated by the mass spectrometer data system for all samples and blanks are reported
11) mass spectra for the target compounds observed in each sample and blank analysis are supplied
12) if identifications of additional compounds are requested by the client, the data for compounds identified and semi-quantitative calculated quantities are reported, with mass spectra of the compounds and the output of the computerized library search
13) forms generated by the Radian Sample and Analysis Management System are supplied
14) chain of custody forms and sample seals.

Mass spectrometric data are reduced in a semi-automated mode; that is, the analyst verifies the identification of every target compound prior to the generation of quantitative data for that compound. When quantitative data are generated, the report from the computer is entered into the project notebook and archived permanently in this notebook. Then, either the analyst or the Data Control Officer generates the additional information which is to be incorporated into the deliverable data package. The quantitative calculations and qualitative identifications are verified by the Laboratory Supervisor, with a subsequent review by the Senior Technical Advisor.

Immediately after completion of analysis of a sample lot, data are summarized in tabular form. Any deviations from the protocol are documented. The stability of the response factors is reported in tabular form, to show that instrumental parameters were in control when the assay was performed. A table of surrogate recoveries is also provided to document the reproducibility of the overall sample preparation and analysis procedures.

A field blank (or trip blank, as provided by the client) will be analyzed for the purpose of providing blank correction. The results of the analysis of all blanks are reported, but no blank correction is reported, except in the case where the amount of a compound reported on the blank is greater than 25% of the sample amount. In that case, no values will be reported for the compound in the samples, since the values are suspect. In addition, laboratory blanks will also be analyzed. The number of blanks analyzed is one blank per ten sample analyses. If sufficient trip blanks are not available, laboratory blanks will be substituted.

13.0 INTERNAL QUALITY CONTROL CHECKS

Quality control procedures have been described in detail in the description of the analytical procedures, since the quality control measures are an integral part of the analytical methods. However, the quality control procedures are summarized below:

1) chromatographic check of 50% of all sampling cartridges prepared to monitor for contamination prior to sending the sampling tubes to the field
2) analysis of at least one laboratory or field (trip) blank for each ten field samples analyzed
3) daily tuning checks for stability of the mass spectrometer
4) daily calibration checks for stability of the mass spectrometric calibration
5) continuous monitoring of surrogate recoveries for every sample and blank analyzed
6) continuous monitoring of signal level for the quantitation standard in every sample and blank analyzed.
7) initial quality check of the data by the analyst, with subsequent review by the Laboratory Supervisor, and another level of review by the Senior Technical Advisor. Final review of the data is provided by the Project Director.

The following criteria are applied for rejection of data:

1) if an analyte is observed in a blank at a level which is 25% or more of the level at which that compound is observed in a sample, no quantitative data are reported for that compound in the sample.
2) if none of the quantitation standards are observed in the analysis of a sample, the data for that sample are rejected.
3) if surrogate recoveries are below 30%, the data for that

sample are rejected.
4) if the level of compound observed in a sample is below the limit of detection calculated for the compound, no value is reported for that compound.

The limits of detection to be expected in the program are compound specific and will vary from batch to batch of samples. The chief factor influencing limits of detection is the amount of water which enters the analytical system, a parameter which is completely out of the control of the analyst. Water entering the analytical system causes several types of problems: 1) a quantity of water which is sufficiently large will cause a shutdown of the entire analytical system because of the failure of the system vacuum and electronics. 2) lower amounts of water will cause distortion of the chromatography, with consequent spreading of the chromatographic peaks and lowering of observed peak areas for analytes. A sharp chromatographic peak with well-defined baseline provides far more reproducible and larger values for quantitation than a peak which is highly asymmetric and wide. 3) an amount of water which is not sufficient to cause visible distortion of the chromatographic peaks can still cause areas of peaks to be lowered from the values observed for the same quantity of compound spiked onto a dry sampling cartridge. This effect is demonstrated daily on the analytical system when field samples (which contain varying amounts of water) are analyzed after the introduction of standards from an almost completely dry static dilution bulb.

An additional factor which influences the limits of detection is the amount of interference encountered from the sample matrix. For example, a sample which is taken near the side of a busy road (and which, therefore, shows high levels of hydrocarbons) will exhibit a proportionately higher detection limit for compounds of interest which elute in the midst of the hydrocarbons. Again, the background matrix is completely out of the control of the analyst.

Under conditions in which no water is introduced into the analytical system and the matrix background level is minimal, the limits of detection shown in the first column of Table 5 will be attainable. In the presence of water in a reasonable range on the samples and not allowing for any interferences from the background, the detection limits shown in the second column of the table below should be attainable.

Table 5. Limits of Detection of Compounds of Interest

Compound	Detection Limit, ng Without Water	With Background	ng/mL EPA 624
trichlorofluoro-methane	0.3	1-5	--
1,1-dichloroethene	0.25	1-5	2.8
methylene chloride	0.8	1-5	2.8
t-1,2-dichloroethene	0.2	1-5	1.6
1,1-dichloroethane	0.15	2-7	4.7
chloroform	0.7	1-5	1.6
1,1,1-trichloroethane	0.8	1-5	3.8
carbon tetrachloride	0.9	2-7	2.8
benzene	0.3	0.5-5	4.4
1,2-dichloroethane	0.9	2-7	2.8
trichloroethene	1.0	3-8	1.9
1,2-dichloropropane	1.0	3-8	6.0
bromodichloromethane	0.7	1-5	2.2
t-1,2-dichloropropene	1.4	4-10	---
toluene	0.5	1-5	6.0
c-1,3-dichloropropene	1.8	4-10	5.0
1,1,2-trichloroethane	1.0	3-8	5.0
tetrachloroethene	1.0	3-8	4.1
dibromochloromethane	0.6	2-7	3.1
chlorobenzene	0.4	1-5	6.0
ethylbenzene	0.8	1-5	7.2
m-, p-xylene	0.3	1-5	---
o-xylene	0.7	1-5	---
bromoform	0.8	1-5	4.7
1,1,2,2-tetrachloro-ethane	0.5	1-5	6.9
m-dichlorobenzene	0.6	1-5	---
p-dichlorobenzene	0.6	1-5	---
o-dichlorobenzene	0.6	1-5	---
styrene	0.4	1-5	---
acetone	0.4	1-5	---
2-butanone	2.0	5-15	---
heptane	1.8	5-15	---
isopropylbenzene	0.3	1-5	---
hexane	0.2	2-6	---
chloromethane	0.2	3-15	---
vinyl chloride	0.4	3-15	---
bromomethane	1.2	3-15	---
chloroethane	2.1	3-15	---

Detection limits from EPA Method 624 (calculated as ng/mL, since the sample is purged from water) are included for comparison with the detection limits which can be expected from Tenax® cartridges. There should likewise be water present when the compounds of interest are purged from water, and these numbers show that, in most cases, detection limits anticipated from thermal desorption and analysis of compounds

of interest from Tenax® which contains some amount of water are comparable to the detection limits which might be anticipated from use of EPA Method 624. Note that theoretical detection limits for some compounds are far lower than the range of values which might be anticipated when these analytes are found on a cartridge with water and possibly other background material. Only water is taken into consideration in the Table above; other background is completely unpredictable. The permanent gases, in particular, are very strongly influenced by the presence of water. Chloroethane, for example, responds poorly in the best of circumstances and, being one of the earliest-eluting compounds, shows a very severe effect in the presence of water. The values tabulated above should be reasonably attainable in the presence of the amounts of water which might be anticipated from sampling on a humid day. If sampling occurs on a rainy day, however, the values may well go substantially above the top of the range listed in the Table, if the samples can be analyzed at all.

14.0 PERFORMANCE/SYSTEM AUDITS

Prior to the initiation of analysis of air toxics samples, the Radian Laboratory participated in the analysis of a series of Performance Evaluation samples. Radian is quite willing to participate in such an evaluation again, if audit samples are available. In the present program, the analysis of the daily calibration sample will serve as a system control.

Participation in the Contract Laboratories Program (CLP) of the Environmental Protection Agency is projected for the Radian Perimeter Park laboratory for late summer/early fall of 1987. At that time, Radian will follow established CLP protocols with regard to the regular analysis of audit samples. At present, the Radian laboratories all over the country are regular participants in the Office of Solid Waste (OSW) Performance Evaluation Sample analysis program, in which samples are sent quarterly to participating laboratories. The sample analyses in this program encompass both organics analysis and metals analysis. The Radian Perimeter Park laboratory has also participated in the analysis of organics and metals through the Environmental Protection Agency's wastewater analysis program as a prerequisite to the analysis of wastewater samples. Radian Perimeter Park laboratory has also participated in the analysis of performance evaluation samples for certification as an analytical laboratory for the state of North Carolina, and for those states with which North Carolina has reciprocal agreements to accept certification.

15.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

Data precision is assessed most reliably by the analysis of replicate samples, which must be supplied by the field personnel. In general, duplicate or triplicate samples are not supplied with a given batch of samples. Results from an analysis of replicate spiked samples, supplied as Performance Evaluation samples, are discussed elsewhere in this Quality Assurance Plan. At any time, by request of the client, if problems with reproducibility are suspected, a set of replicate sampling tubes can be prepared and analyzed, using any or all of the compounds of interest. The only other indirect assessment of precision is the recovery of surrogates for a given batch of samples, where conditions such as background and level of water observed on the sampling tubes might reasonably be expected to be constant. However, this assumption of constant conditions for a given site is only an approximation, since conditions may vary in the course of a day and will almost certainly vary over a period of several days.

Data accuracy is evaluated by analysis of spiked samples to determine the correspondence between found and true values. This type of experiment has been performed only once, with results discussed above.

Completeness is evaluated by comparing the number of analyses for which valid data are generated to the number of sampling tubes supplied for analysis. Completeness is typically 95% or better. The usual reasons for obtaining completeness less than 100% are samples broken in shipment which the laboratory is subsequently unable to analyze or external factors such as power failure or instrument failure.

16.0 CORRECTIVE ACTIONS

Corrective actions are initiated whenever any of the parameters monitored in the program shows a value which is outside the limits for acceptability. The corrective action requires the determination of the source of the problem: some of the possible sources of problems include instrument failure and contamination. The appropriate corrective actions include repair of the instrumentation, where pertinent, and removal of the sources of contamination. It is the responsibility of the analyst to identify problems while the Laboratory Supervisor has the responsibility for initiating and completing corrective actions and advising the Project Director of the nature of the problem and the action taken to resolve it. The Project Director approves all corrective actions and, if necessary, advises the client.

17.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The Laboratory Supervisor responsible for evaluating measurement accuracy, precision, and completeness on a routine basis. The Project Director is responsible for reporting the evaluation of these results to the client and to the Laboratory Technical Director is Quality Assurance Plan criteria are not being met. Reports on corrective actions and their resolution are prepared by the responsible individual and submitted to the Laboratory Supervisor. Appropriate Quality Assurance and Quality Control data are an integral part of every analytical report which is delivered by the laboratory.

18.0 SAFETY

The following safety considerations listed below will be adhered during any planned laboratory activities. Laboratory activities will be performed under the guidance of the Radian Corporate Health and Safety manual and the current laboratory procedures at Radian's Perimeter Park analytical laboratory facility.

The Radian Project Director is responsible for the health and safety of Radian project personnel . All work practices shall comply with the safety requirements prescribed by the Occupational Safety and Health Act of 1970, as amended (29 CFR Part 1910), and all employees will be expected to perform their duties in a responsible, professional, and safe manner. All Radian personnel will be required to read the Radian Corporate Health and Safety plan, and have all their questions answered prior to the start of the laboratory activities.

D

APPENDIX D

STANDARD OPERATING PROCEDURES FOR ANALYSIS OF WATER, LEACHATE AND SEDIMENT USING NON-CLP PROTOCOLS

A) WARZYN PROCEDURES

ANALYTICAL METHODS USED BY WARZYN
FOR THE PAGEL'S PIT RI/FS

The following are standard operating procedures for analyses to be performed by Warzyn on samples from Pagel's Pit Landfill. Methods list detection limits and numbers of QC samples to be performed, but do not specify performance standards. The following table lists performance standards for quality control samples. If QC samples do not meet performance standards, the samples are to be reanalyzed. If standards still are not met, the laboratory QA office is to be notified.

RCW/sss/KDF
[sss-800-92]
12660.08

Attachment 1

QUALITY CONTROL REQUIREMENTS FOR ANALYSES PERFORMED
BY WARZYN AT PAGEL'S PIT LANDFILL

PARAMETER	AUDIT	FREQUENCY*	LIMITS
Arsenic	Lab blank	1 per set	<0.005 mg/L
	Duplicate	1 per 10	10% or <0.005 mg/L
	Calibration check STD	1 per 10 and end of run	90-110% recovery
	Matrix spike	1 per 10	85-115% recovery
	EPA QC Reference STD	1 per set	95% confidence interval
Barium	Lab blank	1 per set	<0.010 mg/L
	Duplicate	1 per 10	10% or <0.005 mg/L
	Calibration check STD	1 per 10 and end of run	90-110% recovery
	Matrix spike	1 per 10	85-115% recovery
	EPA QC Reference STD	1 per set	85-115% recovery
Cadmium	Lab blank	1 per set	<0.0005 mg/L
	Duplicate	1 per 10	10% or <0.005 mg/L
	Calibration check STD	1 per 10 and end of run	90-110% recovery
	Matrix spike	1 per 10	85-115% recovery
	EPA QC Reference STD	1 per 10	95% confidence interval

QUALITY CONTROL REQUIREMENTS FOR ANALYSES PERFORMED BY WARZYN AT PAGEL'S PIT LANDFILL

Chloride	Indicator blank	1 per set	<0.25 ml titrant
	Titrant standardization	2 per titrant solution	<5% RPD
	Check standard	1 per 10 and end of run	90-110% recovery
	Duplicate	1 per 10	<10% or 1 mg/L
	Matrix spike	1 per 10	85-115% recovery
	EPA QC Reference Standard	1 per set	95% confidence interval
Total Alkalinity	Titrant standardization	Daily - 2 per titrant solution	<5% RPD
	Lab Blank	1 per set	<5 mg/L
	Check standard	1 per 10 and end of run	90-110% recovery
	Duplicate	1 per 10	10% or <10 mg/L
	Matrix spike	1 per 10	85-115% recovery
	EPA QC Reference STD	1 per set	95% confidence interval
Total Phenolics	Lab blank	1 per set	<0.005 mg/L
	Duplicate	1 per 10	10% or <0.005 mg/L
	Calibration check STD	1 per 10 and end of run	90-110% recovery
	Matrix spike	1 per 10	85-115% recovery
	EPA QC Reference STD	1 per set	95% confidence interval

QUALITY CONTROL REQUIREMENTS FOR ANALYSES PERFORMED BY WARZYN AT PAGEL'S PIT LANDFILL

Total Cyanide	Lab blank	1 per set	< 0.005 mg/L
	Duplicate	1 per 10	10% or <0.005 mg/L
	Calibration check STD	1 per 10 and end of set	85-115% recovery
	Matrix spike	1 per 10	85-115% recovery
	EPA QC Reference STD	1 per set	95% confidence interval

*Frequencies apply to each matrix individually.

RCW/mml/KDF
[mml-108-88a]
12660.08

ACID DIGESTION FOR WASTEWATER, GROUNDWATER AND EP TOXICITY SAMPLES - "CLP Intent"

Scope and Application: This acid digestion is applicable to all aqueous sample matrices. A nitric/hydrochloric acid digestion is used for all metals which are to be analyzed by the AA-flame technique and for the furnace analysis of antimony. Metals which are to be analyzed by the AA-furnace technique will follow the nitric acid/ hydrogen peroxide digestion.

Method: Nitric and nitric/hydrochloric acid digestions.

Reference: EPA SW-846, "Test Methods for Evaluating Solid Wastes", July, 1982. Methods 3010 and 3020.

Contract Laboratory Program, "Statement of Work", July, 1985.

Sample Handling: Aqueous samples must be acidified with concentrated nitric acid to pH < 2. Set up digestion as soon as possible; digested sample must be analyzed within 6 months.

