Engineering & Science



# Quality Assurance Project Plan
# Operation and Maintenance
# Stoughton City Landfill
# Stoughton, Wisconsin

**April 2006**

**Prepared For:**

**Wisconsin Department of Natural Resources**
**South Central Region**
**3911 Fish Hatchery Road**
**Fitchburg, Wisconsin 53711**

**Prepared By:**

**BT$^2$, Inc.**
**2830 Dairy Drive**
**Madison, Wisconsin 53718**

**BT$^2$ Project #1764**
**Revision 1**

# TABLE OF CONTENTS

## TABLES



## FIGURES



## APPENDICES

*This page intentionally blank.*

# QUALITY ASSURANCE PROJECT PLAN

# FOR OPERATION AND MAINTENANCE AT

# STOUGHTON CITY LANDFILL

REVISION 1

April 2006

Prepared by: BT², Inc.

| | |
|---|---|
| Leslie A. Busse, BT² Project Manager | 4/5/06 Date |
| Sherren Clark, BT² Quality Assurance/ Senior Review Officer | 4-5-06 Date |
| Steven B. Smith, BT² Field Team Manager | 4/5/06 Date |
| Gary A Edelstein, WDNR Project Manager | Date |
| WDNR Quality Assurance Reviewer | Date |

*This page intentionally blank.*

# 1.0 PROJECT DESCRIPTION

## 1.1 Introduction

This section presents site background, summary of investigations, project objectives and scope, overview of sampling and analysis program, anticipated uses of the results, and project schedule.

## 1.2 Site Description

### *1.2.1 Site Location and History*

The Stoughton City Landfill (SCL) site is located in the northeast portion of the City of Stoughton, approximately 13 miles southeast of Madison, in Dane County, Wisconsin (**Figure 1**). The property containing the site encompasses approximately 27 acres and occupies portions of the west half of the southwest quarter and the southwest quarter of the northwest quarter of Section 4, Township 5 North, Range 11 East. A wetland area located along the southeast portion of the present property boundary was the initial area of waste disposal. Wetlands are also located in the north portion of the site, and west of the site along the Yahara River. The Yahara River is located west of the site and is within approximately 400 feet of the site at its closest distance. Existing site conditions are depicted on **Figure 2**.

The landfill operated from 1952 until it was officially closed in 1982. Between 1952 and 1969, the site was operated as an uncontrolled dumpsite. During this time, refuse was usually burned or covered with soil. The site began operation as a state-licensed landfill in 1969. In 1977, the Wisconsin Department of Natural Resources (WDNR) required that the site be closed according to state regulations. Closure activities included construction of a trash transfer station, placement of cover material borrowed from agricultural areas, application of topsoil, and seeding. Closure work was performed according to WDNR regulations from 1978 to 1982. Only brick, rubble, and similar construction materials were accepted at the site during this period.

Common municipal waste and solid and liquid industrial wastes were disposed of at the site during its years of operation. Industrial sludge containing acetone, tetrahydrofuran, toluene, xylene, and other organic substances were disposed of at the site from 1954 until 1962. During this period, the liquid wastes were commonly poured over garbage and burned. It was also reported that some liquid wastes

were poured down boreholes in the west-central portion of the landfill. (These boreholes were drilled as part of field-testing of drilling equipment.)

The site was placed on the National Priorities List (NPL) in June 1986. In March 1988, the two Potentially Responsible Parties (PRPs), Uniroyal Plastics, Inc., and the City of Stoughton, entered into an Administrative Order of Consent (AOC) with the United States Environmental Protection Agency (USEPA) and WDNR. This AOC required the completion of a remedial investigation and feasibility study (RI/FS). A Record of Decision (ROD) was signed for the site in September 1991. The ROD presents the site background and the selected remedial action for the site.

1.3 Summary of the Remedial Action

The landfill remedial actions identified in the ROD included fencing, land use restrictions, construction of an access road, excavation and relocation of waste in contact with groundwater, waste consolidation under final cover system, and placement of a new multilayer soil cover system with a passive landfill gas vent system over the relocated wastes and the landfill.

Permanent fencing and gates were installed around the perimeter of the site to restrict access and to eliminate the potential for exposure to landfill contaminants. Chain-link fencing with a locking gate at the landfill entrance was installed. The need to restrict the site access during remedial construction activities was evaluated, and a temporary fence was included as part of the remedial action plan for the site.

Land use restrictions were used to prevent the installation of public or private water supply wells within 1,200 feet of the property boundary and to prohibit residential development of the site.

A permanent site access road was built to provide access to the site during waste consolidation and capping activities. The access road was constructed along the southern border of the site in a location selected to minimize disruption of the residential area located south of the site and to minimize impact to the wetlands east of the site.

Waste consolidation consisted of excavating wastes in contact with the groundwater along the landfill's northeastern and southeastern boundaries, as well as consolidating the wastes on top of the landfill along

the site's western boundary. This minimized the direct contact of wastes within the groundwater and will result in a reduced impact to the wetlands adjacent to the site's eastern border. Prior to excavation, a dewatering pad was constructed to dewater the saturated waste. This pad consisted of a temporary clay-lined basin on top of the landfill, into which the excavated wastes were placed. The wastes were allowed to drain to a lower portion of the basin, and the water was collected. The dewatered wastes were then placed and compacted on top of the landfill during the regrading phase. A landfill multilayer soil cover system was placed over the existing landfill cover and the relocated waste per Wisconsin Administrative Code (WAC) NR 504.07.

## 1.4 Project Objectives and Scope

### *1.4.1 Specific Objectives*

The objective of the Quality Assurance Project Plan (QAPP) is to establish standard procedures so that the integrity, accuracy, precision, completeness, and representativeness of the samples and field activities are maintained and the required objectives of the operation and maintenance (O&M) program are achieved.

The objectives of the O&M program are to:

- Monitor the movement of the tetrahydrofuran (THF) and dichlorodifluoromethane (DCDFM) plumes to evaluate the effects of the landfill cap and natural attenuation on the THF and DCDFM plumes.
- Evaluate the site groundwater quality following the placement of the landfill cap and compare it to baseline groundwater quality. This reevaluation is to be completed every five years until the THF and DCDFM concentrations fall below the cleanup standards.
- Monitor the concentration of the landfill gases at the site boundary as a percentage of the lower explosive limit (LEL) for the landfill gases.
- Inspect the security fence, access road, and monitoring wells to ensure the site and wells are secure and accessible.
- Evaluate the landfill final cover with respect to the quality of vegetative cover, significant erosion problems, settlement or subsidence of cover, and mowing of cover vegetation.

- Inspect the landfill for stormwater runoff effects and erosion gullies, and evaluate need for backfilling, seeding, or mulching around and on top of landfill.

*1.4.2 Project Target Parameters and Intended Usages*

The groundwater-monitoring program is geared toward two volatile organic compounds (VOCs), THF and DCDFM. **Table 1** summarizes the annual groundwater and landfill gas sampling and analysis program.

All data obtained during sampling at the SCL, including field screening, field measurements, and laboratory analytical results, will be summarized in the Annual Groundwater Monitoring Report. Bimonthly landfill gas monitoring data will be included in the report. The data will be used by the WDNR to assess the effectiveness of the remedial actions at the site.

*1.4.3 Data Quality Objectives*

Data quality objectives (DQOs) are qualitative and quantitative statements that specify the quality of the data required to support decisions made during RI/FS activities and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. The following five analytical levels address various data uses and the quality assurance/quality control (QA/QC) effort and methods required to achieve the desired level of quality:

- DQO Level I: Provides the lowest data quality but the most rapid results. Level I is often used for health and safety monitoring, for preliminary comparison to appropriate or relevant and appropriate requirements (ARARs), during initial site characterization to locate areas for subsequent and more accurate analyses, and for engineering screening of alternatives (bench-scale tests). These types of data include those generated on site through the use of pH, conductivity, temperature, and other real-time monitoring equipment.
- DQO Level II: Provides rapid results and better quality than Level I. This level may include mobile lab-generated data, depending on the level of quality control exercised.
- DQO Level III: Provides an intermediate level of data quality and is used for site characterization. Engineering and analyses may include mobile lab-generated data and some analytical lab methods (i.e., laboratory data with quick turnaround used for screening but without full quality control documentation).

- DQO level IV: Provides the highest level of data quality and is used for purposes of risk assessment, evaluation of remedial alternatives, and PRP determination. These analyses require full Contract Laboratory Program (CLP) analytical and data validation procedures in accordance with USEPA-recognized protocol (includes CLP routine analytical services [RAS]).
- DQO Level V: Refers to analyses conducted by nonstandard protocols; for example, when exacting detecting limits or analysis of an unusual chemical compound is required. These analyses often require method development or adaptation. The level of quality control is usually similar to DQO Level IV data (includes CLP and non-CLP special analytical services [SAS]).

For O&M activities, DQO Analytical Level I will apply to readings generated during health and safety monitoring, water level measurements, and measurement of physical parameters by field instruments (e.g., pH, temperature, conductivity, and turbidity). Landfill gas monitoring for LEL (as methane), organic vapor, percent oxygen, percent carbon dioxide, and pressure will also be DQO Level I and will be measured by field instruments (landfill gas meter, photo-ionization meter).

DQO Level III will apply to all analytical data generated from laboratory analyses. Level III data quality will be generated by a subcontracted laboratory and is consistent with WDNR ch. NR 140.

The DQOs for all associated data collection activities, data types, data uses, and other data quality control factors are summarized in **Table 2**.

## 1.5 Sample Network Design and Rationale

This section presents the rationale for sampling frequency and analysis during the O&M phase of the remedial action. The sampling activities include groundwater monitoring and landfill gas monitoring.

### *1.5.1 Sample Network by Task and Matrix*

The baseline groundwater monitoring samples collected by Roy F. Weston in April 1998 were scheduled through the USEPA CLP for analysis of VOCs, and target analyte list (TAL) metals (filtered and unfiltered).

The O&M phase of the site will follow EPA SW-8260B analytical methods for annual VOC groundwater monitoring. Bottles utilized for the collection of samples will be provided by the subcontracted

laboratory and will be cleaned to USEPA specifications. Trip blanks will be prepared in accordance with USEPA methods and will be sent with the sample containers by the laboratory. A copy of the laboratory standard operating procedure (SOP) is included as **Appendix A**. Sample collection activities will conform to BT$^2$'s standard procedures as presented in **Appendix B**. **Table 2** lists the various analytical methods to be followed.

Based on the ROD and the Remedial Design (RD) Data Collection Report (Roy F. Weston, 1995), the WAC NR 140 preventive action limits (PALs) are identified as the applicable groundwater quality standards for the site. The historical groundwater contaminant results that have PAL or enforcement standard (ES) exceedances are shown in **Table 3**.

The landfill gas monitoring will be performed utilizing a direct reading field meter. The objective of gas probe monitoring is to monitor the concentration of the landfill gases at the site boundary. Parameters monitored include percent LEL as methane, percent oxygen, percent carbon dioxide, pressure, and VOCs by photo-ionization detector (PID).

**Table 1** is a summary of the sampling tasks, matrix, parameters, and frequency for the groundwater and landfill gas monitoring.

*1.5.2 Rationale of Selected Sampling Locations*

The Remedial Design Data Collection Report (Roy F. Weston, 1995) delineated the groundwater plume at the site as two disconnected plumes moving northwest from the site toward the Yahara River. Both plumes had PAL exceedances for both THF and DCDFM. The objectives of the sampling program are to monitor the movement of both plumes annually, evaluate natural attenuation, and to monitor the effect of the landfill cap on the THF and DCDFM plumes.

Biennial evaluations of the entire sampling program will be performed to determine the quality of site groundwater. Specifically, the following points will be evaluated:

- Do analytes need to be added or deleted from the analyte list?
- Is groundwater sampling frequency inadequate or excessive?
- Is the monitoring well network adequate?

- Do wells need to be replaced, installed, or deleted from the sample program?
- Does the analytical data indicate the THF and DCDFM plumes are decreasing or increasing? Should the sampling program continue or be modified?
- Is the landfill gas monitoring frequency inadequate or excessive?

In addition, it has been agreed to by the contractor for O&M services, BT², and the WDNR that the subcontracted laboratory will report any other VOC exceedances of the PAL or ES. These data will be used to help evaluate the sampling program.

### 1.6 Project Schedule

**Table 4** provides an anticipated schedule for project initiation, sampling milestones, monitoring activities, and completion of the project. Specifically, the project schedule includes:

- Semiannual inspections of facility components.
- Bimonthly gas probe monitoring.
- Annual groundwater monitoring and analysis.
- Submission of semiannual facility inspection reports.
- Submission of annual groundwater monitoring report.

## 2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

This section presents overall project organization and management, field, and laboratory responsibilities. Key responsibilities include project management, quality assurance, field operations, and laboratory operations and are discussed in the following subsections. Operational responsibilities involving execution and direct management of the technical and administrative aspects of this project are also discussed.

### 2.1 Management Responsibilities

*WDNR Project Manager*

The WDNR is overseeing the project for the USEPA. The WDNR Project Manager has the overall responsibility for all phases of the RD/RA. The WDNR Project Manager will provide final review/approval of the QAPP. The current WDNR Project Manager is Gary Edelstein.

*BT² Project Manager*

The BT² Project Manager has overall responsibility for ensuring that the project meets the WDNR's objectives and BT²'s quality standards. The BT² Project Manager is also responsible for ensuring that all work is executed in accordance with the WDNR's technical objectives. The BT² Project Manager is also responsible for ensuring that the technical, financial, and schedule objectives are achieved successfully.

The BT² Project Manager will coordinate with the BT² Quality Assurance/Senior Review Officer, the BT² Field Team Manager, and the WDNR Project Manager, and will act as the main point of contact and control for matters concerning the project. The BT² Project Manager is Leslie Busse.

*BT² Quality Assurance/Senior Review Officer*

The BT² Quality Assurance/Senior Review Officer will be responsible for data assessment and overall quality of the O&M phase of this project. The BT² Quality Assurance/Senior Review Officer will also provide final review and approval of all analytical reports to be submitted to the WDNR. The BT² Quality Assurance/Senior Review Officer is Sherren Clark.