Reagents and Apparatus:

1. Hot Plate
2. 250 mL beakers
3. 100 mL graduated cylinders
4. Class A volumetric glassware
5. Milli-Q water
6. Instra-analyzed nitric acid
7. Instra-analyzed HCL acid
8. Stock and standard metal solutions
9. Whatman #42 filter paper
10. Glass funnels
11. Watch glasses
12. 30% hydrogen peroxide

Reagent Preparation:

1. Intermediate and Working Metal Solutions: Refer to the specific metal SOP for instructions on preparation.

Notes:

1. All blanks, duplicates, and spikes, as well as a digested check standard must be carried through this digestion procedure.

Procedure:

A. Digestion For AA-Flame and Antimony by Furnace:

1. All glassware must be acid-washed with 1:1 nitric acid and thoroughly rinsed with Milli-Q water prior to use.

2. Measure out 100 mL aliquots of samples, blanks, and standards into 250 mL beakers using a graduated cylinder.

3. Add 2 mL of 1:1 HNO_3 and 10 mL of 1:1 HCL.

4. Cover with a watch glass and heat on the hot plate until the volume has been reduced to between 25 and 50 mL. Make certain that the sample does not boil.

5. Quantitatively transfer digested samples, blanks, and standards into 100 mL volumetric flasks. Filter samples through Whatman #42 filters, rinse beakers and filters with Milli-Q water and dilute to 100 mL. Alternatively, samples can be transfered to 100 mL volumetric flasks, without filtering. Dilute to volume and let any insoluble material settle overnight.

6. Samples are now ready for analysis using the AA-flame procedure.

B. Digestion For AA-Furnace (except Antimony):

1. All glassware must be acid-washed with 1:1 nitric acid and thoroughly rinsed with Milli-Q water prior to use.

2. Measure 100 mL aliquots of samples, blanks, and standards into 250 mL beakers using a graduated cylinder.

3. Add 1.0 mL of 1:1 HNO_3 and 2 mL of 30% H_2O_2.

4. Cover with a watch glass and heat on the hot plate at 95°C for 2 hours or until the volume is reduced to between 25 and 50 mL. Make certain that the sample does not boil.

5. Quantitatively transfer digested samples, blanks, and standards into 100 mL volumetric flasks. Filter samples through Whatman #42 filters, rinse beakers and filters with Milli-Q water and dilute to 100 mL. Alternatively, samples can be transfered to 100 mL volumetric flask without filtering. Dilute to volume and let any insoluble material settle overnight.

6. Samples are now ready for analysis using the AA furnace procedures.

Quality Control

1. Refer to each specific metal SOP for quality control requirements.

2. If a digested spike is diluted out of the working concentration range (too low to detect) run a manual spike. The data is acceptable if the manual spike is within acceptable ranges. If the manual spike is outside the QC ranges, the sample and spike must be re-digested at a dilution.

Revision Date

Michael Linskens 9/25/87

Michael J. Linskens
Laboratory Manager

9/25/87

Kim Finner

Kim D. Finner
Analytical Laboratory QA/QC Officer

Lawrence D. Andersen

Lawrence D. Andersen
Vice President, Technical Services

ARSENIC - "CLP Intent"

Method: AA - Furnace; Direct Injection

Reference: EPA 1983, Method 206.2

"Analytical Methods for Furnace Atomic Absorption Spectrophotometry", 1982, Perkin-Elmer Corporation

"Techniques in Graphite Furnace Atomic Absorption Spectrophotometry", 1985, Perkin-Elmer Corporation

Contract Laboratory Program, "Statement of Work", July, 1985

Detection Limit: 0.002 mg/L

Optimum Concentration Range: 0.002 - 0.050 mg/L

Sample Handling: Acidify with nitric acid to pH < 2. Analyzed within 6 months. All samples must be digested prior to analysis.

Instrument Conditions:

1. Arsenic electrodeless discharge lamp with lamp energy set at 8.

2. Wavelength: 193.7 nm

3. Slit Width: 0.7 Alternate

4. Mode: Peak height

5. HGA Furnace Programming:

Step 1:	130	(dry temp)	10	(ramp time)	20	(hold time)
Step 2:	1300	(char temp)	10	(ramp time)	10	(hold time)
Step 3:	2500	(atom temp)	0	(ramp time)	3	(hold time)

* Also press the read, record and stop flow buttons; enter t = 3 sec.

Step 4: 2500 (max temp) 0 (ramp time) 3 (hold time)

Press record button.

6. Sample Volume: 20 uL

7. Standards to use for curve set-up: 10.0, 20.0, 50.0 ug/L.

8. Pyrolytic tubes must be used.

Reagent Preparation:

1. Standard Arsenic Solution (1000 ug/L Arsenic): Pipet 1.00 mL of the 1000 ppm stock arsenic solution into a 1000 mL volumetric flask, add 1/2 mL HNO_3, and dilute to the mark with Milli-Q water. Prepare fresh every month.

2. Standards (Prepare fresh every week.):

Concentration of Standard	Volume of Arsenic Standard	Dilute to
0 ug/L	0 mL of 1000 ug/L As	100 mL
10 ug/L	1 mL of 1000 ug/L As	100 mL
20 ug/L	2 mL of 1000 ug/L As	100 mL
50 ug/L	5 mL of 1000 ug/L As	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. Nickel nitrate is added as a matrix modifier to minimize volatilization losses during the drying and charring steps.

4. The use of the background correction is required.

5. High concentrations of phosphorus interfere with this procedure. The gaseous hydride method for arsenic should be used in these cases.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

Prior to Analysis: To 5 mL of digested sample or standard, add 0.1 mL of 5% nickel nitrate solution. Mix well.

If Arsenic is to be analyzed in concentration mode, use the 20 and 50 ug/L arsenic standards and the procedures for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard of 20 ug/L is to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within acceptable ranges or the samples run after the last acceptable calibration standard are to be reanalyzed. Record the calibration standard in the quality control book. The confidence limits are noted in the quality control book.

3. A digested duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a digested duplicate and spike are still required. Duplicates are to be averaged. Spike samples with a standard of twice the concentration of the sample in a 1:1 ratio of sample to standards. Spike recoveries and duplicate results are to be within acceptable ranges or the use of dilution, or method of standard additions is to be applied to reduce the interferences.

4. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions are:

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outside 85 - 115%, standard additions are required.

5. An EPA reference sample will be analyzed with each analysis.

Calculations:

1. Plot concentrations vs. absorbance on graph. Determine unknowns using graph, or

2. Calculate using linear regression, or

3. Calculate using the concentration mode.

Revision Date

Michael Linskens 9/25/87
Michael J. Linskens
Laboratory Manager

9/25/87

Kim Finner
Kim D. Finner
Analytical Laboratory QA Officer

Lawrence D. Andersen
Lawrence D. Andersen
Vice President, Technical Services

BARIUM - "CLP Intent"

Method: AA - Flame; Direct Aspiration

Reference: EPA 1983, Method 208.1

"Analytical Methods for Atomic Absorption Spectrophotometry", 1982, Perkin-Elmer Corporation

Contract Laboratory Program, "Statement of Work", July, 1985

Detection Limit: 0.2 mg/L

Optimum Concentration Range: 1.00 - 10.0 mg/L

Sample Handling: Acidify with nitric acid to pH < 2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

1. Barium hollow cathode lamp with lamp energy set at 25.
2. Wavelength: 553.6 nm
3. Slit Width: 0.2
4. Fuel: Acetylene
5. Oxidant: Nitrous oxide
6. Type of flame: Red
7. Standards to use for curve set-up: 1.00, 2.00, 5.00, 10.0

Reagent Preparation:

1. Standard Barium Solution (100 mg/L Barium): Pipet 10 mL of the 1000 ppm stock barium solution into a 100 mL volumetric flask, add 1/2 mL HNO_3 and dilute to the mark with Milli-Q water. Prepare fresh every 2 months.

2. Standards (Prepare fresh every 1 month.):

Concentration of Standard	Volume of Barium Standard	Dilute to
1.00 mg/L	1 mL of 100 mg/L Ba	100 mL
2.00 mg/L	2 mL of 100 mg/L Ba	100 mL
5.00 mg/L	5 mL of 100 mg/L Ba	100 mL
10.0 mg/L	10 mL of 100 mg/L Ba	100 mL

3. Potassium Chloride Solution: Dissolve 95g KCL in Milli-Q water and dilte to 1 liter using Class A volumetric glassware.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. Barium is easily ionized in the nitrous oxide-acetylene flame. This can be controlled by the addition of potassium. Add 2 mL of 1000 ppm potassium to 100 mL sample and standards.

4. High calcium can cause an enhancement of the barium signal.

5. This flame procedure can be used for digested samples, EP Toxicity samples or any other samples where high concentrations of barium (concentration greater than 1.00 mg/L are expected and low detection limits are not required). EP Toxicity samples must be analyzed by standard additions.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Flame - Direct Aspiration section of this manual.

Potassium chloride solution is added as a matrix modifier to the samples and standards at a ratio of 0.2 mL KCL to 10 mL of sample or standard.

If barium is to be run in concentration mode, use the 5.00 and 10.0 standards and follow the procedure for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistant from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, flame head alignment, lamp alignment, etc.).

2. A quality control calibration standard of 5.00 mg/L is to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standards in the quality control book. The confidence limits are noted in the quality control book.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Duplicates are to be averaged. Spike samples with a standard of twice the concentration of the sample in a 1:1 ratio of sample to standard. Spike recoveries and duplicates are to be within acceptable ranges or the use of dilution or method of standard additions is to be applied to reduce the interferences.

Calculations:

1. Plot concentration vs absorbance on graph. Determine unknowns using graph, or

2. Calculate using linear regression, or

3. Calculate using concentration mode.

Revison Date

Michael Linskens 9/25/87
Michael J. Linskens
Laboratory Manager

9/25/87

Kim Finner
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Lawrence D. Andersen
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BARIUM - "CLP Intent"

Method: AA - Furnace; Direct Injection

Reference: EPA 1983, Method 208.2

"Analytical Methods for Furnace Atomic Absorption Spectrophotometry", 1982, Perkin-Elmer Corporation

"Techniques in Graphite Furnace Atomic Absorption Spectrophotometry", 1985, Perkin-Elmer Corporation

Contract Laboratory Program, "Statement of Work", July, 1985

Detection Limit: 0.005 mg/L

Optimum Concentration Range: 0.005 - 0.050 mg/L

Sample Handling: Acidify with nitric acid to pH < 2. All samples must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

1. Barium hollow cathode lamp with lamp energy set at 25.
2. Wavelength: 553.6 nm
3. Slit Width: 0.2 Alternate
4. Mode: Peak height
5. HGA Furnace Programming:

Step 1:	110	(dry temp)	15	(ramp time)	15	(hold time)
Step 2:	1000	(char temp)	15	(ramp time)	15	(hold time)
Step 3:	2400	(atom temp)	0	(ramp time)	5	(hold time)

* Also press the read, record and stop flow buttons; enter t = 5 sec.

Step 4: 2700 (max temp) 1 (ramp time) 5 (hold time)

Press Record button

6. Sample Volume: 20 uL
7. Standards to use for curve set-up: 10.0; 20.0; 50.0; 100 ug/L.
8. Pyrolytic tubes must be used.

Reagent Preparation:

1. Standard Barium Solution (1000 ug/L Barium): Pipet 1.00 mL of the 1000 ppm stock barium solution into a 1000 mL volumetric flask, add 1/2 mL HNO_3, and dilute to the mark with Milli-Q water. Prepare fresh every month.

2. Standards (Prepare fresh every week.):

Concentration of Standard	Volume of Barium Standard	Dilute to
0 ug/L	0 mL of 1000 ug/L Ba	100 mL
10.0 ug/L	1 mL of 1000 ug/L Ba	100 mL
20.0 ug/L	2 mL of 1000 ug/L Ba	100 mL
50.0 ug/L	5 mL of 1000 ug/L Ba	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. The use of halide acids should be avoided.

4. The alignment of the furnace is very critical, stray light can be a major interference.

5. The barium flame procedure is recommened where concentrations of greater than 1.0 mg/L are expected.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

If barium is to be run in concentration mode, use the 20.0 and 50.0 standards and follow the procedures for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard of 20.0 ug/L is to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within the acceptable ranges or the samples run after the last acceptable calibration standard are to be reanalyzed. Record the calibration standard in the quality control book. The confidence limits are noted in the quality control book.

3. A digested duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a digested duplicate and spike are still required. Duplicates are to be averaged. Spike samples with a standard of twice the concentration of the sample in a 1:1 ratio of sample to standards. Spike recoveries and duplicates are to be within acceptable ranges or the use of dilution, or method of standard additions is to be applied to reduce the interferences.

4. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions are:

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outsie 85 - 155%, standard additions are required.

5. An EPA reference sample will be analyzed with each analysis.

Calculations:

1. Plot concentrations vs. absorbance on graph. Determine unknowns using graph, or

2. Calculate using linear regression.

3. Calculate using the concentration mode.

Revision Date

9/25/87

Michael Linskens 9/25/87
Michael J. Linskens
Laboratory Manager

Kim Finner
Kim D. Finner
Analytical Laboratory QA Officer

Lawrence D. Anderson
Lawrence D. Anderson
Vice President, Technical Services

CADMIUM - "CLP Intent"

Method: AA - Furnace; Direct Injection

Reference: EPA 1983, Method 213.2

"Analytical Methods for Furnace Atomic Absorption Spectrophotometry", 1982, Perkin-Elmer Corporation

"Techniques in Graphite Furnace Atomic Absorption Spectrophotometry", 1985, Perkin-Elmer Corporation

Contract Laboratory Program, "Statement of Work", July, 1985

Detection Limit: 0.0002 mg/L

Optimum Concentration Range: 0.0002 - 0.0020 mg/L

Sample Handling: Acidify with nitric acid to pH < 2. All samples must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

1. Cadmium hollow cathode lamp with lamp energy set at 4.
2. Wavelength: 228.8 nm
3. Slit Width: 0.7 Alternate
4. Mode: Peak area
5. HGA Furnace Programming:

Step 1:	120	(dry temp)	10	(ramp time)	20	(hold time)
Step 2:	250	(char temp)	10	(ramp time)	20	(hold time)
Step 3:	2100	(atom temp)	2	(ramp time)	3	(hold time)

* Also press the read, record and stop flow buttons; enter t = 5 sec

Step 4:	2700	(max temp)	1	(ramp time)	5	(hold time)

Press record button.

6. Sample Volume: 20 uL
7. Standards to use for curve set-up: 0.50, 1.00, 2.00, ug/L
8. Pyrolytic tubes must be used.

Reagent Preparation:

1. Standard Cadmium Solution (1000 ug/L Cadmium): Pipet 1.00 mL of the 1000 ppm stock cadmium solution into a 1000 mL volumetric flask, add 1/2 mL HNO_3, and dilute to the mark with Milli-Q water. Prepare fresh every month.

2. Working Cadmium Solution (100 ug/l Cadmium): Pipet 10 mL of the 1000 ug/l cadmium into a 100 mL volumetric flask and dilute to the mark with Milli-Q water. Prepare fresh every month.

3. Standards (Prepare fresh every week.):

Concentration of Standard	Volume of Cadmium Standard	Dilute to
0.00 ug/L	0 mL of 100 ug/L Cd	100 mL
0.50 ug/L	0.5 mL of 100 ug/L Cd	100 mL
1.00 ug/L	1 mL of 100 ug/L Cd	100 mL
2.00 ug/L	2 mL of 100 ug/L Cd	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. The use of background correction is required.

4. The cadmium flame procedure is recommended where concentrations of greater than 0.10 mg/L are expected.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

Use of peak area is required.

If cadmium is to be analyzed in concentration mode, use the 1.00 and 2.00 ug/L cadmium standards and follow the procedures for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard of 1.00 ug/L is to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within the acceptable ranges or the samples run after the last acceptable calibration standard are to be reanalyzed. Record the calibration standard in the quality control book. The confidence limits are noted in the quality control book.

3. A digested duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a digested duplicate and spike are still required. Duplicates are to be averaged. Spike samples with a standard of twice the concentration of the sample in a 1:1 ratio of sample to standards. Spike recoveries and duplicates are to be within acceptable ranges or the use of dilution, or method of standard additions is to be applied to reduce the interferences.

4. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions is:

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outside 85 - 115%, standard additions are required.