## 2.2 Field Responsibilities

*BT² Field Team Leader*

The BT² Field Team Leader will be responsible for day-to-day activities at the site and will report directly to the BT² Project Manager. Specific responsibilities include:

- Day-to-day coordination with the BT² Project Manager on technical issues relevant to the site.
- Develop and implement field-related workplans, assure schedule compliance, adhere to the QAPP, and coordinate with the BT² Quality Assurance/Senior Review Officer.
- Coordinate and manage field staff during sampling.
- Act as field sample custodian and coordinate with subcontractor laboratory managers.
- Implement QC for field data.
- Write and approve text for field team efforts.
- Identify problems at the field team level, resolve difficulties, consult with BT² and WDNR Project Managers, and implement and document corrective actions.
- Prepare drafts of all required reports and site facility inspection logs.

The BT² Field Team Leader is Steven Smith.

2.3 Laboratory Responsibilities

The groundwater monitoring well samples will be analyzed by a subcontracted laboratory. The subcontracted laboratory is *TestAmerica, Inc.* of Watertown, Wisconsin. The Project Manager for TestAmerica is Dan Milewsky. The Quality Assurance Coordinator is Paul Junio.

*2.3.1 Laboratory Project Manager*

The Laboratory Project Manger will report to the BT² Field Team Leader and will be responsible for:

- Coordinating laboratory analyses
- Supervising in-house chain of custody (COC)
- Scheduling sample analyses
- Overseeing data review
- Overseeing preparation of analytical reports
- Approving final analytical reports prior to submission to BT²

*2.3.2 Laboratory Quality Assurance Officer*

The Laboratory QA Officer has the overall responsibility for data after it leaves the laboratory. The Laboratory QA Officer will:

- Overview laboratory quality assurance
- Overview QA/QC documentation
- Conduct detailed data review
- Determine whether to implement laboratory corrective actions
- Define appropriate laboratory QA procedures
- Prepare laboratory Standard Operation Procedures (SOPs)

## 3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective for this project is to develop and implement procedures for field sampling, COC, laboratory analysis, and reporting that will provide results which can be used by the WDNR to

evaluate remediation progress. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, reporting of data, internal QA, audits, preventive maintenance of field equipment, and corrective actions are described in other sections of this QAPP.

## 3.1 Precision

### *3.1.1 Definition*

Precision is a measure of the degree to which two or more measurements are in agreement.

### *3.1.2 Field Precision Objectives*

Field precision is assessed through the collection and measurement of field duplicates at a rate of one duplicate per ten analytical samples. The total numbers of duplicates for this project is provided in **Table 1**.

### *3.1.3 Laboratory Precision Objectives*

Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) and relative standard deviations (RSD) for three or more replicate samples. Precision control limits are included in the SOPs from TestAmerica in **Appendix A**.

## 3.2 Accuracy

### *3.2.1 Definition*

Accuracy is the degree of agreement between an observed value and an accepted reference value.

### *3.2.2 Field Accuracy Objectives*

Accuracy in the field for sample collection is assessed through the use of field and trip blanks and through the adherence to all sample handling, preservation, and holding times.

Field measurements are assessed by continuing calibration checks on the field instruments as described in each instrument's specific SOP.

*3.2.3 Laboratory Accuracy Objectives*

Laboratory accuracy is assessed through the analysis of matrix spikes (MS) or standard reference materials (SRM) and the determination of percent recoveries. Accuracy control limits are given in the provided in the SOPs from *TestAmerica, Inc.* (**Appendix A**).

## 3.3 Completeness

*3.3.1 Definition*

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

*3.3.2 Field Completeness Objectives*

Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. Field completeness for this project will be greater than 90 percent.

Validity of field measurements is based on adherence to the field instrument's SOP and through acceptable continuing calibration checks.

*3.3.3 Laboratory Completeness Objectives*

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. Laboratory completeness for this project will be greater than 95 percent.

## 3.4 Representativeness

*3.4.1 Definition*

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

*3.4.2 Measures to Ensure Representativeness of Field Data*

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the Field Sampling Plan (FSP) (outlined in **Section 4.0**) is followed and that proper sampling techniques are used.

*3.4.3 Measures to Ensure Representativeness of Laboratory*

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample holding times, and analyzing and assessing field duplicate samples. The sampling network is designed to provide data representative of facility conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data, physical setting and processes and constraints inherent to the Superfund program.

## 3.5 Comparability

3.5.1 Definition

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is also dependent on similar QA objectives.

*3.5.2 Measures to Ensure Comparability of Field Data*

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the FSP is followed and that proper sampling techniques are used.

*3.5.3 Measures to Ensure Comparability of Laboratory Data*

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented in the QAPP. Comparability is also dependent on similar QA objectives.

## 3.6 Level of Quality Control Effort

Field blank, trip blank, method blank, duplicate, SRM and MS samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs. Field and trip blanks will be collected for groundwater samples.

Field and trip blanks consisting of distilled water, will be submitted to the analytical laboratory to provide the means to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination at the facility which may cause the sample contamination. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Trip blanks will be collected for volatile organic samples only. Trip blanks are prepared prior to the sampling event in the actual sample containers and are kept with the investigative samples throughout the sampling event. They are then packaged for shipment with other samples and sent for analysis. There will be one trip blank included in each sample shipping container. At no time after their preparation are the sample containers opened before they reach the laboratory.

Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures. Matrix Duplicate samples (MSD) are analyzed to check for sampling and analytical reproducibility. Matrix Spike samples (MS) provide information about the effect of the sample matrix on the digestion and measurement methodology. All MSs are performed in duplicate and are hereinafter referred to as matrix spike/matrix spike duplicate (MS/MSD) samples. One MS/MSD will be prepared for every 20 or fewer investigative samples. MS/MSD samples are designated for organic analyses only.

The general level of the QC effort will be one field duplicate and one field blank for every ten or fewer investigative samples. The number of duplicate and field blank samples to be collected is listed in **Table 1**.

## 4.0 FIELD SAMPLING PLAN (FSP)

This Field Sampling Plan describes the field sampling protocols to be followed as part of the O&M for the landfill remediation at the SCL site in Stoughton, Wisconsin. Specifically, the FSP addresses the following:

- Sampling plan rationale
- Field sampling procedures
- Numbers, locations, and types of samples

- QA/QC of field sampling
- Sample numbering system
- Sample containers and preservation
- Sample packaging and shipment
- COC procedures
- Documentation
- Sampling team organization
- Management of investigation-derived wastes
- Sample container procurements

During the O&M, additional field sampling may be necessary. If the additional field sampling is not covered in this FSP, an addendum to the FSP should be made at the appropriate time, and approval should be obtained by the WDNR before initiating fieldwork.

## 4.1 Sample Network Design and Rationale

This section presents the rationale for sampling frequency and analysis during the O&M phase of the remedial action. The sampling activities include groundwater and landfill gas monitoring and are summarized in **Table 1**.

### *4.1.1 Groundwater Monitoring*

The groundwater monitoring has the following objectives:

- Monitor the movement of the THF and DCDFM plumes annually to evaluate the effects of natural attenuation and the landfill cap on the THF and DCDFM plumes.
- Evaluate the site groundwater quality following the placement of the landfill cap and compare it to baseline groundwater quality. This reevaluation is to be completed every five years until the THF and DCDFM concentrations fall below the PALs.