5. An EPA reference sample will be analyzed with each analysis.

Calculations:

1. Plot concentrations vs. peak area on graph. Determine unknowns using graph, or

2. Calculate using linear regression, or

3. Calculate using concentration mode.

Revision Dates

9/25/97

Michael Linskens 9/25/97
Michael J. Linskens
Laboratory Manager

Kim Finner
Kim D. Finner
Analytical Laboratory QA Officer

Lawrence D. Andersen
Lawrence D. Andersen
Vice President, Technical Services

TOTAL PHENOLICS - Distillation

Scope and Application: This method is applicable to the analysis of drinking water, surface water, domestic and industrial wastewater and solids.

Method: Automated 4AAP with distillation.

Reference: EPA 1983, Method 420.2.

Detection Limit: 0.005 mg/L

Optimum Range: 0.005 - 0.5 mg/L

Sample Handling: Aqueous samples are to be collected in a 500 mL glass container with a teflon-lined cap. Preserve to pH<2 with sulfuric acid and refrigerate at 4°C. Non-aqueous samples are to be collected in a glass container and refrigerated at 4°C. Sample should be analyzed within 28 days of collection.

Reagents and Apparatus:

1. Buffer solution
2. Phenol
3. Copper sulfate
4. Glass distillation apparatus
5. 500 mL distillation flasks
6. 500 mL erlenmeyer flasks
7. Glass funnel
8. 250 mL graduated cylinder
9. Lachat Instrument
10. Milli-Q water
11. Sodium hydroxide
12. Concentrated sulfuric acid
13. Ammonium chloride
14. Ammonium hydroxide
15. Phenol manifold.

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. Buffer solution: Dissolve 67.6 g NH_4Cl in 572 mL conc. NH_4OH and dilute to 1000 mL in a volumetric flask with Milli-Q water.

2. Stock phenol solution (1000 mg/L): Dissolve 1.000 g phenol in freshly boiled and cooled Milli-Q water in a volumetric flask and dilute to 1 liter.

3. Working phenol solution A (10 mg/L): Dilute 10 mL of stock phenol solution to 1 liter with Milli-Q water in a volumetric flask. (Prepare fresh daily!)

4. Copper sulfate solution: Dissolve 100 g $CuSO_4 \cdot 5H_2O$ in Milli-Q water and dilute to 1 liter in a volumetric flask.

5. NaOH (1N): Dissolve 40 g NaOH in Milli-Q water in a 1 liter volumetric flask, dilute to the mark.

6. NaOH (6N): Dissolve 240 g NaOH in Milli-Q water in a 1 liter volumetric flask, dilute to the mark.

7. H_2SO_4 (1N): Dilute 28 mL of concentrated H_2SO_4 with 500 mL Milli-Q water in a 1 liter volumetric flask, and dilute to the mark.

Notes:

1. Interferences:

   a) Oxidizing agents such as chlorine interfere. Add excess ferrous ammonium sulfate immediately after sampling to remove interference. If chlorine is not removed, the phenols may be partially oxidized and results will be low.

   b) Sulfur compounds interfere. Interferences are eliminated by acidifying the sample to a pH of less <4 with H_3PO_4 or H_2SO_4 and aerating briefly by stirring and adding $CuSO_4$.

2. If the solutions do not remain a blue to green color after the addition of the $CuSO_4$, or lose the color upon heating, additional $CuSO_4$ is required. Record the additional amount of $CuSO_4$ required on the data sheet.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed and Milli-Q water rinsed prior to analysis. Glassware which is difficult as stated can be soaked in a chromate solution for a few hours and then washed as usual.

2. For aqueous samples: Using a 250 mL graduated cylinder, place 250 mL of preserved sample into a distillation flask.

   For solid samples: Weigh out 0.5-1.0 g and place in a distillation flask. Add 250 mL Milli-Q water.

To Spike samples: Pipet 2.5 mL of 10 mg/L working phenol solution A into 250 mL of sample. This corresponds to a 0.1 mg/L spike.

3. Adjust the sample to a pH 4 $\pm$ 0.1, using 1N H_2SO_4 or 6N NaOH. Add 5 mL $CuSO_4$ solution. Add several of boiling chips to the flask.

4. Set flask on mantle, connect to condenser, and place a labeled 250 mL volumetric flask under tip of condenser. Turn the mantle to medium-high heat.

5. Distill about 200 mL of sample. Stop the distillation, and when boiling ceases, add about 50 mL of warm Milli-Q water (warm water is required to prevent the flask from cracking.) to the flask. Resume distillation until 250 mL have been collected.

6. If the distillate is turbid, filter through a prewashed 0.45 micron membrane filter.

7. Distillates are ready for analysis. Proceed with Lachat SOP# PhenAA for automated color development.

Quality Control:

1. The standards do not need to be carried through the distillation procedure.

2. A reagent blank is to be analyzed with each set of samples. This blank is to be carried through the distillation step and is used as a check for contamination.

3. A quality control calibration standard of 0.40 mg/L (which has been carried through the distillation step) is to be analyzed with each run. The last sample analyzed in the run is to be the calibration standard. These standards must be within the acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standards in the quality control book. The confidence limits are noted in the quality control book.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Duplicates are to be averaged. Spike recoveries and duplicate results are to be within acceptable ranges.

Calculation:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer.

2. To calculate the spike:

$$\% \text{ recovery} = \frac{[\text{Total mL}][\text{Conc. of}]}{[\text{SA + STD}][\text{ spike }]} - \frac{[\text{ml SA }][\text{Conc. of}]}{[\text{ used}][\text{ SA }]} \times 100$$

$$(\text{mL STD used})(\text{Conc. STD})$$

Where SA = sample
STD = standard

TOTAL PHENOLICS - AUTOANALYZER

Scope and Application: This method is applicable to distilled groundwater, drinking water, wastewater, sediments and soils. All samples must be distilled prior to analysis with the autoanalyzer.

Reference: EPA, 1983, Method 420.2

Lachat Instruments, 1986, Method 10-210-00-1-A
Standard Methods, 16th Edition, pages 556-561

Detection Limit: 0.005 mg/L

Optimum Concentration Range: 0.005 - 0.500 mg/L

Sample Handling: Samples should be capped and refrigerated at 4°C after distillation.

Instrument Conditions:

1. Pump speed: 35
2. Cycle period: 40 seconds
3. Load period: 20 seconds
4. Inject period: 20 seconds
5. Inject to start of peak period: 14 seconds
6. Inject to end of peak period: 40 seconds
7. Gain: 200x10 (use 10x gain)
8. Zero: 350
9. Interference filter: 500 nm
10. Sample loop: 120 cm
11. Standards for standard curve: 0, 0.050, 0.100, 0.200, 0.500 mg/L

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. Degassed Milli-Q water - 2 options:

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.

   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water. (15 min/20 L.) Store in cubitainer.

2. 4-aminoantipyrine color reagent: In a 250 mL volumetric flask, dissolve 0.16 g of 4-aminoantipyrine ($CH_3NN(C_6H_5)COC(HN_2)\cdot CCH_3$), in approximately 150 mL of Milli-Q water and dilute to 250 mL. Prepare fresh daily.

3. Buffered potassium ferricyande (pH 10.3): In a 1 liter volumetric flask, dissolve 2.0 g potassium ferricyanide ($K_3Fe(CN)_6$), 3.1 g boric acid (H_3BO_3) and 3.75 g potassium chloride (KCl) in 800 mL of water. Add 46.8 mL of 1 M sodium hydroxide and dilute to the mark. Filter through a 0.45 micron membrane filter before each use. Stable for one month, if refrigerated.

4. Stock Phenol (1000 mg/L): In a 1 liter volumetric flask, dissolve 1.00 g of phenol in 500 mL of Milli-Q water. Add 2 mL concentrated H_2SO_4 as a preservative. Dilute to 1000 mL. (1.0 mL = 1.0 mg phenol).

5. Working Stock Standard (10.0 mg/L): In a 500 mL volumetric flask, pipet 5.0 mL of the stock phenol and dilute to the mark with Milli-Q water. (Prepare fresh daily!)

6. Standards (Prepare fresh daily!)

Concentration of Standard	Letter Identifier	Volume of 10 mg/L working standard	Dilute to
0.000 mg/L	A	0.0	100 mL
0.050 mg/L	B	0.5	100 mL
0.100 mg/L	C	1.0	100 mL
0.200 mg/L	D	2.0	100 mL
0.500 mg/L	E	5.0	100 mL

Note: Computer refers to standards by letter.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. The gain and zero are guidelines, they must be set each day to optimize.

3. Interferences:

   - Interferences are eliminated or reduced to a minimum by the distillation procedure.

- Color will interfere, dilute the sample and spike the sample to confirm the quality of the result.

- Turbidity will interfere. If present, filter through a 0.45 micron membrane filter prior to analysis on Lachat System.

- A peak with a shoulder indicates an interference. If this occurs, dilute the sample. If the problem still exists, redistill the sample and it's spike at a dilution.

System Operation:

1. Refer to "Auto Analyzer Operation Start-up procedure". (SOP# LAA-section A.)

2. Spikes will be distilled at a level of 0.4 mg/L. The calibration check standard is 0.100 mg/L (D).

3. Analyze a blank and a 0.1 standard at the beginning of each run. An EPA standard will only be analyzed monthly due to the inavailability of outside standards.

4. If a sample and spike are overrange:

   - Dilute the sample and spike if dilution is $\leq$ 1:5. The distilled standard will still be detectable.
   - Dilute the sample and do a manual spike if dilution is > 1:5. A manual spike is prepared by mixing 5 mL diluted sample + 5 mL of 0.2 standard.

5. Refer to Auto Analyzer shut-down proceduere. (SOP# LAA-section B.)

Quality Control:

1. Establish a standard curve with the standards listed above A laboratory blank will be analyzed and must be less than the detection limit, otherwise troubleshooting must be performed. Record the check standard in the check book. The concentration should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check reagents, pump tubing, etc.).

2. A quality control calibration standard of 0.100 mg/L is to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within the acceptable ranges

or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standards in the quality control book. The confidence limits are noted in the quality control book.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Duplicates are to be averaged. Spike recoveries and duplicates are to be within acceptable ranges or trouble-shooting must be performed.

Calculations:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer.

ALKALINITY - AUTOANALYZER

Scope and Application: This method is applicable to drinking water, surface water, groundwater and wastewater.

Reference: EPA 1983, Method 310.2
Lachat Instruments 1986, Method 10-303-31-1-C

Sample Handling: Refrigerate at 4°C and analyze within 14 days of collection.

Detection Limit: 5.0 mg/L as $CaCO_3$

Optimum Concentration Range: 5.0 - 500 mg/L

Instrument Conditions:

1. Pump speed: 35
2. Cycle period: 60 seconds
3. Load period: 30 seconds
4. Inject period: 15 seconds
5. Inject to start of peak period: 10 seconds
6. Inject to end of peak period: 56 seconds
7. Gain: 150 x 10
8. Zero: 180
9. Interference filter: 410 nm
10. Sample loop: 90 cm
11. Standards for curve set-up: 0, 20.0, 50.0, 100, 250, 500 mg/L.

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise stated.)

1. Degassed Milli-Q water - 2 options:

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.

   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water (15 min/20 L.) Store in cubitainer.

2. Stock alkalinity standard (1000 mg/L as Na_2CO_3): In a 1 liter volumetric flask, dissolve 1.060 g of anhydrous primary standard grade sodium carbonate (Na_2CO_3-dried at 140°C for 4 hours) in approximately 900 mL of helium purged Milli-Q water, and dilute to mark.

3. Standards: (Prepare fresh every 2 months.)

Concentration of Standard	Letter Identifier	Volume of Alk. Standard	Dilute to
0 mg/L	A	0	200 mL
5 mg/L	B	1.0	200 mL
20.0 mg/L	C	4.0	200 mL
50.0 mg/L	D	10.0	200 mL
100 mg/L	E	50.0	500 mL
250 mg/L	F	125.0	500 mL
500 mg/L	G	100.0	200 mL

NOTE: Final volumes are not the same!
Computer refers to standards by letter.

4. Sodium hydroxide (0.1M): In a 1 liter flask, dissolve 4.0 g sodium hydroxide (NaOH) and dilute to the mark with Milli-Q water.

5. Hydrochloric acid (0.1M): In a 1 liter flask, dilute 8.3 mL of concentrated HCL in Milli-Q water and dilute to the mark.

6. KHP buffer (25.0 mM, pH 3.1): In a 1 liter flask, dissolve 5.10 g of potassium acid phthalate (KHP) ($KHC_3H_4O_4$) in approximately 500 mL of helium purged Milli-Q water. Add 87.6 mL of 0.1M HCL and dilute to the mark. Adjust the pH of the buffer to 3.1 with 0.1M HCL or 0.1M NaOH. STORE IN GLASS AND PREPARE MONTHLY!

7. Methyl orange reagent: In a 1 liter volumetric flask, dissolve 0.125 g of methyl orange indicator in about 700 mL of helium-purged Milli-Q water and dilute to the mark. Store in glass!

Notes:

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. The gain and zero settings are guidelines and must be adjusted each day to optimize.

3. The alkalinity standards can be combined with chloride and sulfate standards for use with the 3 channel method.

4. Turbidity will interfere. Samples must be filtered prior to analysis. (Use Whatman #1 or #4.)

5. Color will interfere, dilute the sample and also spike this sample to confirm the quality of the result.

System Operation:

A. Refer to "Auto Analyzer Operation start-up procedure." (IOP# LAA-section A)

B. Analyze a blank and an EPA check standard at the beginning of each run.

C. Use 125 mg/L for the spike level.

D. The calibration check standard is 100 mg/L (D).

E. Refer to "Auto Analyzer shut-down procedure". (IOP# LAA-section B)

Quality Control:

1. Establish a standard curve with the standards listed above A laboratory blank will be analyzed and must be less than the detection limit, otherwise troubleshooting must be performed. Record the check standard in the check standard book. The concentration should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check reagents, pump tubing, valves, etc.).

2. A quality control calibration standard of 100 mg/L is to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within the acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standards in the quality control book. The acceptable limits are noted in the quality control book.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Duplicates are to be averaged. Spike samples with a standard in a 1:1 ratio of sample to standard. (Example: 5 mL sample + 5 mL 20.0 standard.) Spike recoveries and duplicates are to be within acceptable ranges or troubleshooting must be performed.

Calculation:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer.

CHLORIDE - AUTOANALYZER

Scope and Application: This method is applicable to drinking water, surface water, groundwater, and wastewater.

References: EPA 1983, Method 325.2
Lachat Instruments 1986, Method 10-117-07-1-B

Sample Handling: Refrigerate at 4°C and analyze within 28 days of collection.

Detection Limit: 1.0 mg/L.

Optimum Concentration Range: 1.0 - 100 mg/L

Instrument Conditions:

1. Pump speed: 35
2. Cycle speed: 30 seconds
3. Load period: 15 seconds
4. Inject period: 15 seconds
5. Inject to start of peak period: 8 seconds
6. Inject to end of peak period: 35 seconds
7. Gain: 200
8. Zero: 250
9. Interference filter; 480 nm
10. Sample loop: 20 cm
11. Standards for curve set-up: 0, 10.0, 20.0, 50.0, 80.0, 100.

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. Degassed Milli-Q water - 2 options:

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.

   b. Bubble helium, using the fritted gas dispersion tube, through the Milli-Q water. (15 min/20 L.) Store in cubitainer.

2. Stock chloride standard (1000 mg/L Cl): In a 1 liter volumetric flask, dissolve 1.648 g of primary grade sodium chloride (NaCl), previously dried at 103°C, in 500 mL Milli-Q water. Dilute to the mark and invert to mix.

3. Standards: (Prepare fresh every 2 months.)

Concentration of Standard	Letter Identifier	Volume of Cl Standard	Dilute to
0 mg/L	A	0	200 mL
5 mg/L	B	1.0	200 mL
10.0 mg/L	C	2.0	200 mL
20.0 mg/L	D	4.0	200 mL
50.0 mg/L	E	25.0	500 mL
80.0 mg/L	F	40.0	500 mL
100 mg/L	G	20.0	200 mL

Note: Final volumes are not the same!
Computer refers to standards by letter.

4. Stock mercuric thiocyanate reagent: In a 1 liter volumetric flask, dissolve 4.17g of mercuric thiocyanate ($Hg(SCN)_2$) in one liter of methanol. Invert to mix. Store in amber glass.

   CAUTION: Mercury is a very toxic metal. WEAR GLOVES!