### *4.1.2 Routine Groundwater Monitoring*

Routine groundwater monitoring will be conducted annually. The objective of the routine groundwater monitoring is to monitor the movement of THF and DCDFM plumes. Therefore, only VOC analysis will be performed. The selected monitoring wells located on the western edge of the landfill (13 monitoring

wells) will be used for the routine groundwater monitoring. These include monitoring wells 3D, 4D, 5D, 7I, 8I, 9S, 9I, 9D, 10S, 10I, 13I, 14S, and 14I. Sampling procedures are described in **Appendix B**.

*4.1.3 Gas Probe Monitoring*

The objective of gas probe monitoring is to monitor the concentration of the landfill gases at the site boundary. Parameters monitored include percent LEL as methane, percent oxygen, percent carbon dioxide, pressure, and VOCs by photo-ionization detector (PID).

During the predesign activities, Roy F. Weston, Inc., used a combustible gas indicator (CGI) to periodically monitor the concentration of the landfill gases as a percentage of the LEL for the landfill gases at the monitoring probes outside the site boundary. The percent LEL readings at these locations during the predesign monitoring were 0. During the remedial action (RA), a series of landfill gas monitoring probes were installed outside the waste boundary. These probes will be monitored bimonthly to verify that the methane concentration is below 25 percent of the LEL.

4.3 Field Investigation Protocols

The following sections detail the procedures that will be followed during the O&M field sampling activities. All sample container preservation and volume requirements are outlined in **Table 5**. All activities will follow BT²'s standard procedures, which are included as **Appendix B**.

*4.3.1 Water Level Measurement*

Prior to the sampling of monitoring wells, water level measurements will be collected. The water level data will be used in determining the approximate direction of groundwater flow, and will provide information on lateral and vertical hydraulic gradients. The following protocols will be used during water level measurement:

- The water level probe and cable will be decontaminated prior to each use with a distilled water rinse.
- Depth to water will be measured with an electrical sounding device (accuracy ± 0.01 feet). The reference point for this measurement will be the top of the well riser pipe. Measurements will be converted into elevations (i.e., mean sea level), using established survey information.
- The depth to water and the time will be recorded in a field book.

*4.3.2 Groundwater Monitoring Well Sampling Procedures*

Monitoring wells will be sampled using a submersible pump utilizing a very slow flow rate (0.2 to 2 liters per minute [l/min]) or by a dedicated bailer. Sampling equipment and all downhole equipment will be decontaminated pursuant to the protocols outlined in **Table 6**. Each sample will be collected using the following methodology as spelled out in **Appendix B**.

- The depth to the water level in the well and the total depth of the well will be measured with an electrical sounding device (accuracy ± 0.01 feet). The depth to water and the time of measurement will be recorded. The reference point for these depths will be the top of the well riser pipe.
- The volume of standing water in the well will be calculated. Volume of water in a 2-inch-diameter well (gallons) = length (feet) x 0.16 (gallons/foot). For a 4-inch-diameter well (gallons) = length (feet) x 0.65 (gallons/foot). For a 6-inch-diameter well (gallons) = length (feet) x 1.47 (gallons/foot).
- Per Sec. 2.4.A of the WDNR Groundwater Sampling Field Manual (Publ. DG-038-96), a submersible pump that has been decontaminated prior to use will be used for purging and sampling utilizing a very slow flow rate (<2.0 l/min). Tubing will be thick and of minimal length to exclude atmospheric gases.
- Well purging will be conducted at low flow rates (1.0 to 4.0 l/min) with the pump intake just above or within the screened interval. Field measurements of pH, temperature, conductivity, dissolved oxygen, and turbidity will be made over time. Stabilization of these well purging parameters (± 0.25 units for pH, ± 0.5°C for temperature, ± 10 percent for conductivity, ± 0.1 mg/l for dissolved oxygen, and ± 1 units for turbidity) indicates equilibrated conditions. Well purging will continue until four purge volumes have been removed.
- In the event that the monitoring well pumps dry before three volumes have been removed, the well will be allowed to recharge for 15 minutes and then be pumped dry again before sampling. All purge water will be containerized and managed in accordance with **Section 9** protocols.
- Samples will be collected directly from the pump after the well purging has been completed.
- Sampling bottles will be filled at an angle in order to limit splashing and bubbling. VOC sample bottles will be preserved with hydrochloric acid (HCl) prior to the addition of the sample. The VOC sample bottles will be filled such that no air space is present in the bottle after it is capped. If bubbles appear after the bottle is capped, additional sample (water) will be added and the bottle

resealed. If the sample has to be discarded and a new sample collected, a new, preserved VOC container will be used to collect the sample. If bubbles persist, an unpreserved VOC sample will be collected. (The BT² Field Team Manager will note the absence of the preservative on the sample paperwork and in the field logbook.)

- For the shallow wells that are sampled via a dedicated bailer, Section 2.4.2 of the WDNR Groundwater Sampling Field Manual (Publ. DG-038-96), will be followed. The bailer will be slowly lowered and raised in the water column. A bottom-emptying device will be used to decant samples from the bailer.

*4.3.3 Gas Probe Monitoring Procedures*

The gas probes will be measured by using the direct reading GEM2000 Landfill Gas Meter and a photo-ionization detector (PID). The landfill gas meter and the PID will be field calibrated prior to each monitoring event by the procedures described in **Appendix B**. At each gas probe, the GEM2000 will be used to purge the stagnant air inside the gas probe utilizing the internal pump of the meter for 2 minutes. Following well purging, the two meters will measure the gas probe for percent LEL as methane, percent oxygen, percent carbon dioxide, pressure, and VOCs (PID).

4.4 Decontamination Requirements

All sampling equipment will be decontaminated before being used to collect a sample. The decontamination protocol for sampling equipment is presented in **Table 6**. The management of water generated during decontamination will be in accordance with the requirements outlined in **Section 9**. All decontamination wastewater will be containerized.

4.5 Field Quality Control Samples

The O&M sampling effort will include the following types of field QC samples:

- Field duplicates
- Field blanks
- Trip blanks

This section of the QAPP explains the purpose of each type of QC sample. Sample containers and handling and shipment procedures used for QC samples are identical to those used for the investigative samples. Each field QC sample will be documented on a COC form.

*4.5.1 Field Duplicate Samples*

Field duplicate samples will be collected at selected locations during water sampling at 1 per 10-sample frequency using procedures identical to those for the investigative samples. Duplicate samples will be analyzed for the same parameters as the investigative samples and one duplicate will be analyzed for THF and DCDFM only while the other duplicate will be analyzed for the full list VOCs. Duplicate samples will be collected by alternatively filling two sets of sample bottles from the same sample unit. The VOC analysis fraction for each duplicate sample will be collected immediately after the VOC fraction for the investigative sample, in order to minimize the possibility of loss of VOCs during sample collection.

*4.5.2 Field Blanks*

One field blank sample will be collected during the annual groundwater monitoring event. The field blank will be obtained by pumping deionized water over and through a decontaminated well sampling pump, and collecting the water in the required sample containers. The field blank will be analyzed for the full list of VOCs in accordance with the same analytical methodologies. The field blank will be identified as such on the sample documentation.

*4.5.3 Trip Blanks*

One trip blank sample will be enclosed in each sample shipment container in which aqueous VOC samples are included. Trip blanks will consist of two 40-milliliter (ml) glass vials. All sample handling, packaging, and preservation requirements for the trip blanks will be identical to the investigative VOC sample aliquot. The 40-ml vials for each trip blank will be filled by the laboratory. Preparation of the trip blank will entail the pouring of ultra pure water (HPLC-grade water) into the 40-ml vial (leaving no airspace) and carefully securing the caps to ensure the absence of air bubbles. The sealed bottles will be subsequently placed in a sample container and accompany field personnel to the sample site. All trip blanks will be shipped to the laboratories in containers with other VOC samples. The trip blank will be documented and identified as such on all sample documentation.

## 5.0 CUSTODY PROCEDURES

Custody is one of several factors that is necessary for the admissibility of environmental data as evidenced in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained by the subcontracted laboratory.

A sample or evidence file is under your custody if:

- the item is in actual possession of a person; or
- the item is in the view of the person after being in actual possession of the person; or
- the item was in actual physical possession but is locked up to prevent tampering; or
- the item is in a designated and identified secure area.

### 5.1 Field Custody Procedures

Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that persons going to the facility could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in the document control center when not in use. Each logbook will be identified by the project-specific document number.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in ink, signed, and dated and no erasures will be made. If an incorrect entry is made, the information will be crossed out

with a single strike mark, which is signed and dated by the sampler. The number of the photographs taken of the station, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in **Section 4** of this QAPP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume, and number of containers. The sample packaging and shipment procedures summarized below will ensure that the samples will arrive at the laboratory with the COC intact.

- The BT$^2$ Field Team Manager is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.
- All bottles will be identified by use of sample labels with sample numbers, sampling locations, date/time of collection, and type of analysis.
- Sample labels are to be completed for each sample using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample label because the ballpoint pen would not function in freezing weather.
- Samples are accompanied by a properly completed COC form. The sample numbers and locations will be listed on the COC form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.
- Samples will be properly packaged on ice at 4° C for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in and secured to the inside top of each sample box or cooler.
- All shipments will be accompanied by the COC record identifying the contents. The original record will accompany the shipment and copies will be retained by the sampler for returning to the sampling office.
- Samples will be picked up by the laboratory courier the next day after the samples are collected in the field.

5.2 Laboratory Custody Procedures

Laboratory custody procedures for sample receiving and login, sample storage and numbering, tracking during sample preparation and analysis, and storage of data are described in the laboratory SOPs.

5.3 Sample Identification System

Sample containers will be labeled prior to being filled. Each sample label shall, at minimum, indicate

- Sample type
- Date/time of sample collection
- Sampler's initial
- Required analyses
- Type of preservative
- $BT^2$ sample location number

5.4 Final Evidence Files

Per the O&M Bid Package dated July 2005, the WDNR has stated that the subcontracted laboratories shall prepare an entire data package complete with QC information. The laboratories will retain, but have available for distribution, the data package for a minimum of five years, in the event that either the WDNR or the USEPA would want to have the data validated.

## 6.0 CALIBRATION PROCEDURES AND FREQUENCY

This section describes the calibration procedures and the frequency at which these procedures will be performed for both field and laboratory instruments.

6.1 Field Instrument Calibration

The field instruments will be calibrated as described in field SOPs. Field instruments include a pH meter, thermometer, conductivity meter, organic vapor meter, and a landfill gas meter. As a rule, instruments will be calibrated daily prior to use and will be recalibrated every 20 samples. See **Appendix B** for the specific procedures used.

All the calibration procedures performed will be documented in the field logbook and will include the date/time of calibration, name of person performing the calibration, reference standard use, temperature at which readings were taken, and the readings. Multiple readings on one sample or standard, as well as readings on replicate samples, will likewise be documented.

*6.1.1 pH Meter*

The pH meter will be calibrated with standard buffer solutions before being taken into the field. In the field, the meter will be calibrated daily with two buffer solutions before use. The range of the buffer solutions will be at least three or more pH units apart and will bracket the expected pH of the sample being measured. Refer to the specific SOP included in **Appendix B**.

The calibrations performed, standard used, and sample pH values are to be recorded in the field notebook. Appropriate new batteries will be purchased and kept with the meters to facilitate immediate replacement in the field as necessary.

*6.1.2 Thermometer*

Temperature readings will be taken off of the pH meter and conductivity meter. During groundwater monitoring, the temperature readings from both the pH and conductivity meters will be compared to ensure proper readings. The temperature will be recorded from the pH meter. If there is more than 15 percent difference from these two instruments, a direct-reading mercury thermometer will be used for all temperature readings.

*6.1.3 Conductivity Meter*

The conductivity cells of the specific conductivity meter will be cleaned and checked against known conductivity standards before being taken to the field. In the field, the instrument will be checked daily with NIST traceable reference standards. The calibration procedure is described in **Appendix B**.

All readings and calibrations should be recorded in the field notebook.

*6.1.4 Photo-ionization Detector (PID)*

The PID will be calibrated daily with 100 ppm isobutylene span gas. Records of the calibration, dates, and analyst name will be recorded in the instruments logbook.

*6.1.5 GEM2000 Landfill Gas Meter (GEM2000)*

The GEM2000 Landfill Gas Meter is factory calibrated and a calibration self check is performed in the field to correct for any small drifts in the electronics and transducers that may occur over time. The GEM2000 meter can also be checked against calibration span gases purchased by BT². See **Appendix B** for the procedures and operation.

6.2 Laboratory Instrument Calibration

The specific calibration procedures, including continued calibration verification, intervals for verification, and standard preparations are described in the SOPs supplied by the laboratories in **Appendix A**.

## 7.0 DATA REDUCTION, VALIDATION, REPORTING, AND MANAGEMENT

All data generated through in-field activities, or by the laboratory operation shall be reduced prior to reporting. No data shall be disseminated by the laboratory until it has been subjected to these procedures which are summarized in subsections below.

7.1 Data Reduction

*7.1.1 Field Data Reduction Procedures*

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. Only direct-read instrumentation will be employed in the field. The use of pH meters, thermometers, an OVA, and a probe to measure specific conductance will generate some measurements directly read from the meters following calibration per manufacturer's recommendations as outlined in **Section 6** of this QAPP. Such data will be written into field logbooks immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed, and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, when the results forms required for this study are being filled out, the Field Manager, identified in **Section 2** of this QAPP, will proof the forms to determine whether any transcription errors have been made by the field crew.

*7.1.2 Laboratory Data Reduction Procedures*

Laboratory data reduction procedures will be followed according to the following protocol: A raw analytical data will be recorded in numerically identified laboratory notebooks. These notebooks will be issued only by the Laboratory QA Manager. Data are recorded in this notebook along with other pertinent information, such as the sample identification number and the sample tag number. Other details will also be recorded in the lab notebook, such as the analytical method used (SOP #), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings, and the raw data. Each page of the notebook shall be signed and dated by the analyst. Copies of any strip chart printouts (such as gas chromatograms) will be maintained on file. Periodic review of these notebooks by the Lab QA Manager takes place prior to final data reporting. (Records of notebook entry inspections are maintained by the Lab QA Manager.)