5. Stock ferric nitrate reagent (0.5M): In a 1 liter volumetric flask, dissolve 202.0g of ferric nitrate ($Fe(NO_3)_3 \cdot 9H_2O$) in approximately 800 mL of deionized water. Add 25 mL of concentrated nitric acid and dilute to one liter. Invert to mix.

6. Combined color reagent: Mix 150 mL of stock mercuric thiocyanate solution with 150 mL of stock ferric nitrate reagent and dilute to 1000 mL with deionized-water. Vacuum filter through a 0.45 micron membrane filter.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum working range.

2. The gain and zero settings are guidelines and must be adjusted each day to optimize.

3. The chloride standards may be combined with alkalinity and sulfate standards for use with the 3 channel method.

4. Any sample with turbidity must be filtered prior to analysis. (Use Whatman #1 or #4.)

5. Color is an interference, dilute the sample and also spike this sample to confirm the quality of the result.

System Operation:

A. Refer to "Auto Analyzer Operation Start-up procedure". (IOP# LAA-section A)

B. Analyze a blank and an EPA check standard at the beginning of each run.

C. Use a 40 ppm Cl for the spike level.

D. The calibration check standard is 50 mg/L (E).

E. Refer to "Auto Analyzer Shut-down procedure". (IOP# LAA-section B)

Quality Control:

1. Establish a standard curve with the standards listed above A laboratory blank will be analyzed and must be less than the detection limit, otherwise troubleshooting must be performed. Record the check standard in the check standard book. The concentration should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check reagent, pump tubing, valves, etc.).

2. A quality control calibration standard of 50.0 mg/L is to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard is still required. The last sample analyzed in the run is to be the calibration standard. These standards must be within the acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standards in the quality control book. The acceptable limits are noted in the quality control book.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Duplicates are to be averaged. Spike samples with a standard in a 1:1 ratio of sample to standard. (Example: 5 mL sample + 5 mL 20.0 standard.) Spike recoveries and duplicates are to be within acceptable ranges or troubleshooting must be performed.

Calculations:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer.

CYANIDE, TOTAL

Scope and Application: This method is applicable to the determination of cyanide in drinking water, surface water, groundwater, sludges, soils, and industrial wastes.

Method: Distillation, Spectrophotometric

Reference: EPA 1983, Method 335.2

SW-846, Method 9010

Standard Methods, 16th Edition, Method 412.

Detection Limit: 0.005 mg/L

Optimum Range: 0.01 - 0.20 mg/L

Sample Handling: Preserve with sodium hydroxide to pH>12 and refrigerate at 4°C. Analyze within 14 days.

Reagents and Apparatus:

1. Cyanide reflux distillation apparatus
2. Spectrophotometer, set at 578 nm
3. 25 mL and 50 mL graduated cylinders
4. Vacuum pump
5. Heating mantle
6. Thermometer
7. 250 mL volumetric flask
8. 50 mL mixing cylinders
9. Eppendorf macropipeter, 1-5 mL
10. Sodium hydroxide solution, 1.25N
11. Sulfuric acid, concentrated
12. Magnesium chloride solution
13. Sodium phosphate solution
14. Chloramine-T solution
15. Pyridine-barbituric acid solution
16. Deionized water
17. Bismuth nitrate solution
18. Sulfamic acid solution
19. Acetic acid, concentrated
20. Sodium thiosulfate crystals

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. Sodium Hydroxide, 1.25N: Dissolve 50.0 g NaOH in D.I. water and dilute to 1 liter in a volumetric flask. Store in plastic bottle.

2. Sodium Hydroxide, 0.25N: Dilute 200 mL of 1.25N sodium hydroxide solution to 1 liter with D.I water in a volumetric flask. Store in plastic bottle. (This solution is used for diluting samples.)

3. Magnesium Chloride Solution: Dissolve 510.0 g $MgCl_2 \cdot 6H_2O$ in D.I. water and dilute to 1 liter.

4. Sodium dihydrogenphosphate Solution, 1M: Dissolve 138.0 g sodium dihydrogenphosphate ($NaH_2PO_4 \cdot H_2O$) in D.I water and dilute to 1 liter in a volumetric flask. (Store in refrigerator.)

5. Chloramine-T Solution: Dissolve 1.0 g chloramine-T in D.I. water and dilute to 100 mL in a volumetric flask. Store in refrigerator. Prepare fresh daily.

6. Pyridine-Barbituric Acid Reagent: Prepare in hood! Transfer 15.0 g barbituric acid to a 250 mL volumetric flask. Wash down sides of flask and wet barbituric acid with D.I. water. Add 75 mL pyridine with graduated cylinder and mix. Add 15 mL concentrated hydrochloric acid and mix. Cool, dilute to 250 mL with D.I in a volumetric flask and mix. Store in refrigerator. Place in a dark bottle for storage.

7. Stock Cyanide Solution, 1000 mg/L: Dissolve 2.51 g KCN and 2.0 g KOH and dilute to 1 liter with D.I. water in a volumetric flask. Standardize against standard silver nitrate titrant each use. Refer to Standard Methods, 16th Edition, pp. 336-337, Method 412C for the standard procedure. Solution loses strength with time. If, upon standardizing, the solution falls below 980 mg/L cyanide, the stock must be remade; otherwise, prepare fresh every 6 months. CAUTION: TOXIC!

8. Standard Cyanide Solution, 5 mg/L: Dilute 5 mL of stock cyanide solution to 1 liter with D.I. water using a volumetric flasks. Prepare fresh daily.

9. Bismuth nitrate Solution: Dissolve 30.0 g of $Bi(NO_3)_3$ in 100 mL of D.I. water. While stirring, add 250 mL of concentrated acetic acid. Stir until dissolved. Dilute to 1 liter with D.I. water.

10. Sulfamic Acid Solution: Dissolve 40.0 g of sulfamic acid in D.I. water. Dilute to 1 liter.

11. Cyanide Standards: Prepare by pipetting the volumes noted below into 250 mL volumetric flasks, adding 50 mL of 1.25N NaOH, and diluting to the mark with D.I. water. (The 1.25N NaOH must be added-very important!)

mL of Standard Cyanide Solution, 5 mg/L	Cyanide Concentration mg/L
0.5	0.01
1.0	0.02
5.0	0.10
10.0	0.20
20.0	0.40

Notes:

1. The colorimetric determination should be performed in the hood or by the downdraft area.

2. If samples need to be diluted during the colorimetric determination, take an aliquot of sample, using volumetric pipettes, and dilute to 50 mL with 0.25N sodium hydroxide solution. Continue with procedure as written.

3. After the addition of chloramine-T, CNCl is formed. CAUTION: CNCl is a toxic gas; avoid inhalation.

5. CAUTION: Use care in handling of samples with cyanide because of the toxicity. Avoid skin contact, inhalation, or ingestion. ALWAYS HAVE A RESPIRATOR IN AREA WHEN DOING THIS TEST.

   If a sample begins to bump or back up the tube, quickly increase the flow rate, pull out the inlet tube, turn the heat off. The distillation must be repeated on a fresh sample.

   If a sample does boil over proceed as follows:

   - Put on respirator
   - Turn heat off (For your proctection, use gloves.)
   - Pull inlet tube out
   - Put sample into hood

5. Oxidizing agents, such as chlorine, interfere by decomposing cyanides. If chlorine is believed to be present, put a drop of sample on potassium iodide starch paper. If paper turns bluish, add a few crystals of sodium thiosulfate ($Na_2S_2O_3$) to the sample, mix, and retest. Continue adding sodium thiosulfate until free from chlorine. Then, add 0.1 g sodium thiosulfate in excess.

6. Sulfides interfere by forming thiocyanate at a high pH. If sulfides are believed to be present, put a drop of sample on lead acetate test paper treated with acetic acid buffer solution at pH 4. Darkening of paper indicates sulfides. If sulfides are present, add 50 mL of bismuth nitrate solution after the air rate is set through the air inlet tube. Mix for 3 minutes prior to addition of H_2SO_4. Alternatively, $Cd(NO_3)_2 \cdot 4H_2O$, $CdCO_3$ or $PbCO_3$ can be added after the distillation, but prior to color development. Bismith nitrate added prior to the distillation process is the preferred choice.

7. Fatty acids, high carbonates, and aldehydes can interfere. Refer to Standard Methods for troubleshooting.

8. High concentrations of NO_3 and NO_2 can give false positive results. If samples contain high concentrations of NO_3 and/or NO_2, add 50 mL of sulfamic acid solution after the air rate is set through the air inlet tube. Mix for 3 minutes prior to addition of H_2SO_4.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed, and deionized rinsed prior to analyses. Dichromate and acetone may also be used to clean the glassware prior to the soap and water wash.

2. The standard curve is to consist of the following standards:

   deionized water blank
   0.01 mg/L
   0.02 mg/L
   0.10 mg/L
   0.20 mg/L
   0.40 mg/L

   These standards do not have to be carried through the distillation step.

3. Connect and set up cyanide reflux distillation apparatus as shown in Figure 2. (See pg. CN1-7)

4. Pour 500 mL of sample into cyanide distilling flask. If a solid or semi-solid sample is to be analyzed, use a 1-5 g sample size and add 500 mL of D.I. water to the distilling flask. (Record the amount of sample used.)

   To Spike: Add 10 mL of standard cyanide solution to the 500 mL of sample.

5. Using a graduated cylinder, add 50 mL 1.25N sodium hydroxide to the absorber tube and connect.

6. Turn on vacuum pump and adjust so that one bubble per second enters the distillation flask through the air inlet tube.

7. Add 50 mL of $Bi(NO_3)_3$ solution to flask.

8. Slowly add 25 mL concentrated sulfuric acid through the air inlet tube. Rinse the tube with D.I. water and wait for about 2-3 minutes, until the sulfuric acid has been dispersed into the sample.

9. Using a graduated cylinder, add 20 mL magnesium chloride solution into the air inlet tube and rinse the tube with D.I. water.

10. Turn heating mantle on to 60-63% of scale. Watch vacuum rate carefully and adjust as necessary maintaining a rate of one bubble per second. As the temperature increases, bubbling increases, and the solution can be drawn from the absorption tube or blown out the air inlet tube. Reflux for one hour after the sample comes to a boil.

11. Turn off heat and continue vacuum for 15 minutes.

12. Disconnect absorber and shut off vacuum pump.

13. Pour solution from absorber tube into a 250 mL volumetric flask. Using D.I. water, rinse the absorption tube (3 times) and add to the volumetric flask. Dilute to mark with washings from the absorber. Mix by inverting.

Note: Steps 14-19 should be done under the hood.

14. Pour 50 mL of the distillate into a 100 mL mixing cylinder. If less than 50 mL of sample is used, dilute to 50 mL with 0.25N NaOH solution.

15. Add 15 mL sodium phosphate solution.

16. Using the Eppendorf micropipeter, add 2 mL chloramine-T solution. Mix and wait 1 to 2 minutes.

    Note: Sulfide will form a cloudy precipitate with the addition of the chloramine-T. If any cloudiness is seen, treat the distillate with $CdCO_3$ to remove excess sulfide. If precipitate is very heavy, redistill sample adding additional $BiNO_3$ solution.

17. Using the Oxford macropipeter, add 5.0 mL pyridine-barbituric acid solution.

18. Add D.I. water to the 100 mL mark. Stopper and mix.

19. After 8 but before 15 minutes, read absorbance on a spectrophotometer set at 578 nm. Use sipper cell and zero spectrophotometer with the undistilled reagent blank.

Quality Control:

1. Establish a standard curve with the standards listed in the procedure section. Record the absorbance of the 0.10 mg/L standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, check the wavelength, spectrophotometer bulb, solutions and do other necessary troubleshooting.

2. A reagent blank is to be analyzed with each set of samples. This blank is to be carried through the distillation steps as a check for contamination.

3. A quality control calibration standard of 0.10 mg/L cyanide is to be analyzed initially, and at a minimum after every 10 samples. This standard is to be carried through the entire procedure including the distillation step. The last sample analyzed in the run is to be the calibration standard. These standards must be within acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standards in the quality control book. The confidence limits are noted in the quality control book.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Duplicates are to be averaged. Spike recoveries and duplicate results are to be within acceptable ranges.

5. Aqueous and solid/semi-solid samples are separate matrices. Duplicates and spikes are required for each matrix type.

<u>Calculation</u>: Calculate the cyanide in the samples as follows:

$$\text{CN, mg/L} = \frac{A}{B} \times \frac{250}{C}$$

A = concentration from standard curve
B = mL of original sample for distillation
C = mL of color development

Calculate the spike recovery as follows:

$$\frac{(\text{Spike value} - \text{sample value})}{0.10} \times 100 = \%\ \text{recovery}$$

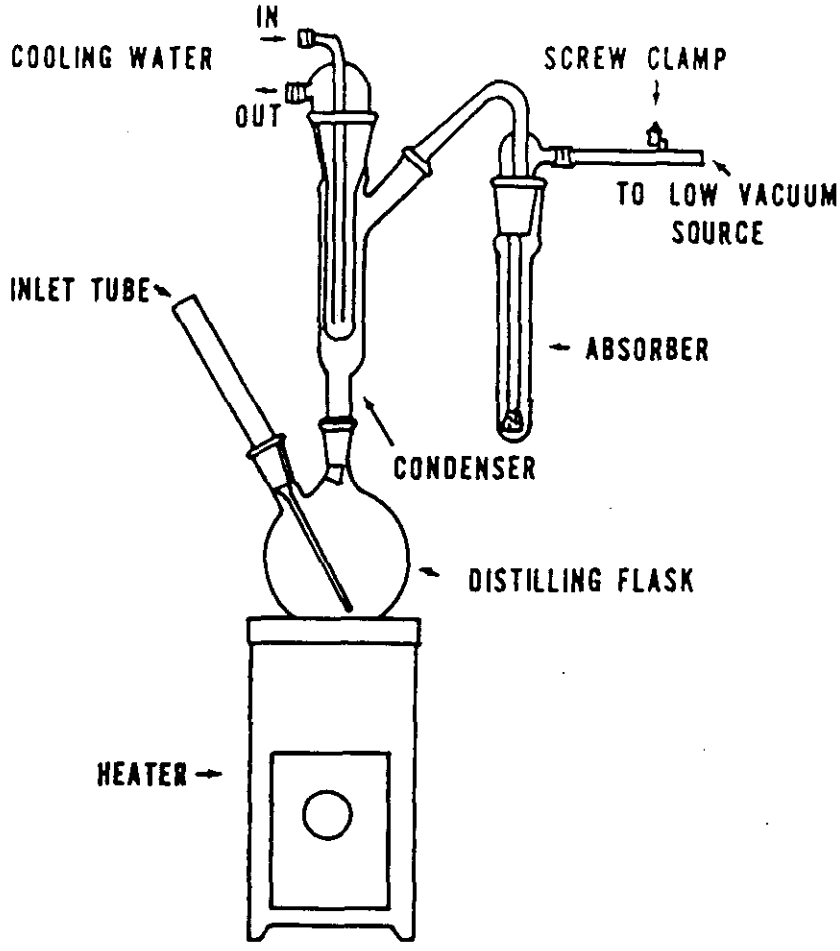

FIGURE 2

CYANIDE DISTILLATION APPARATUS

	Revised Date
Michael J. Linskens Laboratory Manager	7-23-86
Kim D. Finner Analytical Laboratory QA/QC Officer	4-27-87
Thomas J. Lynch QA Officer	

B) HAZLETON PROCEDURES

VOLATILE ORGANICS ANALYSIS USING HAZLETON'S METHOD

Within this Appendix is a standard operating procedure (SOP) describing the method to be used by Hazleton Laboratories America, Inc., in analyzing samples for volatile organic compounds. The following supplements information in the method description.

The VOC target analyte list for this study based on prior data collected at the site is:

1. tetrachloroethene
2. trichloroethene
3. cis-1,2 dichloroethene
4. trans-1,2 dichloroethene
5. vinyl chloride
6. 1,1,1 trichloroethane
7. 1,1 dichloroethane
8. chloroethane
9. 1,2 dichloroethane
10. 1,2 dichloropropane

Spiking compounds to be used for MS/MSD samples at a 20 ppb spike are:

1. trichloroethene
2. cis-1,2 dichloroethene
3. 1,1,1 trichloroethane
4. chloroethane
5. benzene

Surrogates to be used are:

1. 1,4-dichlorobenzene (for HECD)
2. flurobenzene (for PID)

Method recovery data are not currently available for one of the target compounds (chloroethane), but will be available prior to use of the method.

Quality assurance objectives will be as stated in the method SOP with the following exceptions:

a. Prepare calibration standards at a minimum of three concentration levels. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in the samples or should define the working range of the detector.

b. Acceptable spike recoveries should be 85 to 115 percent. Surrogates recoveries acceptable ranges are from 80 to 120 percent. Samples will be re-analyzed if outside the surrogate range. If surrogate recoveries are outside the acceptable range on re-analysis the data will be flagged and footnoted as such.

c. Instruments will be recalibrated when results of continuing calibration exceed 20 relative percent difference (RPD) from the intitial calibration for target compounds and 40% for non-target compounds.

d. Matrix spike/matrix spike duplicate will be performed with each set of twenty or less samples analyzed with 15% RPD as acceptance criteria.

e. A method blank will be run at least daily.

f. Hazleton will be responsible for an initial data assessment of GC-GC/MS confirmation data. Any unresolved problems will be addressed in their case narrative. Any GC-GC/MS data that remain unresolved will be flagged, with limitations noted.

Reportables for the GC analysis using Hazleton's method will consist of:

a. Initial calibration summary and chromatograms
b. Continuing calibration summary.
c. QC package including MS/MSD recoveries and RPDs with chromatograms.
d. Blank package including blank summary table with chromatograms.
e. Sample results and chromatograms.
f. Case narrative containing summary of any QC, sample, shipment and analytical problems, and documentation of all internal decision tree process used. Outline problems encountered and final solutions.

Hazleton is instructed to analyze field blank samples prior to analyzing tip blank samples. If the field blank is "clean" at the method detection limit, the trip blank will not be analyzed.

Samples for GC/MS confirmation will be preselected based on results of prior sampling. Samples will be selected to cover the range of concentrations detected with a uniform distribution within the range.

GC/MS will be used to confirm the presence of detected compounds by Hazleton's method. Instrument conditions will conform to those used for CLP RAS volatiles analysis. A 25 ml purge volume will be used rather than the normal 5 ml in order to provide approximately comparable sensitivity (see Appendix A for detection limits).

Double the normal sample volume (4, 40 ml VOA vials) will be collected for all VOC analyses. This will allow some flexibility in the selection of samples for confirmation by GC/MS and provide sufficient sample for MS/MSD analyses. A minimum of 10% GC/MS confirmation of samples is anticipated.

If dilutions are performed, results of analysis for both diluted and undiluted samples must be reported.

[sss 800-91]

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PROCEDURE TITLE: Determination of Volatile Synthetic Organic Compounds in Water by Purge and Trap Method

AREA OF APPLICABILITY: Hazleton Laboratories America, Inc. Environmental Analysis

SCOPE AND APPLICATIONS:

1. This method covers the determination of these 36 volatile organics.

Parameter	CAS Number
Chloroethane	75-00-3
Benzene	71-43-2
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
2-Chloroethyl vinyl ether	100-75-8
Chloroform	67-66-3
Chloromethane	74-87-3
Dibromochloromethane	124-48-1
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-1
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethane ene	75-35-4
cis-1,2-Dichloroethane ene	156-59-2
trans-1,2-Dichloroethane ene	156-60-5
Dichloromethane	75-09-2
1,2-Dichloropropane	78-87-5
cis-1,3-Dichloropropene	10061-01-5
trans-1,3-Dichloropropene	10061-02-6
Ethylbenzene	100-41-4
Styrene	100-42-5
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichloroflouromethane	75-69-4
Vinyl chloride	75-01-4
O-Xylene	95-47-6
m-Xylene	108-38-3
p-Xylene	106-42-3

2. This is a purge and trap GC method applicable to the determination of the compounds listed above in drinking water, ground water, and wastewater.

3. The GC conditions and quantitation limits for each parameter are listed in Table 1.

PRINCIPLE:

1 An inert gas is bubbled through a 5.0-mL water sample contained in a specially designed purging chamber. The volatiles are efficiently transferred from the aqueous phase to the vapor phase. The vapor phase is swept through a solvent tube where the volatiles are trapped. After the purge is completed, the trap is heated and backflushed with gas to desorb the volatiles into a GC system. A temperature program is used in the GC system to separate the volatiles before detection with a photoionization detector and then a halide-specific detector (connected in series).

2. GC-MS according to Method 524.1 can be used to confirm GC identifications and measurements if sufficient concentrations are present.

SENSITIVITY, PRECISION, ACCURACY:

The limits of detection listed in Table 1 represent sensitivities that can be achieved in ground water in the absence of interference.

Interlaboratory precision and accuracy data for some compounds have been published by the U.S. EPA (Federal Register, Methods 601 and 602, References 5 and 6). Preliminary in-house precision and accuracy for this method are presented in Tables 2 and 4.

REFERENCES:

1. U.S. Environmental Protection Agency, "National Primary Drinking Water Regulations," Fed. Register, 40(141) (November 13, 1985).

2. U.S. Environmental Protection Agency, Method 501.1, "The Analysis of Trihalomethanes in Finished Waters by the Purge and Trap Method," Environmental Monitoring and Support Laboratory, Cincinnati, Ohio (November 1979).

3. U.S. Environmental Protection Agency, Method 502.1 for "Volatile Halogenated Organic Compounds in Water by Purge and Trap Gas Chromatography," Environmental Monitoring and Support Laboratory, Cincinnati, Ohio (November 1985).

4. U.S. Environmental Protection Agency, Method 503.1 for "Volatile Aromatic and Unsaturated Organic Compounds in Water by Purge and Trap Gas Chromatography," Environmental Monitoring and Support Laboratory, Cincinnati, Ohio (November 1985).

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5. U.S. Environmental Protection Agency, Method 601 for "Purgeable Halocarbons in Municipal and Industrial Wastewater," Fed. Register, 49(209) (October 26, 1984).

6. U.S. Environmental Protection Agency, Method 602 for "Purgeable Aromatics in Municipal and Industrial Wastewater," Fed. Register, 49(209) (October 26, 1984).

7. Minnesota Department of Health, "Determination of Volatile Organics in Water by Purge and Trap Method, Minn. DOH, Division of Environmental Health, Method Code 465-B (January 10, 1983).

APPROVED BY: _____ DATE: _____
David C. Hills
Environmental Analysis

REVIEWED BY: _____ DATE: _____
Debra Curley Arndt
Manager, Quality Assurance Unit

(1005D)

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SAFETY PRECAUTIONS:

1. The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each laboratory is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Additional references to laboratory safety are available for the information of the analyst.

2. The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, 1,4-dichlorobenzene, hexachlorobutadiene, tetrachloroethene, trichloroethene, carbon tetrachloride, bis-2-chloroisopropyl ether, 1,2-dichloroethane, 1,1,2,2-tetrachloroethene , 1,1,2-trichloroethane, chloroform, 1,2-dibromomethane, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

INTERFERENCES:

1. Samples may be contaminated during shipment or storage by diffusion of volatile organics through the septum seal into the sample. Field blanks must be analyzed to determine if contamination has occurred.

2. During analysis, major contaminant sources are volatile materials in the laboratory (dichloromethane and chloroform) and impurities in the inert purging gas and the sorbent trap. The analytical system must be demonstrated to be free from contamination by running laboratory blanks under the condition of analysis. When potential interfering peaks are noted in laboratory reagent blanks, the analyst must eliminate the problem before analyzing samples. The use of polytetraflouroethylene (PTFE) tubing and thread solvents, and stainless steel flow controllers should be used where applicable in the system.

3. Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. Rinsing the sample syringe and purging device twice with organic free water between samples is a preventative technique. Screening of samples on a alternate system by head space/direct injection can be used when levels requiring dilutions are suspected. Analyses of laboratory reagent blanks must be run after analysis of a sample containing high levels, to demonstrate acceptable background levels.

4. The lamp window on the photoionization detector will have to be cleaned occasionally because of a film build up that reduces sensitivity. Excess water will cause a negative baseline deflection in the baseline (PID). The method provides for a trap bake period to prevent this problem.

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QUALITY ASSURANCE/QUALITY CONTROL:

1. The performance of the entire analytical system can be checked daily using the data collected from the analysis of method blanks, field blanks, sample matrix spikes, sample matrix spike duplicates, and calibration check standards.

2. The performance of the analytical system for each individual sample and standard is monitored by the use of surrogate standards. Results for the surrogates should be within ± 20% of the true value (2 µg/L 1,4-Dichlorobutane and 5 µg/L fluorobenzene).

3. EPA quality control check standards are run at least quarterly. If measured analyte concentrations are not within acceptance limits provided with sample, check the entire analytical procedure to locate and correct the problem source.

APPARATUS AND EQUIPMENT

1. Sample containers. Forty-milliliter screw cap vials with PTFE-faced silicone septum seals. Wash vials and seals with detergent, rinse with tap, distilled water, and dry at 105°C (septum dry for 1 hour only), allow to cool in area known to be free of organics.

2. Purge and Trap Systems (Tekmar LSC-2)

   2.1 All Glass Purging Device. Capable of accepting 5-mL samples with a water column at least 3 cm deep. A glass frit installed at the base of sample chamber allowing the purge gas to pass through the water column as finely divided bubbles with a diameter <3 mm at the origin.

   2.2 Volatile Trap. At least 25 cm long and have an inside diameter of at least 0.105 in. starting from the inlet, the trap must contain the following amounts of adsorbents: 1/3 of 2,6-diphenylene oxide polymer, 1/3 of silica gel, and 1/3 of coconut charcoal. Prior to daily use, the trap is conditioned for 10 minutes at 220°C while backflushing with an inert gas flow of at least 20 mL/minute. The trap effluent is vented to the room.

   2.3 Desorber. Capable of rapidly preheating the trap to 180°C, then desorbing the trap to the GC column while maintaining the temperature at 180°C.

3. Gas Chromatography System

   3.1. Gas Chromatograph (Varian 3700 GC). Capable of temperature programming and achieving an initial column temperature of 30°C. Variable-constant differential flow controllers capable of maintaining constant flow rates throughout the desorption and temperature program.

3.2 Gas Chromatography Column. 60 m x 0.75 mm i.d. wide bore glass capillary, coated with VOCOL (Supelco No. 2-3731) at 1.50 µm d_f. The column flow rate of helium carrier gas is established at 15 mL/minute, make-up gas flow rate of helium carrier gas is established at 15 mL/minute. The column temperature is programmed to hold at 30°C for 8 minutes; increased to 190°C at 4°C/minute, or until all expected compounds have eluted. Sample chromatograms obtained with the above column are presented in Figures 1 and 2.

3.3 Two detectors in series are utilized in this method. First, a nondestructive photoionization detector capable of detecting compounds with an ionization voltage less than or equal to the energy of the ultraviolet light source. Second, an electrolytic conductivity detector specific for halogenated compounds (Hall 700A).

3.4 HNU photoionization detector (HNU-Systems, Inc., Model PI-52) equipped with a 10.2 eV ultraviolet lamp. Table 4 shows accuracy and precision data collected with this detector. Estimated instrument detection limits are expressed in Table 3. The PID operating conditions are as follows:

Detector base temperature: 170°C
Detector input: 10
Detector offset: 360
Recorder attenuation: 32
Carrier gas: Helium at total 30 mL/minute

3.5 Tracor Hall Model 700A electroconductivity detector. Table 2 shows accuracy and precision data collected with this detector. Instrument detection limits are expressed in Table 1. The operating conditions used to collect these data are as follows.

Reactor tube: Nickel 1/16 in. OD
Reactor temperature: 900°C
Reactor base temperature: 250°C
Electrolyte: 100% n-propyl alcohol
Electrolyte flow rate: 0.8 mL/minute
Reaction gas: Hydrogen at 40 mL/minute
Carrier gas: Helium at total 30 mL/minute

3.4 Syringe and Syringe Valves

3.4.1 Syringes - 5-mL glass hypodermic with Luerlock tip (two each)

3.4.2 Micro syringes - 25 and 100 µL

3.4.3 Gas syringe - 1.0 and 5 mL, gas tight with shut off valve

3.5 Miscellaneous

5.5.1 Standard storage containers - 3.7-mL screw cap amber vials

5.5.2 Mininert valves - Screw cap

REAGENTS

1. Methanol, demonstrated to be free of analytes (spike 100 mL into 5-mL reagent water and analyze. Should produce less than 0.4 µg/L response).

2. Reagent water, producing less than 0.2 µg/L response for those compounds that are monitored. Prepare by boiling distilled or natural waters for 15 minutes followed by a 1-h purge with inert gas while temperature is held at 90°C or carbon filtered. Store in clean, narrow mouthed crimp top PTFE-lined septa bottles.

3. Stock standard: Commercial mixed stock solutions are available (Supelco Purgeables A, B, and C) that contain most of the compounds of interest at a concentration of 0.2 mg/mL. Stock solutions must be prepared from neat, as follows for those compounds not included in the commercial mixes.(1)

   3.1 Methanol (24.5 mL) is placed in a 25-mL volumetric flask. Allow flask to stand unstoppered for 10 minutes or until all alcohol-wetted surfaces have dried and tare.

   3.2 Using a 100-µL syringe, add 50 mg of assayed reference material to the flask. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask. Retare the flask and add 50 mg of the next compound. Repeat process until all compounds have been added.

   3.3 Dilute to volume, and stopper. Mix by inverting flask several times. The resulting solution will contain each analyte at a concentration of 2.0 mg/mL.

   3.4 Store stock standard solutions in 3-mL vials equipped with PTFE mininert valve tops at <0°C. All standards must be replaced each month.

(1) The following compounds must be made from neat: Cis-1,2-Dichloroethane, o-xylene, m-xylene, p-xylene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, 1,2-Dichlorobenzene, Styrene.

3.5 Secondary Dilution Standards. Using stock standards, prepare secondary dilution standards in methanol. The secondary dilution standards are prepared at concentrations that can easily be diluted to prepare aqueous calibration standards that will bracket the working range of the method.

3.5.1 To prepare secondary dilution standards place 9.0 mL of methanol into a 10 mL volumetric flask.

3.5.2 Inject exactly 250 µL of the Supelco Purgeable A stock solution, 250 µL of the Supelco Purgeable B stock solution, and 25 µL of the stock solution prepared from neat (above) into the methanol. When prepared as above, the solution will contain each analyte at a concentration of 5 ng/µL.

Separate secondary dilution standards should be prepared weekly for the gases from the Supelco Purgeables C mix.

3.5.3 Store secondary dilution standards in 3-mL glass vials equipped with PTFF mininert valve screw tops. Storage conditions and times described for stock standard solutions also apply to secondary dilution standard solutions.

4. Sample spiking solution: Place 9.5 mL of methanol into a 10 mL volumetric flask, add 250 µl of Supelco Purgeable A or Supelco Purgeable B (not both). Dilute to volume and mix. The resulting solution will contain the analytes listed in Table 5 at a concentration of 5 ng/µl. Store at <0°C. The sample spiking solution should be discarded after 1 month.

5. Surrogate standard: Prepare from neat 1,4-Dichlorobutane (50 mg) and flourobenzene (125 mg) as in Section 3 above. The resulting stock solution will contain bromochloromethane at 2.0 mg/mL and flourobenzene at 5.0 mg/mL.

5.1 Dilute 25 µl of stock in 25 mL of methanol. The internal surgogate standard when prepared as above will contain 1,4-Dichlorobutane at a concentration of 2 ng/µl and flourobenzene at a concentration of 5 ng/µl.

SAMPLE COLLECTION, PRESERVATION, AND STORAGE

1. Sample Collection

1.1 Replicate field blanks must be handled along with each sample set, which is composed of the samples collected from the same general sampling site at approximately the same time. At the laboratory, fill a minimum of two sample bottles with reagent water, seal, and ship to the sampling site along with empty sample bottles. Wherever a set of samples is shipped and stored, it must be accompanied by field blanks.

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1.2 For samples collected to determine compliance with total trihalomethane regulations (40 CFR Part 141.30), add 2.5 to 3 mg reducing agent per 40 mL to the empty sample bottles and blanks just prior to shipping to the sampling site.

1.3 Collect all samples in duplicate (triplicate when high levels requiring screening and dilution are suspected). Fill sample bottles to overflowing. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed.