All calculations are checked by the Supervisor at the conclusion of each operating day. Errors are noted, corrections are made, but the original notations are crossed out legibly.

Quality control data (e.g., laboratory duplicates, surrogates, matrix spikes, and matrix spike duplicates) will be compared to the method acceptance criteria. Data considered to be acceptable will be entered into the laboratory computer system. Data summaries will be sent to the Laboratory QA Manager for review. If approved, data are logged into the project database format. Unacceptable data shall be appropriately qualified in the project report. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits, and any other anomalous conditions encountered during sample analysis. After the Lab QA Manager approves these data, they are considered ready for third-party data validation and will be stored.

7.2 Data Reporting

Data reporting procedures shall be carried out for field and laboratory operations as indicated below.

*7.2.1 Field Data Reporting*

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field, and documentation of all field calibration activities.

*7.2.2 Laboratory Data Reporting*

The Laboratory QA Manager must perform a final review of the report summaries to determine if the report meets the project requirements. The report summary shall consist of:

- Date of issuance
- Laboratory analysis performed
- Laboratory batch number
- Project name and number
- Conditions of samples "as-received" along with a copy of the COC
- Data footnotes and descriptions of the footnotes
- Sample results
- Quality control report with MS/MSD results, lab control sample results, method blank results, calibration check compounds, and system performance check compound results
- Results of any other VOCs detected above the PAL or ES

The data validation package will contain:

1. Case Narrative:
   - Date of issuance
   - Laboratory analysis performed
   - Any deviations from intended analytical strategy
   - Laboratory batch number
   - Numbers of samples and respective matrices
   - Quality control procedures utilized and also references to the acceptance criteria
   - Laboratory report contents
   - Project name and number
   - Condition of samples "as-received"
   - Discussion of whether or not sample holding times were met
   - Discussion of technical problems or other observations which may have created analytical difficulties
   - Discussion of any laboratory quality control checks which failed to meet project criteria

- Signature of the Laboratory QA Manager

2. Chemistry Data Package:
   - Case narrative for each analyzed batch of samples
   - Summary page indicating dates of analyses for samples and laboratory quality control checks
   - Cross referencing of laboratory sample to project sample identification numbers
   - Data qualifiers to be used should be adequately described
   - Sample preparation and analyses for samples
   - Sample results
   - Raw data for sample results and laboratory quality control samples
   - Results of (dated) initial and continuing calibration checks, and GC/MS tuning results
   - MS and MS duplicate recoveries, laboratory control samples, method blank results, calibration check compounds, and system performance check compound results
   - Labeled (and dated) chromatograms/spectra of sample results and laboratory quality control checks
   - Results of tentatively identified compounds

7.3 Data Management

There are several types of data that will be part of the overall database. These include physical data and chemical data. The chemical data are also of two varieties: field-measured parameters (VOC vapor concentrations, for example) and analytical results from the laboratory. Physical data and field-measured parameters will be entered into an electronic database format. Each staff member associated with field activities will proof and validate the data that they collect, and then provide it to the Field Team Manager for electronic format entry. All electronic data will be considered non-validated.

The laboratory shall provide chemical data in an acceptable electronic format that has been checked for accuracy. The laboratory shall also provide a hard copy of the data, which shall be filed as indicated above.

Occasionally, data may be recorded incorrectly in the field and, as a result, produce an illogical, unreasonable, or incorrect result in a calculated value. If this situation is noted during the initial data entry checking process, the action taken will be noted on the original field data sheet. Once data has been

checked and entered into the authoritative database, any changes to the data will need to be documented as to what the changes are, why they are being made, and who is making the changes.

*7.3.1 Records Retention*

The subcontracted laboratories will retain all analytical records for five years following the termination of activities, in the event that either regulatory agency would want to have the data validated. The labs shall prepare an entire data package complete with QC information.

*7.3.2 Archiving Data*

In order to provide for the long-term security and integrity of the data, the data will be archived in a secure format and location. In addition to archiving field data, data as received from the laboratory will also be archived.

*7.3.3 Project File Requirements*

BT² will maintain project files, including the raw laboratory data generated, during the O&M activities of this project. The project file requirements will be continually expanded, as necessary, due to additional activities and data generation. The project files will consist of, at a minimum, the following information:

- Field books, field raw data
- Groundwater sampling logs
- COC forms
- Laboratory analytical reports - hard copy and electronic
- Investigation Workplans/SAPs, including QAPP, DMP, and Health and Safety Plan
- Correspondence in, out, and telephone correspondence
- Project Progress Reporting
- Figures and/or photographs
- Survey data
- Aerial photographs
- Any other applicable files

## 8.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities can be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the FSP and QAPP, if requested by the WDNR.

8.1 Field Performance and System Audits

*8.1.1 Internal Field Audits*

*8.1.1.1 Internal Field Audit Responsibilities*

Internal audits of field activities including sampling and field measurements would be conducted by the BT$^2$ Quality Assurance/Senior Review Officer.

*8.1.1.2 Internal Field Audit Frequency*

These audits would verify that all established procedures are being followed. Internal field audits frequency would be requested by the WDNR.

*8.1.1.3 Internal Field Audit Procedures*

The audits would include examination of field sampling records, field instrument operating records, sample collection, handling, and packaging in compliance with the established procedures, maintenance of QA procedures, COC, etc. Follow-up audits would be conducted to correct deficiencies, and to verify that QA procedures are maintained throughout the remediation. The audits would involve review of field measurement records, instrumentation calibration records, and sample documentation. The results of the field audit would be submitted to the WDNR as requested.

*8.1.2 External Field Audits*

*8.1.2.1 External Field Audit Responsibilities*

External field audits may be conducted by the WDNR.

*8.1.2.2 External Field Audit Frequency*

External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of the WDNR.

*8.1.2.3 Overview of the External Field Process*

External field audits will be conducted according to the field activity information presented in the QAPP.

8.2 Laboratory Performance and Systems Audits

*8.2.1 Internal Laboratory Audits*

*8.2.1.1 Internal Laboratory Audit Responsibilities*

The internal laboratory audit would be conducted by the BT² Quality Assurance/Senior Review Officer, if requested by the WDNR.

*8.2.1.2 Internal Laboratory Audit Frequency*

The internal lab system audits would be done on a basis to be determined by the WDNR.

*8.2.1.3 Internal laboratory Audit Procedures*

The internal lab system audits would include an examination of laboratory documentation on sample receiving, sample login, sample storage, COC procedures, sample preparation and analysis, instrument operating records, etc. The BT² Quality Assurance/Senior Review Officer would evaluate the laboratory's practices to ensure the laboratory maintains acceptable QC performance. Results of the audit would be submitted to the WDNR as requested.

*8.2.2 External Laboratory Audits*

*8.2.2.1 External Laboratory Audit*

An external audit may be conducted by the WDNR.