1.4 When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 10 minutes). Adjust the flow to above 500 mL/minute and collect duplicate samples from the flowing stream.

1.5 When sampling from an open body of water, fill a 1-qt wide-mouth bottle or 1-L beaker with sample from a representative area, and carefully fill duplicate sample bottles from the 1-qt container.

2. Sample Preservation

2.1 The samples must be chilled to 4°C on the day of collection and maintained at that temperature until analysis. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be at 4°C on arrival at the laboratory.

2.2 Sample Storage

2.2.1 Store samples and field reagent blanks together at 4°C until analysis. The sample storage area must be free of organic solvent vapors.

2.2.2 Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and replaced.

CALIBRATION AND STANDARDIZATION

1. Calibration

Prepare calibration standards by carefully adding 20.0 to 50.0 µL of secondary dilution standards to organic-free reagent water. Rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as fast as possible after injection. Mix aqueous standards by inverting the flask three times only. Discard the contents contained in the neck of the flask. Fill the sample syringe from the standard solution in the expanded area of the flask. Aqueous standards are not stable and should be discarded after 1 hour.

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1.2 Aqueous calibration standards are prepared at the following concentrations:

0.5 µg/L - 20 µL of secondary dilution standard into 200 mL of reagent water.

2.0 µg/L - 20 µL of secondary dilution standard into 50 mL of reagent water.

5.0 µg/L - 25 µL of secondary dilution standard into 25 mL of reagent water.

20 µg/L - 20 µL of secondary dilution standard into 5 mL of reagent water.

50 µg/L - 50 µL of secondary dilution standard into 5 mL of reagent water.

1.3 Starting with the standard of lowest concentration, analyze each calibration standard and tabulate peak height or area response versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Check the validity of the calibration curve daily by analyzing a mid- and low-range calibration check standards.

1.4 All of the peaks contained in the standard chromatograms must be sharp and symmetrical. Peak tailing significantly in excess of that shown in the method chromatogram (Figures 1 and 2) must be corrected. If only the peaks eluting before chloroform or ethylbenzene (Hall/PID) give random responses or unusually wide peak widths, are poorly resolved, or are missing, the problem is usually traceable to the trap/desorber. If only brominated compounds show poor peak geometry or do not properly respond at low concentrations, replace the trap. Excessive detector reactor temperatures can also cause low bromoform response. If negative peaks appear in the chromatogram, replace the ion exchange column and replace the electrolyte in the detector.

PROCEDURE

1. Initial Conditions. Adjust purge gas (helium) to 40 mL/minute. Establish GC conditions equivalent to those presented in Section 3 of "Equipment and Apparatus."

2. Trap Conditions:

Purge 10 minutes at ≤30°C
Desorb 4 minutes at 180°C
Bake 10 minutes at 220°C

3. Before processing any samples, an organic free water blank (see Reagent Water) is analyzed to check the analytical system for interferences. Two calibration check standards are then analyzed.

   3.1 One standard at 20 µg/L (20 µL of secondary dilution standard into 5 mg reagent water) is used to check the validity of the calibration curve. All target compounds must fall within ±20% of the true value. If any compound fails this criteria, analyze a duplicate 20 µg/L calibration check. If the value(s) is still off by more than ±20%, then prepare a new calibration curve.

   3.2 The second check standard at 0.5 µg/L (20 µL of secondary dilution standard into 200 mL reagent water) is analyzed and the results used to document the sensitivity of the system for the target compounds.

4. At least 10% or one sample per set should be spiked (10 µL spiking solution into the 5 mL sample syringe) and analyzed in duplicate.

   4.1 If the recovery for a particular parameter does not fall within ±10% of the expected value (10 µg/L), the results reported for that parameter in all samples processed as part of the same set must be labeled as suspect.

   4.2 Check the precision between replicate analyses. A properly operating system should perform with an average relative standard deviation of less than 10%. Poor precision is generally traceable to pneumatic leaks, especially around the sample purger and detector reactor inlet and exit (Hall 700A), to an improperly adjusted lamp intensity power (PID), electronic problems, or sampling and storage problems.

5. <u>Sample Introduction</u>. Remove the plungers from two 5-mL syringes and attach a closed syringe valve to each. Warm the sample to room temperature, open the sample (or standard) bottle, and carefully pour the sample into one of the syringe barrels to just short of overflowing. Replace the syringe plunger, invert the syringe, and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Add 5 µl of the internal surrogate standard to the sample through the syringe valve. Close the valve. Fill the second syringe in an identical manner from the same sample bottle. Reserve this second syringe for a reanalysis if necessary.

   Attach the sample syringe valve to the syringe valve on the purging device. Be sure that the trap is cooler than 30°C, then open the sample syringe valve and inject the sample into the purging chamber. Close both valves and initiate purging. Purge the sample for 11.0 minutes ±0.1 minute at ambient temperature.

6. Start sample desorb and GC temperature program, and drain purge device, rinse three times with organic free water.

CALCULATIONS:

1. Identify each compound in the sample chromatogram by comparing the retention time of the suspect peak to retention times generated by the calibration standards.

2. Determine the concentration of the unknowns by using split calibration curves or by comparing the peak height or area of the unknowns to the peak height or area of the standards as follows.

$$\text{Concentration of unknown } (\mu g/L) = \frac{\text{Peak height sample}}{\text{Peak height standard}} \times \text{Concentration of standard } (\mu g/L)$$

3. Report the results for the unknown samples in µg/L. Round off the results to the nearest 0.1 µg/L for values less than 20 µg/L. Two significant figures for values greater than 20 µg/L.

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TABLE 1. Retention times and minimum detection limits for purgeable halocarbons. (Hall 700A)

Compound	Retention time (min.)	Detection limit * (µg/l)
Chloromethane	3.13	1.00 #
Vinyl Chloride	3.13	1.00 #
Chloroethane	3.55	1.00 #
Bromomethane	3.55	1.00 #
Trichloroflouromethane	3.71	0.50 #
1,1-Dichloroethene	4.32	0.36
Dichloromethane	4.91	1.29
trans-1,2-Dichloroethene	5.25	0.28
1,1-Dichloroethane	6.02	0.28
cis-1,2-Dichloroethene	7.33	0.57
Chloroform	7.50	0.67
Bromochloromethane (I.S.)	8.00	--
1,1,1-Trichloroethane	8.74	0.16
Carbon tetrachloride	9.38	0.41
1,2-Dichloroethane	10.17	0.28
Trichloroethene	11.91	0.54
1,2-Dichloropropane	12.62	0.29
Bromodichloromethane	13.41	0.16
2-Chloroethylvinyl ether	14.88	0.86
cis-1,3-Dichloropropene	15.36	0.31
trans-1,3-dichloropropene	17.16	0.14
1,1,2-Trichloroethane	17.56	0.39
Tetrachloroethene	18.12	0.28
Dibromochloromethane	19.01	0.20
Chlorobenzene	20.96	0.69
Bromoform	24.06	1.26
1,1,2,2-Tetrachloroethane	25.00	0.50
1,3-Dichlorobenzene	28.46	0.99
1,4-Dichlorobenzene	28.88	0.99
1,2-Dichlorobenzene	30.02	0.99

Column conditions: Supelco VOCOL coated 60m x .75mm wide bore capillary column. Helium carrier rate at 15mL/min. Helium makeup gas at 15mL/min. Column temperature held at 30°C for 8 min. then programmed at 4°C/min. to 190°C.

* Detection limits based on 5 mL purge volume.
# Estimated detection limit.

TABLE 2. Average recovery and standard deviation for 20µg/l calibration checks, purgeable halocarbons. (Hall 700A)

Compound	Average recovery (µg/l)	Standard deviation (µg/l)
Chloromethane	N.D.	N.D.
Vinyl Chloride	19.1	2.4
Chloroethane	N.D.	N.D.
Bromomethane	N.D.	N.D.
Trichloroflouromethane	N.D.	N.D.
1,1-Dichloroethene	20.9	2.5
Dichloromethane	20.7	2.7
trans-1,2-Dichloroethene	20.5	2.6
1,1-Dichloroethane	19.9	2.3
cis-1,2-Dichloroethene	18.6	1.5
Chloroform	18.6	1.8
Bromochloromethane (I.S.) *	11.1	0.9
1,1,1-Trichloroethane	19.9	1.8
Carbon tetrachloride	18.9	2.3
1,2-Dichloroethane	21.6	2.9
Trichloroethene	20.4	2.8
1,2-Dichloropropane	19.9	2.2
Bromodichloromethane	19.3	1.9
2-Chloroethylvinyl ether	21.6	1.2
cis-1,3-Dichloropropene	19.1	1.4
trans-1,3-Dichloropropene	19.4	1.3
1,1,2-Trichloroethane	18.0	1.3
Tetrachloroethene	19.9	2.2
Dibromochloromethane	19.2	1.4
Chlorobenzene	20.2	2.0
Bromoform	17.8	2.4
1,1,2,2-Tetrachloroethane	20.0	1.5
1,3-Dichlorobenzene	15.7	1.9
1,4-Dichlorobenzene	11.4	0.8
1,2-Dichlorobenzene	10.7	1.3

* Internal Standard at 10µg/l

N.D. Not Determined, performance criteria will be determined when sufficient data has been collected.

TABLE 3. Retention times and estimated minimum detection limits for purgeable aromatics. (PID 10.2eV)

Compound	Retention time (min.)	Detection limit (µg/l)
Benzene	10.42	1.00
Toluene	16.50	0.50
Chlorobenzene	21.30	**
Ethylbenzene	21.58	1.00
m+p-Xylene	21.83	1.00
o-Xylene	23.22	1.00
Styrene	23.43	3.00
1,3-Dichlorobenzene	28.80	**
1,4-Dichlorobenzene	29.22	**
1,2-Dichlorobenzene	30.37	**

Column conditions: Supelco VOCOL coated 60m x .75mm wide bore capillary column. Helium carrier rate at 15mL/min. Helium makeup gas at 15mL/min. Column temperature held at 30°C for 8 min. then programmed at 4°C/min. to 190°C.

** Data determined on Hall 700A.

TABLE 4. Average recovery and standard deviation for 20µg/l calibration checks, purgeable aromatics. (PID 10.2eV)

Compound	Average recovery (µg/l)	Standard deviation (µg/l)
Benzene	16.56	3.37
Toluene	17.64	2.98
Chlorobenzene	**	**
Ethylbenzene	19.16	4.85
m+p-Xylene	N.D.	N.D.
o-Xylene	21.79	2.32
Styrene	N.D.	N.D.
1,3-Dichlorobenzene	**	**
1,4-Dichlorobenzene	**	**
1,2-Dichlorobenzene	**	**

** Recovery data determined on Hall 700A.
N.D. Not Determined, performance criteria will be determined when sufficient data has been collected.

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TABLE 5. Listing of Compounds Found in Matrix Spiking Solutions, Supelco Purgeables A and Supelco Purgeables B.

Purgeables A

Methylene chloride
Trichloroflouromethane
1,1-Dichloroethylene
1,1-Dichloroethane
Chloroform
1,2-Dichloropropane
Trichloroethylene
1,1,2-Trichloroethane
Dibromochloromethane
2-Chloroethyl vinyl ether
Tetrachloroethylene
Chlorobenzene

Purgeables B

Trans-1,2-Dichloroethylene
1,2-Dichloroethane
1,1,1-Trichloroethane
Bromodichloromethane
Trans-1,3-Dichloropropene
Cis-1,3-Dichloropropene
Benzene
Bromoform
1,1,2,2-Tetrachloroethane
Toluene
Ethyl Benzene

FIGURE 1. Standard chromatogram, purgeable halocarbons, HALL 700A. ATTACHMENT
Analytes at 2ug/L.

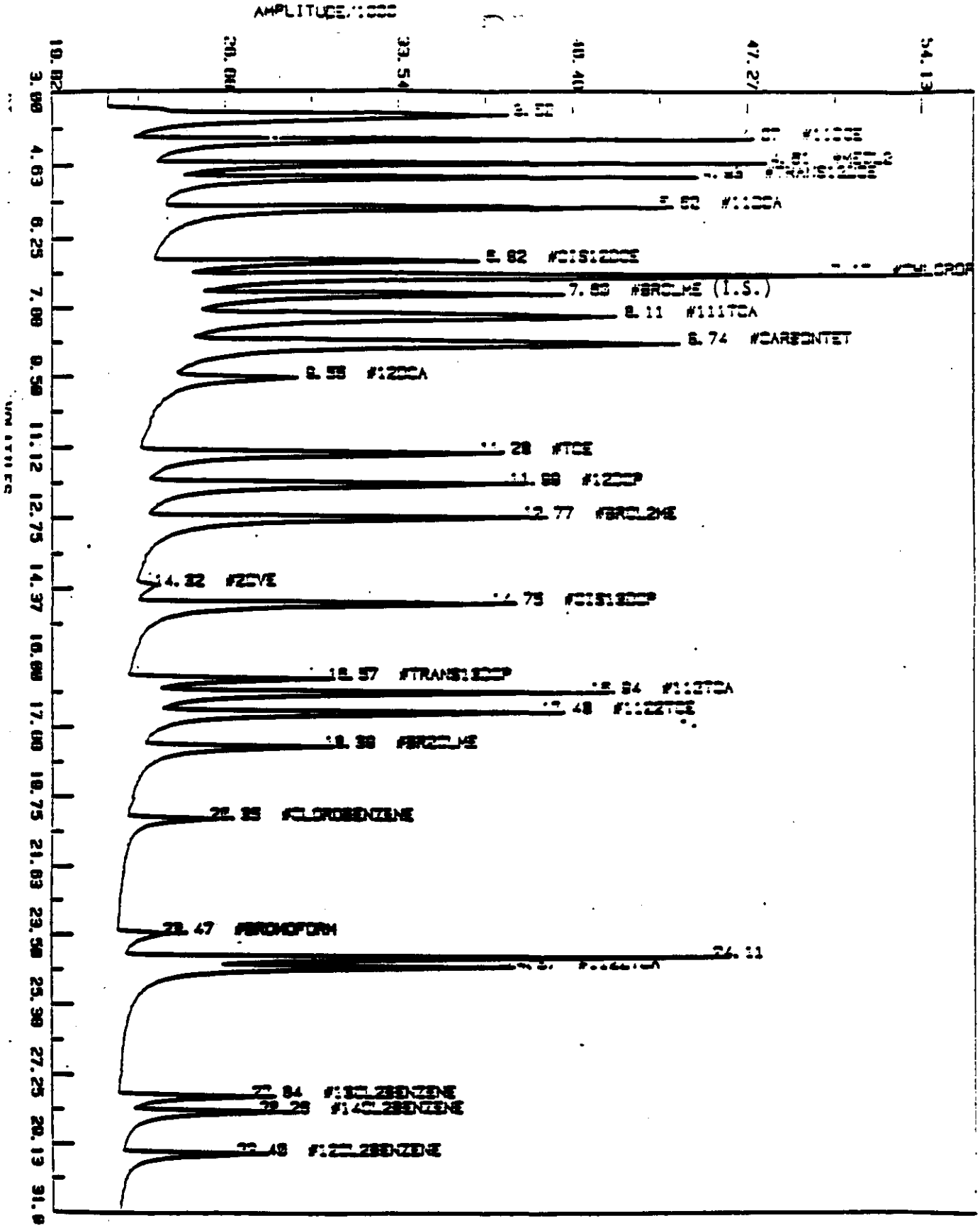
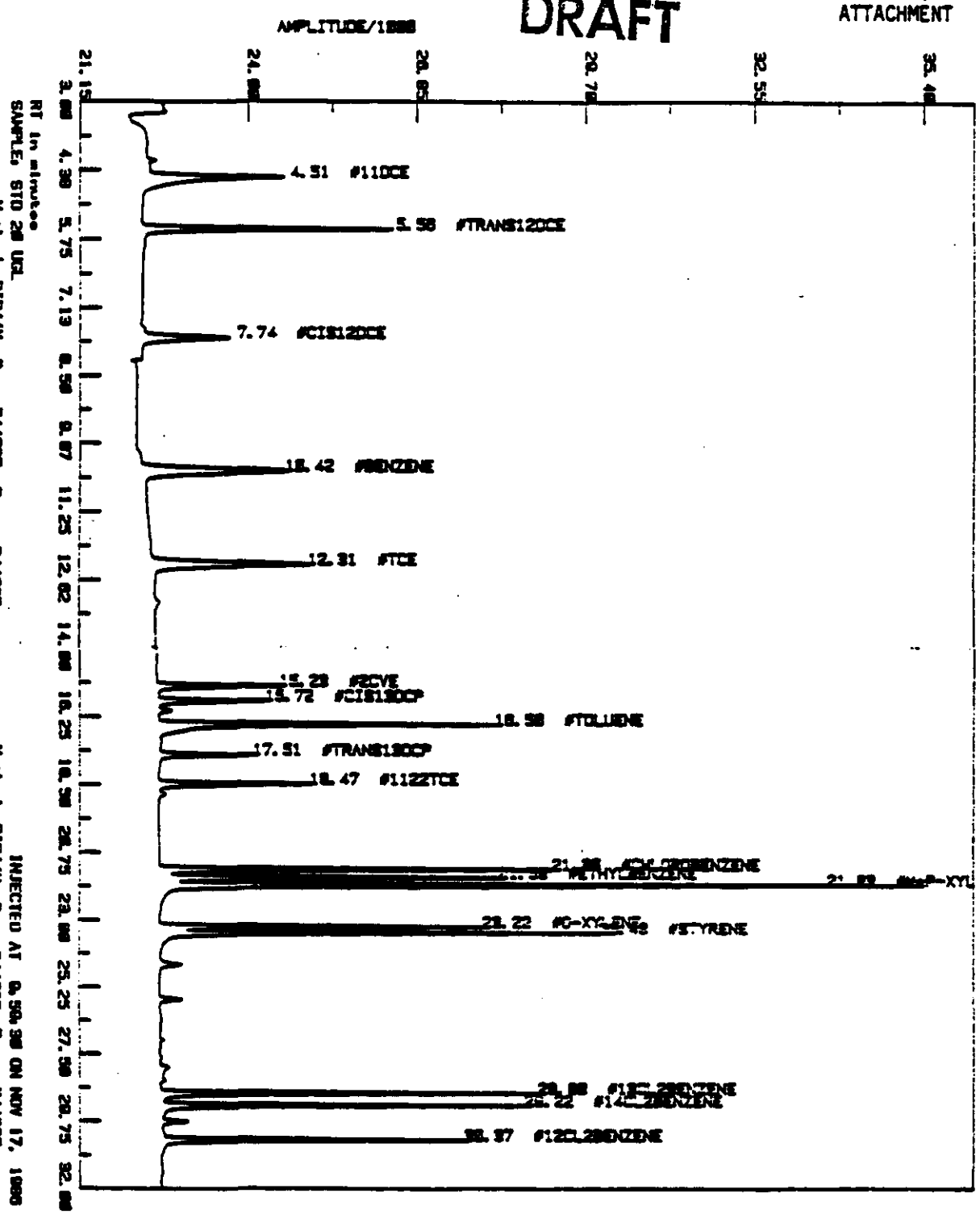


FIGURE 2. Standard chromatogram, purgeable aromatics, PID (10.2 eV). Analytes at 20ug/L.

DRAFT

MP-P601,P602-MA
ATTACHMENT



APPENDIX E

FIELD MEASUREMENT OF PERMEABILITY USING THE BAIL-DOWN TEST

WARZYN

FIELD MEASUREMENT OF PERMEABILITY
BAIL-DOWN TEST

INTRODUCTION

The objective is to determine hydraulic parameters (transmissivity, storativity, hydraulic conductivity) of the water-bearing strata. Single well aquifer tests are used because they may be conducted using a minimum of equipment, personnel, and time. They may also be done at many points within an aquifer and may be used to better plan a full-scale pumping test.

PROCEDURES

Each bail-down test is conducted by measuring the static water level with an electric water level indicator or cloth tape with attached sounding device, placing a pressure transducer (connected to a Hermit data logger[1]), below the water level, and removing one bailer volume from the well. The recovery of the water level back to the static level is recorded over the necessary period of time, using a logarithmic sampling mode on the data logger.

The electric water level tape and transducer are wiped, first with methanol and then with deionized water as they are placed in the wells, to prevent cross-contamination between wells. The bailer is lowered with an attached 1/8-inch stainless steel cable. The bailer and cable are decontaminated between uses by washing and rinsing with Liquinox soap and water, rinsing three times with methanol, and rinsing three times with deionized water. The bailer and cable are then allowed to air-dry on steel supports and are covered with new sheet plastic.

DATA REDUCTION

The data accumulated during the bail-down tests are used to calculate hydraulic parameters using several published methods. Hydraulic conductivity is calculated for shallow, unconfined wells using the Bouwer and Rice method (1976). The NAVFAC method (1971) is used for comparison. Transmissivities and storativities for the deeper, confined wells are determined using the curve matching method described by Cooper, et al. (1967). For comparison, hydraulic conductivities are also calculated using the Hvorslev method (1951). All methods assume an infinite, homogenous, isotropic aquifer and an instantaneous change in head in the well.

The method described by Bouwer and Rice (1976) is based upon modifications to the Thiem equation, with the use of an analog model. A straight line is drawn through a semi-log plot of relative head versus time, and the hydraulic conductivity is calculated using the slope of that line and the geometry of the well and aquifer. The formulation assumes that draw-down of the water table around the well is negligible, that flow in the

---

1 Hermit Environmental Data Logger, Model SE1000B, In-Situ, Inc., Laramie, Wyoming 82070.

capillary fringe may be ignored, and that well losses are negligible. It is applicable to completely or partially penetrating wells in unconfined aquifers, but may be used for confined aquifers that receive water from the upper confining layer.

In the NAVFAC method (1971), a straight line is also drawn through a semi-log plot of recovery data for unconfined aquifers. The method is based on the Hvorslev method. It assumes that the well is cased below the water table, and the ratio of the screen length to the well radius (L/R) is greater than eight.

The Cooper, et al. (1967) formulation calculates the transmissivity of an aquifer by matching a plot of relative head (linear sclae) versus time (logarithmic scale) to one of a set of type curves. The method assumes that the change in head after a known volume of water is injected or removed is instantaneous and that the (non-flowing) well is screened over the entire thickness of an artesian aquifer. It is directly applicable to fully penetrating screened wells in confined aquifers, but may be used to determine the transmissivity of the portion of an aquifer over which a partially penetrating well is screened, assuming no vertical flow occurs.

The Hvorslev method (1951) is based on a solution of the LaPlace equation and does not account for aquifer storage. A straight line is drawn through a semi-log plot of relative head versus time. The time that would be required for complete equalization of head difference if the original rate of inflow were maintained (defined as the basic time lag, T_0, and equal to the time when H-h/H-Ho = 0.37) is used to calculate the hydraulic conductivity. The value of T_0 is measured graphically, and the ratio of the piezometer length to radius is assumed to be greater than eight (L/R>8).

REFERENCES

Bouwer, H. and Rice, R.C., 1976. "A Slug Test for Determining Hydraulic Conductivity of Unconfined Aquifers with Completely or Partially Penetrating Wells", Water Resources Research, Vol. 12, No. 3, pp. 423-428.

Cooper, H.H., Bredehoeft, J.D. and Papadopulos, I.S., 1967. "Response of a Finite-Diameter Well to an Instantaneous Charge of Water", Water Resources Research, Vol, 3, No. 1, pp. 263-269.

Hvorslev, M.J., 1951. "Time Lag and Soil Permeability in Groundwater Observations", U.S. Army Corps of Engineers Waterways Exp. Sta. Bull. 36, Vicksburg, MA.

NAVFAC, DM-7, 1971. Design Manual - "Soil Mechanics, Foundations and Earth Structures", Department of the Navy, Naval Facilities Engineering Command.

[jam-13-6]

F

APPENDIX F

Example Sample Documentation Forms

SAMPLE IDENTIFICATION RECORD

CASE/JOB NUMBER	MATRIX	SAMPLE NUMBER	LAB	LAB NUMBER	CHAIN OF CUSTODY	DATE SAMPLED	DATE SHIPPED	AIRBILL NUMBER	SAMPLE TAG NUMBER

University Research Lab
P.O. Box 5385
Madison, Wisconsin 53705
(608) 273-0440

CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME
SAMPLERS: *(Signature)*	

LAB NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO OF CONTAINERS							REMARKS

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*		Date / Time	

Remarks

Distribution: White — Accompanies Shipment; Yellow — Laboratory File; Pink — Coordinator Field Files

NO 6 0806

Project Number	Month/Day/Year	Time	Designate	
			Comp.	Grab

Tag No.	Sample Location	Samplers (Signatures)
[illegible]		

Lab Sample No.	Remarks:																			

Preservative:
HNO_3 H_2SO_4 NaOH None
Other _____
Filtered Unfiltered

ANALYSES

BOD Anions	
Solids (TSS) (TDS) (SS)	
COD, TOC, Nutrients	
Phenolics	
Mercury	
Metals	
Cyanide	
Oil and Grease	
Organics GC/MS	
Semi Volatiles	
Volatile Organics	
Pesticides / PCB's	

WARZYN

Warzyn Engineering Inc.
One Science Court
University Research Park
P.O. Box 5385
Madison, Wisconsin 53705

WARZYN

№ 1230

CHAIN OF CUSTODY SEAL

WARZYN ENGINEERING INC.
ONE SCIENCE COURT
UNIVERSITY RESEARCH PARK
P.O. BOX 5385
MADISON, WI 53705
(608) 273-0440

G

APPDENDIX G

DOCUMENT CONTROL AND THE EVIDENTIARY FILE SYSTEM FOR PAGEL'S PIT LANDFILL RI/FS

APPENDIX G
DOCUMENT CONTROL AND THE EVIDENTIARY FILE SYSTEM FOR PAGEL'S PIT LANDFILL RI/FS

ACCOUNTABLE DOCUMENTS

Accountable documents will include all logbooks, field data records, correspondence, sample tags, graphs, chain-of-custody records, and other sample documentation forms used, original data including laboratory bench sheets, photographic prints and planning documents.

FILE STRUCTURE

Documents will be arranged in the evidentiary file using the format specified in Table 1 (attached).

LOGGING OF DOCUMENTS

Documents will be received by the Document Control Officer who will log them and assign a number to each such that documents within each document subclass are separately serialized. An exception to this will be sample tags, chain-of-custody forms or other documents that are numbered prior to assignment for use.

DOCUMENT ACCESS

Project documents will be secured in a separate, locked file cabinet. Access will be limited by the Document Control Officer to project personnel. A check-out log will be maintained as a record of access.

EVIDENCE FILE AUDIT

Upon project completion, the Warzyn Quality Assurance Officer will audit the evidence file for completeness. Results of the audit will be documented on the attached form and kept in the Final Evidence file.

FINAL DISPOSITION OF FILE CONTENTS

The Final Evidence file will be maintained by Warzyn as described in Section XX., Record Preservation, of the Consent Order. The file will be maintained during the pendancy of the Consent Order and for a minimum of four years after its termination.

TABLE 1

DOCUMENT CLASSES AND STRUCTURE
FOR SUPERFUND EVIDENTIARY FILES

Document Classes	File	Contents
File Index		Check out logs and list of active files
Contracts/ Proposals/Bids	A	Proposals, contracts, purchase orders, specifications-COPIES ONLY
Financial	B	Summary of invoice status; invoices correspondence re: accounts receivable; copy of budget and project task setup; COPIES ONLY
Correspondence	C	Various incoming and outgoing letters, memorandums, diary notes
		C1-In-house correspondence; diary notes and memos
		C2-Outgoing letters/memos
		C3-Incoming correspondence
		C4-Correspondence logged
Work Plan Documents	D	Documents other than proposals; including Health and Safety plans, Sampling plans, QAPPs, permit plans; special instructions/outlines for conducting the project; Work Plans; WEI plans and specifications

QA File	E	QA Work Plan and budget; project history file; sample documentation records, etc.
		E1-Sample tags
		E2-Chain-of-Custody Records
		E3-Receipt of Samples forms
		E4-Transfer of Samples forms
Field Data	F	Original field data/notebooks
		F1-Field boring logs
		F2-Well construction details
		F3-Geotechnical testing
		F4-Geophysical testing
		F5-Water Quality testing
		F6-Daily field logs
		F7-Baildown testing
		F8-Structural testing
		F9-Miscellaneous/Other
Laboratory Data	G	Laboratory test data, including original analytical logbooks, lab data, calculations, bench records, graphs, etc. for original data and quality control data
		G1-Analytical laboratory data
		G2-Geotechnical laboratory data
		G3-Materials testing laboratory data
		G4-Subcontracted laboratory data

Calculations	H	Calculations, quantity estimates, computer printouts of tabularized data
Photographs	I	Photographs, stereo pairs, site maps (published zoning, topography, geology, groundwater, bedrock, negatives)
Originals	J	Warzyn original reports or drafts
Warzyn Reports	K	Copies of the project report or previous pertinent WEI reports
Warzyn Drawings	L	Reference list of report drawings; copies or reduced copies of original drawings. Note: original or reduced mylars will be stored separately
Other Reports/ Drawings	M	Non-Warzyn reports and drawings including literature, references, etc.
Miscellaneous	N	Other file information which does not fit into other categories; file must be named
Checkprint	O	TEMPORARY FILE of checkprints, draft reports or other work in progress. File must be removed upon job completion.

12660.00
RCW/sss/JAH
[sss-400-82]

DOCUMENT AUDIT CHECKLIST*

PROJECT NO. _____ DATE OF AUDIT _____

PROJECT LOCATION _____ SIGNATURE OF AUDITOR _____

FILE LOCATION _____

Yes__ No__ 1. Have individual files been assembled (field investigation, laboratory, other)?

Comments: _____

_____

_____

Yes__ No__ 2. Is each file inventoried?

Comments: _____

_____

_____

Yes__ No__ 3. Is there a list of accountable documents?

Comments: _____

_____

_____

Yes__ No__ 4. Are all accountable documents present or accounted for?

Comments: _____

_____

_____

* From NEIC Procedure Manual for the Evidence Audit of Enforcement Investigations by Contractor Evidence Audit Teams, EPA-300/9-81-003-R, April, 1984.

Yes__ No__ 5. Is a document numbering system used?

Comments:_____

_____

_____

Yes__ No__ 6. Has each document been assigned a document control number?

Comments:_____

_____

_____

Yes__ No__ 7. Are all documents listed on the inventory accounted for?

Comments:_____

_____

_____

Yes__ No__ 8. Are there any documents in the file which are not on the inventory?

Comments:_____

_____

_____

Yes__ No__ 9. Is the file stored in a secure area?

Comments:_____

_____

_____

Yes__ No__ 10. Are there any project documents which have been declared confidential?

Comments:_____

_____

_____

Yes__ No__ 11. Are confidential documents stored in a secure area separate from other project documents?

Comments:_____

_____

_____

Yes__ No__ 12. Is access to confidential files restricted?

Comments:_____

_____

_____

Yes__ No__ 13. Have confidential documents been marked or stamped "Confidential"?

Comments:_____

_____

_____

Yes__ No__ 14. Is confidential information inventoried?

Comments:_____

_____

_____

Yes__ No__ 15. Is confidential information numbered for document control?

Comments:_____

_____

_____

Yes__ No__ 16. Have any documents been claimed confidential under TSCA?

Comments:_____

_____

_____

[jap-800-64a]

H

APPENDIX H

ACKNOWLEDGEMENT OF ABILITY TO PERFORM ANALYSES BY PERFORMING LABORATORIES

HAZLETON LABORATORIES AMERICA, INC.
3301 KINSMAN BLVD. • P.O. BOX 7545 • MADISON, WI 53707 • (608) 241-4471 • TLX 703956 HAZRAL MDS UD

Mr. Robert Wendt
Warzyn Engineering Inc.
One Science Court
University Research Park
Madison, Wisconsin 53705

December 30, 1987

RE: Remedial Investigation and Feasibility Study, Pagel's Pit Landfill, Winnnebago County, Illinois.

Dear Mr. Wendt:

Hazleton Laboratories America, Inc. Madison, Wisconsin has reviewed the Pagel's Pit QAPP (Revision No. 1 of 8/87). We also understand that an additional revision with minor changes is currently being prepared.

We fully understand the project requisites and are prepared to commit the required resources for the timely completion of this important environmental work. We understand that the Round 1 sampling schedule is projected to start at mid to late January of 1988. We will be able to meet your needs as long as this anticipated schedule is adhered to.