*8.2.2.2 External Laboratory Audit Frequency*

An external lab audit may be conducted at least once prior to the initiation of the sampling and analysis activities. These audits may or may not be announced and are at the discretion of the WDNR.

8.2.2.3 Overview of the External Laboratory Audit Process

External lab audits will include (but not be limited to) review of laboratory analytical procedures, laboratory on-site audits, and/or submission of performance evaluation samples to the laboratory for analysis.

## 9.0 PREVENTATIVE MAINTENANCE

9.1 Field Instrument Preventative Maintenance

The field equipment for this project includes landfill gas meter, thermometers, pH meter, conductivity meter, and photo-ionization meter. Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Field instruments will be checked and calibrated daily before use. Calibration checks will be documented on the Field Meter/calibration log sheets. The maintenance schedule and troubleshooting procedures for field instruments are indicated in a submitted table. Critical spare parts such as tape, pH probes, and batteries will be kept on site to reduce downtime. Backup instruments and equipment will be available on site or within 1-day shipment to avoid delays in the field schedule.

9.2 Laboratory Instrument Preventative Maintenance

As part of their QA/QC Program, a routine preventative maintenance program is conducted by the laboratories to minimize the occurrence of instrument failure and other system malfunctions. TestAmerica has an internal group to perform routine scheduled maintenance, and to repair or to coordinate with the vendor for the repair of all instruments. All laboratory instruments are maintained in accordance with the manufacturer's specifications and the requirements of the specific method employed. This maintenance is carried out on a regular, scheduled basis, and is documented in the laboratory instrument service logbook for each instrument. Emergency repair or scheduled manufacturer's maintenance is provided under a repair and maintenance contract with factory representatives.

## 10.0 CORRECTIVE ACTION

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out of quality control performance which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation, and data assessment. All corrective action proposed and implemented will be documented in the regular quality assurance reports to management. Corrective action should only be implemented after approval by the $BT^2$ Project Manager, or the $BT^2$ Field Team Manager. If immediate corrective action is required, approvals secured by telephone from the $BT^2$ Project Manager or Field Team Manager should be documented in an additional memorandum.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem will be responsible for notifying the $BT^2$ Project Manager, who in turn will notify the WDNR Project Manager. Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established quality control procedures in the QAPP or FSP will be identified and corrected in accordance with the QAPP. The $BT^2$ Field Team Manager will issue a nonconformance report for each nonconformance condition.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by stop-work order by the WDNR Project Manager.

### 10.1 Field Corrective Action

Corrective action in the field can be needed when the sample network is changed (i.e., more/less samples, sampling locations other than those specified in the QAPP, etc.), sampling procedures and/or field analytical procedures require modification, etc., due to unexpected conditions. Technical staff and project personnel will be responsible for reporting all suspected technical or QA nonconformance's or suspected deficiencies of any activity or issued document by reporting the situation to the $BT^2$ Project Manager or designee. This manager will be responsible for assessing the suspect problems in consultation with the

BT² Quality Assurance/Senior Review Officer on making a decision based on the potential for the situation to impact the quality of the data. If it is determined that the situation warrants a reportable nonconformance requiring corrective action, then a nonconformance report will be initiated by the BT² Field Team Manager.

The BT² Field Team Manager will be responsible for ensuring that corrective action for nonconformances are initiated by:

- Evaluating all reported nonconformance's
- Controlling additional work on nonconforming items
- Determining disposition or action to be taken
- Maintaining a log of nonconformance's
- Reviewing nonconformance reports and corrective actions taken

Eensuring nonconformance reports are included in the final site documentation in project files

If appropriate, the Field Team Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the corrective actions are completed. Corrective action for field measurements may include:

- Repeat the measurement to check the error
- Check for all proper adjustments for ambient conditions such as temperature
- Check the batteries
- Re-calibration
- Check the calibration
- Replace the instrument or measurement devices
- Stop work (if necessary)

The Field Team Manager or the designee is responsible for all site activities. In this role, the Field Team Manager at times is required to adjust the site programs to accommodate site-specific needs. When it becomes necessary to modify a program, the Field Team Manager notifies the BT² Project Manager of the anticipated change and implements the necessary changes after obtaining the approval of the Project Manager. The Field Team Manager for the SCL site is responsible for the controlling, tracking, and

implementation of the identified changes. Reports on all changes will be distributed to all affected parties, which include the WDNR Project Manager. The $BT^2$ Project Manager will be notified whenever program changes in the field are made and will notify the WDNR Project Manager if the situation warrants.

Implementation of corrective actions will be performed by the Field Team Manager and field team. Corrective action will be documented in quality assurance reports to the entire project management.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by the WDNR.

10.2 Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, during, and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, potentially high concentration samples may be identified during sample login or just prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the laboratory QC Coordinator to approve the implementation of corrective action. The submitted standard SOPs specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, automatic reinjection/reanalysis when certain quality control criteria are not met, etc.

## TABLES

**Table 1**
**Summary of Annual O&M Sampling and Analysis Program**
**Stoughton City Landfill, Stoughton, Wisconsin - BT[2] Project #1764**

| | | | | Investigative | | | Field Duplicate | | | Field Blank | | | MS/MSD[3] | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| O&M Task | Sample Matrix | Field Parameters | Laboratory Parameters | No. | Freq. | Total | No. | Freq. | Total | No. | Freq. | Total | No. | Freq. | Total | Matrix Total[4] |
| Routine Groundwater Monitoring | Groundwater | Water Level, pH, Conductivity, Temperature, Turbidity, Dissolved Oxygen | THF, DCDFM only[2] | 6 | 1 | 6 | 1 | 1 | 1 | 0 | 0 | 0 | NA | NA | NA | 7 |
| Routine Groundwater Monitoring | Groundwater | Water Level, pH, Conductivity, Temperature, Turbidity, Dissolved Oxygen | Full list VOCs[2] | 7 | 1 | 7 | 1 | 1 | 1 | 1 | 1 | 1 | NA | NA | NA | 9 |
| Landfill Gas Probe Monitoring | Air | LEL as Methane, Oxygen, Carbon dioxide, VOCs by PID, and Pressure | None | 3 | 6 | 18 | NA | NA | NA | NA | NA | NA | NA | NA | NA | 18 |

NOTES:

1. The groundwater monitoring wells to be sampled are wells: 3D,4D, 5D, 7I, 8I, 9S, 9I, 9D, 10S, 10I, 13I, 14S, 14I.
2. Full list VOCs to be analyzed on samples from wells: 9S, 9I, 9D, 10S, 10I, 14S, 14I. THF and DCDFM only to be analyzed on samples from wells: 3D, 4D, 5D, 7I, 8I, 13I.
3. Matrix spike/matrix spike duplicate (MS/MSDs) are not additional samples, but are samples on which the MS/MSD analysis will be performed by the laboratory. MS/MSDs will be performed on the organic samples only. Duplicate/spike analyses are performed on the inorganic samples.
4. The matrix total does not include trip blank samples or MS/MSD samples. One trip blank will be included with each VOC sample shipment.