Please feel free to contact me should you require additional information.

We look forward to being of continued service to Warzyn Engineering, Inc.

Sincerely,

David C. Hills
Manager, Environmental Analyses

CC:
C. Altstaetter
File

RADIAN
CORPORATION

August 10, 1987

Mr. Bob Wendt
Warzyn Engineering
1 Science Court
University Research Park
P.O. Box 5385
Madison, WI 53705

RECEIVED
WARZYN ENGINEERING INC
MADISON

Dear Bob:

I am sending this letter to acknowledge your request for Radian to perform HSL volatile analyses of ten(10) pairs of sorbent tubes according to the methodology described in our Quality Assurance Plan dated June, 1987.

We understand that you will require the analyses in September and are fully-prepared to respond to your request. Please let me know if I can provide any further information.

Sincerely,

James B. Homolya
Program Manager

cc: D. Wagoner, Radian

Progress Center/3200 E. Chapel Hill Rd./Nelson Hwy./P.O. Box 13000/Research Triangle Park, N.C. 27709/(919)541-9100

APPENDIX I

INTERNAL CHAIN OF CUSTODY PROCEDURES FOR HAZLETON LABORATORIES AMERICA

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America, Inc.
Quality Assurance Unit

DEPARTMENT OPERATING PROCEDURE

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AMERICA, INC.
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DATE: 2/11/87
REPLACES: 4/21/86

PROCEDURE IDENTIFICATION: EPA WA-82-A155 Hazardous Waste Analysis

PROCEDURE TITLE: Chain of Custody Procedures

AREA OF APPLICABILITY: Hazleton Laboratories America, Inc.
Environmental Analysis

PURPOSE:

Future legal proceedings may necessitate Hazleton Laboratories America, Inc. (HLA) to produce documentation that traces the custody of samples from receipt through completion of analysis. In lieu of this, it is necessary for HLA to guarantee the continuous custody of samples by either the sample custodian or the lab analyst.

PROCEDURE:

The following procedures for documentation of chain of custody for samples received under Contract WA-82-A155 will be followed in addition to or as an addendum to those previously stated under OP-DIV 7 and OP-PROJ 2.

1. All samples will be stored locked in refrigerators located on the third and fourth floors. A list of sample numbers, arranged by EPA case number, will be maintained by the sample custodian.

   1.1 All bottles received for extractable compounds (semivolatiles or pesticides) will be stored at 4°C in one of two double-door refrigerators located on 3 South in Room 301.

   1.2 All bottles received for volatile analysis will be stored at 4°C on 4 South in the Mass Spectrometry area, in the refrigerator designated for volatile samples only.

2. Only the designated sample custodian and supervisory personnel will have keys to the double-door refrigerators.

3. Samples will remain in the designated refrigerators until removed for sample preparation and/or analysis.

4. All transfers of samples into or out of the double door refrigerators will be documented on an internal chain of custody record (see attached). These records are maintained by the sample custodian.

5. Once a sample is removed from a refrigerator by the analyst, he/she is responsible for the custody of the sample. Each analyst must return samples to the double door refrigerator before the end of the working day. Samples are not allowed to sit on the bench over the evening.

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DATE: 2/11/87
REPLACES: 4/21/86

APPROVED BY: Mark A. Wirtz — DATE 2-11-87
Mark Wirtz
Environmental Analysis

David C. Hills — DATE 2-11-87
David Hills
Project Leader

REVIEWED BY: Debra Curley Arndt — DATE 2/11/87
Debra Curley Arndt
Manager, Quality Assurance Unit

(1458E)

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OP-PROJ. 1
ATTACHMENT

HAZLETON LABORATORIES
AMERICA, INC.

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EPA WA-82-A155 HAZARDOUS WASTE ANALYSIS

Chain of Custody

Case No. _____

Storage Location _____
Storage Date _____

Laboratory Sample No.	Removed by	Date/Time Removed	Reason	Date/Time Returned

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OP-PROJ. 1
ATTACHMENT

PROJECT SPECIFIC DOCUMENTATION

PROJECT IDENTIFICATION: EPA WA-82-A155 Hazardous Waste Analysis

PROCEDURE TITLE: Chain of Custody Procedures

DISTRIBUTION:

Name	Title	Department/Cost Center No.
David C. Hills	Section Supervisor	Environmental Analysis/6004
Mark Wirtz	Documentation Custodian/ Sample Custodian	Environmental Analysis/6004

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PROCEDURE IDENTIFICATION: EPA WA-82-A155 Hazardous Waste Analysis

PROCEDURE TITLE: Sample Receipt and Sample Logging Procedures

AREA OF APPLICABILITY: Hazleton Laboratories America, Inc.
Environmental Analysis

DISTRIBUTION:

See attached list.

PURPOSE:

These procedures are to be used in conjunction with the aforementioned project because of the nature of the samples and the special chain of custody procedures required.

SAFETY PRECAUTIONS:

All samples received under this contract should be considered hazardous and appropriate precautions should be taken when handling these samples. Upon initial receipt at Hazleton Laboratories America, Inc. (HLA) facilities, sample coolers should be inspected for any damage or leakage. Under no circumstances should any personnel other than the sample custodian or project leader open coolers. If damage or leakage is noted, stay clear of the coolers and notify the sample custodian or project leader immediately.

PROCEDURE:

1. The project leader will notify the sample custodian in writing of incoming samples.

2. When samples arrive at HLA, the shipping and receiving clerk or the receptionist will notify the sample custodian. The custodian will collect the samples and deliver them to a hood located on 3 South. The following procedures will be done by the sample custodian.

3. Examine the shipping container and record the following information in the project log book (one container per form).

   3.1 The presence/absence of custody seal on the shipping container.

   3.2 The condition of the custody seal (i.e., intact, broken).

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4. Open the shipping container in a well ventilated hood, remove the enclosed sample documents, and record the following information in the log book.

   4.1 The presence/absence of the chain of custody record(s).

   4.2 The presence/absence of Sample Management Office (SMO) forms (Traffic Reports, Chronicles).

   4.3 The presence/absence of airbills and/or bills of lading documenting shipment of the samples.

5. Remove the samples from the container and record the following information in the log book.

   5.1 Condition of samples (intact, broken, leaking, etc). Any broken and/or leaking samples should be carefully repacked, labeled as a Biohazard, and returned to the sponsor. The project leader should be notified and will contact the EPA project officer that the sample was received broken and returned.

   5.2 The presence/absence of sample tags.

   5.3 Sample tag numbers.

6. Compare the following documents to verify agreement of the information contained on them.

   6.1 Chain of custody records.

   6.2 Sample tags.

   6.3 SMO forms.

   6.4 Airbills or bills of lading.

   Document agreement and/or any discrepancies found. If discrepancies are found, contact the SMO for clarification and notify the appropriate laboratory personnel.

7. If there are no problems with the sample shipment, sign the chain of custody record in the "Received for lababoratory by:" box on the document. If problems are noted, sign for shipment and note problems in the "Remarks" box or reference other forms detailing the problems.

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8. Log-in the Samples.

   8.1 Figure 1 is an example of a type of form the sample custodian could use in order to log-in samples and to record the information previously described.

9. Send preprocessing request form to Sample Entry requesting that HLA numbers be assigned to the samples, and that they be logged onto the LIMS under the appropriate mnemonic for tracking and billing.

   9.1 The following LIMS mnemonics will be used for this contract:

   HGMS Hazardous Waste GCMS Analysis
   EPAP - Priority Pool Pesticides

   9.2 Upon receipt of sample HLA numbers, log these HLA numbers into the book.

10. Sample Storage.

   10.1 All bottles received for extractable compounds (semivolatiles or pesticides) will be stored at 4°C in one of two double-door refrigerators located on 3 South in Room 301.

   10.2 All bottles received for volatile analysis will be stored at 4°C on 4 South in the Mass Spectrometry area, in the refrigerator designated for volatile samples only. At the time of storage a storage blank will be prepared identified with the case number and date prepared. This blank will be analyzed with those samples stored on the designated date to monitor the potential for cross-contamination of samples during storage.

APPROVED BY: Mark A. Wirtz — DATE 2-11-87
Mark Wirtz
Environmental Analysis

David C. Hills — DATE 2-11-87
David Hills
Project Leader

REVIEWED BY: Debra Curley Arndt — DATE 2/11/87
Debra Curley Arndt
Manager, Quality Assurance Unit

(1459E)

DATE: _____

OP-PROJ. 2
ATTACHMENT

[illegible] CUSTODIAN SIGNATURE: _____

DOCUMENT CONTROL

[illegible] THE APPROPRIATE RESPONSE

Custody Seal	present/absent intact/not intact
Chain-of-Custody	present/absent
Sample Tags	present/absent
Sample Tag Numbers	listed/not listed on chain-of-custody
SMO Forms	present/absent

CASE NUMBER _____

AIRBILL NUMBER _____

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[illegible]TE [illegible]IVED	TIME RECEIVED	CHAIN-OF-CUSTODY RECORD NUMBER	SMO SAMPLE NUMBERS	CORRESPONDING		DOES INFORMATION ON CUSTODY RECORDS, TRAFFIC REPORTS, AND SAMPLE TAGS AGREE?	REMARKS CONDITION OF SAMPLE SHIPMENT
				SAMPLE TAG NUMBERS	ASSIGNED ~~LAB~~ VFT NUMBERS		

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OP-PROJ. 2
ATTACHMENT

PROJECT SPECIFIC SOP DISTRIBUTION

PROJECT IDENTIFICATION: EPA WA-82-A155 Hazardous Waste Analysis

PROCEDURE TITLE: Sample Receipt and Sample Logging Procedures

DISTRIBUTION:

Name	Title	Department/Cost Center No.
David C. Hills	Project Leader	Environmental Analysis/6004
William Hamilton	Manager	Facility Services/6080
Paul Jacoby	Section Supervisor	Shipping and Receiving/6075
Judy Santos	Section Supervisor	Sample Entry/6099
Diane Loram	Manager	General Services/6072 (Building Receptionist)
Mark Wirtz	Documentation Custodian/ Sample Custodian	Environmental Analysis/6004

DEPARTMENT OPERATING PROCEDURE

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PROJECT IDENTIFICATION: EPA WA-82-A155 Hazardous Waste Analysis

PROCEDURE TITLE: Duties and Responsibilities of the Documentation Custodian and Procedures for Data Assembly and Filing

AREA OF APPLICABILITY: Hazleton Laboratories America, Inc. Environmental Analysis

DISTRIBUTION:

See attached list.

PURPOSE:

To specify the duties and procedures of the documentation custodian for the above-mentioned contract.

PROCEDURE:

1. The documentation custodian will organize and assemble all documents relating to each EPA case.

2. This procedure will ensure that all documents to be submitted to EPA are compiled in one location, preferably in single case files arranged by SMO sample number.

   2.1 Prepare case file folders as follows:.

      2.1.1 Assign one folder to each case according to SMO case number.

      2.1.2 Place all documents, sample tags, SMO forms, and laboratory-generated data, pertaining to one case in the assigned folder.

      2.1.3 Arrange all documents by type within the case folders, i.e., all sample tags together, all Traffic Reports together, all hardcopy Mass Spectra together, etc.

      2.1.4 File these document case files in one location in a secure area.

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3. Document Numbering and Inventory Procedure.

3.1 Assignment of Accountable Numbers to laboratory-generated data.

3.1.1 Inventory each document of a case and assign it a serialized number (an identifier) associating it with a particular case. For example:

SMO case =	-	serialized document folder
5081		01

3.1.2 Inventory and number all documents pertaining to each case including the following:

3.1.2.1 Sample traffic records, weekly reports.

3.1.2.2 Custody records, sample tags, airbills, internal custody records.

3.1.2.3 Laboratory log books, personal log books, instrument log books, benchsheets.

3.1.2.4 Laboratory data (sorted by sample), calibration and quality control results.

3.1.2.5 Data summaries and reports.

3.1.2.6 All other documents, forms, or records referencing the samples.

3.2 Preparation of a Document Inventory.

3.2.1 Prepare a document inventory list to provide a record of all documents and their corresponding document numbers that are included in the completed case file.

3.2.2 If documents for a case are sent to an EPA Region for enforcement action or other action, the laboratory will retain a copy of the document inventory list for that case.

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APPROVED BY: Mark A. Wirtz
Mark Wirtz
Environmental Analysis
DATE 4-17-86

David C. Hills
David Hills
Project Leader
DATE 4-17-86

REVIEWED BY: Debra Curley Arndt
Debra Curley Arndt
Manager, Quality Assurance Unit
DATE 4/21/86

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OP-PROJ. 3
ATTACHMENT

DOCUMENT CHECKLIST

Document Control Number		Number of Pages
	File Inventory	
	Sample Tags	
	Sample Tracking Documents	
	EPA Chain of Custody	
	Airbills	
	Organic Traffic Reports	
	Extraction Worksheets/Screens	
	Copies of Analyst's Notebook Pages	
	Identification/Quantitation Worksheets	
	Copies of GC and GC-MS Operation Logs	
	Copies of Standard Preparation Log	
	Other Correspondence/Memos	
	QC Summary Package	
	Data Summary Package	
	Sample Data Package	
	Raw QC Data Package	
	Standard Package	
	Pesticide Raw Data	

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PROJECT IDENTIFICATION: EPA WA-82-A155 Hazardous Waste Analysis

PROCEDURE TITLE: Duties and Responsibilities of the Documentation Custodian and Procedures for Data Assembly and Filing

DISTRIBUTION:

Name	Title	Department/Cost Center No.
David Hills	Section Supervisor	Environmental Analysis/6004
Mark Wirtz	Documentation Custodian/ Sample Custodian	Environmental Analysis/6004

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DEPARTMENT OPERATING PROCEDURE

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PROJECT IDENTIFICATION: EPA WA-82-A155 Hazardous Waste Analysis

PROCEDURE TITLE: Duties and Responsibilities of the Sample Custodian

AREA OF APPLICABILITY: Hazleton Laboratories America, Inc.
Environmental Analysis

DISTRIBUTION:

See attached list.

PURPOSE:

To specify the duties of the sample custodian for the above-mentioned contract.

PROCEDURE:

Duties and responsibilities include:

1. Receiving samples (OP-PROJ 2).

2. Inspecting sample shipping containers for presence/absence and condition of:

   2.1 Custody seals, locks, "evidence tape", etc.

   2.2 Container breakage and/or container integrity.

3. Recording condition of both shipping containers and sample containers (bottles, jars, cans, etc.) in log books or on appropriate forms.

4. Signing appropriate documents shipped with samples (i.e., chain of custody record(s), SMO Traffic Reports, etc).

5. Verifying and recording agreement or non-agreement of information on sample documents (i.e., sample tags, chain of custody records, Traffic Reports, airbills, etc.) in log books or on appropriate forms. If there is non-agreement, record the problems, contact the SMO for direction, and notify appropriate laboratory personnel.

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6. Initiating sample analysis procedures on appropriate laboratory documents or according to laboratory standard operating procedures including establishing case and sample files and inventory sheets.

7. Labeling samples with HLA sample numbers.

8. Securely storing samples, sample extracts, and spent samples.

9. Controlling access to stored samples and assuring that HLA standard operating procedures are followed when samples are removed from and returned to storage (see OP-PROJ 1).

10. Assuring that sample tags are removed from sample containers and are given to the laboratory personnel for inclusion in the sample case file. Missing tags are to be accounted for by putting a written memo into the sample case file. Information from the tags should be included in the memo, if available.

11. Monitoring storage conditions for proper sample preservation such as, refrigeration temperature and prevention of cross-contamination.

12. Returning shipping containers to the proper sampling teams.

APPROVED BY: Mark S. Wirtz — DATE 4/17/86
Mark Wirtz
Environmental Analysis

David C. Hills — DATE 4-17-86
David Hills
Project Leader

REVIEWED BY: Debra Curley Arndt — DATE 4/21/86
Debra Curley Arndt
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PROJECT SPECIFIC

PROJECT IDENTIFICATION: EPA WA-82-A155 Hazardous Waste Analysis

PROCEDURE TITLE: Duties and Responsibilities of the Sample Custodian

DISTRIBUTION:

Name	Title	Department/Cost Center No.
David Hills	Section Supervisor	Environmental Analysis/6004
Mark Wirtz	Documentation Custodian/ Sample Custodian	Environmental Analysis/6004

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