By: S. Smith
Date: 3/10/06
Checked by:

**Table 2**
**Summary of Data Quality Objectives**
**Stoughton City Landfill, Stoughton, Wisconsin - BT² Project #1764**

| Analyte | State Required Quantitation Limit (µg/l)[1] | Preventive Action Limit (PAL) (µg/l) | Enforcement Standard (ES) (µg/l) | Laboratory Method Detection Limit (MDL) (µg/l) | Laboratory Limit of Quantitation (LOQ) (µg/l) | Analytical Method |
|---|---|---|---|---|---|---|
| **Groundwater - Volatile Organic Compounds** | | | | | | |
| Benzene | 10 | 0.5 | 5 | 0.2 | 0.67 | SW 8260B |
| Bromobenzene | 10 | – | – | 0.2 | 0.67 | SW 8260B |
| Bromochloromethane | 10 | – | – | 0.5 | 1.7 | SW 8260B |
| Bromodichloromethane | 10 | 0.06 | 0.6 | 0.2 | 0.67 | SW 8260B |
| Bromoform | 10 | 0.44 | 4.4 | 0.2 | 0.67 | SW 8260B |
| Bromomethane | 10 | 1 | 10 | 0.2 | 0.67 | SW 8260B |
| n-Butylbenzene | 10 | – | – | 0.2 | 0.67 | SW 8260B |
| sec-Butylbenzene | 10 | – | – | 0.25 | 0.83 | SW 8260B |
| tert-Butylbenzene | 10 | – | – | 0.2 | 0.67 | SW 8260B |
| Carbon Tetrachloride | 10 | 0.5 | 5 | 0.5 | 1.7 | SW 8260B |
| Chlorobenzene | 10 | – | – | 0.2 | 0.67 | SW 8260B |
| Chlorodibromomethane | 10 | – | – | 0.2 | 0.67 | SW 8260B |
| Chlorethane | 10 | 80 | 400 | 1 | 3.3 | SW 8260B |
| Chloroform | 10 | 0.6 | 6 | 0.2 | 0.67 | SW 8260B |
| Chloromethane | 10 | – | – | 0.2 | 0.67 | SW 8260B |
| 2-Chlorotoluene | 10 | – | – | 0.5 | 1.7 | SW 8260B |
| 4-Chlorotoluene | 10 | – | – | 0.2 | 0.67 | SW 8260B |
| 1,2-Dibromo-3-chloropropane | 10 | 0.02 | 0.2 | 0.5 | 1.7 | SW 8260B |
| 1,2-Dibromoethane (EDB) | 10 | 0.005 | 0.5 | 0.2 | 0.67 | SW 8260B |
| Dibromomethane | 10 | – | – | 0.2 | 0.67 | SW 8260B |
| 1.2-Dichlorobenzene | 10 | 60 | 600 | 0.2 | 0.67 | SW 8260B |
| 1.3-Dichlorobenzene | 10 | 125 | 1,250 | 0.2 | 0.67 | SW 8260B |
| 1.4-Dichlorobenzene | 10 | 15 | 75 | 0.2 | 0.67 | SW 8260B |
| Dichlorodifluoromethane (DCDFM) | 10 | 200 | 1,000 | 0.5 | 1.7 | SW 8260B |
| 1,1-Dichloroethane | 10 | 85 | 850 | 0.5 | 1.7 | SW 8260B |
| 1.2-Dichloroethane | 10 | 0.5 | 5 | 0.5 | 1.7 | SW 8260B |
| 1,1-Dichloroethene | 10 | 0.7 | 7 | 0.5 | 1.7 | SW 8260B |
| cis-1,2-Dichloroethene | 10 | 7 | 70 | 0.5 | 1.7 | SW 8260B |
| trans-1,2-Dichloroethene | 10 | 20 | 100 | 0.5 | 1.7 | SW 8260B |
| 1,3-Dichloropropane | 10 | 0.02 | 0.2 | 0.25 | 0.83 | SW 8260B |
| 2,2-Dichloropropane | 10 | – | – | 0.5 | 1.7 | SW 8260B |
| 1,1-Dichloropropene | 10 | – | – | 0.5 | 1.7 | SW 8260B |
| cis-1,3-Dichloropropene | 10 | 0.02 | 0.2 | 0.2 | 0.67 | SW 8260B |
| trans-1,3-Dichloropropene | 10 | 0.02 | 0.2 | 0.2 | 0.67 | SW 8260B |
| Isopropyl Ether | 10 | – | – | 0.5 | 1.7 | SW 8260B |
| Ethylbenzene | 10 | 140 | 700 | 0.5 | 1.7 | SW 8260B |
| Hexachlorobutadiene | 10 | – | – | 0.5 | 1.7 | SW 8260B |
| Isopropylbenzene | 10 | – | – | 0.2 | 0.67 | SW 8260B |
| p-Isopropyltoluene | 10 | – | – | 0.2 | 0.67 | SW 8260B |
| Methylene Chloride | 10 | 0.5 | 5 | 1 | 3.3 | SW 8260B |
| Methyl tert-Butyl Ether | 10 | 12 | 60 | 0.5 | 1.7 | SW 8260B |
| Naphthalene | 10 | 8 | 40 | 0.25 | 0.83 | SW 8260B |
| n-Propylbenzene | 10 | – | – | 0.5 | 1.7 | SW 8260B |
| Styrene | 10 | 10 | 100 | 0.2 | 0.67 | SW 8260B |
| 1,1,1,2-Tetrachloroethane | 10 | 7 | 70 | 0.25 | 0.83 | SW 8260B |
| 1,1,2,2-Tetrachloroethane | 10 | 0.02 | 0.2 | 0.2 | 0.67 | SW 8260B |
| Tetrachloroethene | 10 | 0.5 | 5 | 0.5 | 1.7 | SW 8260B |
| Tetrahydrofuran | 10 | 10 | 50 | 0.5 | 1.7 | SW 8260B |
| Toluene | 10 | 200 | 1,000 | 0.2 | 0.67 | SW 8260B |
| 1,2,3-Trichlorobenzene | 10 | – | – | 0.25 | 0.83 | SW 8260B |
| 1,2,4-Trichlorobenzene | 10 | 14 | 70 | 0.25 | 0.83 | SW 8260B |
| 1,1,1-Trichloroethane | 10 | 40 | 200 | 0.5 | 1.7 | SW 8260B |
| 1,1,2-Trichloroethane | 10 | 0.5 | 5 | 0.25 | 0.83 | SW 8260B |
| Trichloroethene | 10 | 0.5 | 5 | 0.2 | 0.67 | SW 8260B |
| Trichlorofluoromethane | 10 | – | – | 0.5 | 1.7 | SW 8260B |
| 1,2,3-Trichloropropane | 10 | 12 | 60 | 0.5 | 1.7 | SW 8260B |
| 1,2,4-Trimethylbenzene | 10 | 96 | 480 | 0.2 | 0.67 | SW 8260B |
| 1,3,5-Trimethylbenzene | 10 | 96 | 480 | 0.2 | 0.67 | SW 8260B |
| Vinyl chloride | 10 | 0.02 | 0.2 | 0.2 | 0.67 | SW 8260B |
| Xylenes | 10 | 1,000 | 10,000 | 0.5 | 1.7 | SW 8260B |
| **Groundwater - Field Parameters** | | | | | | |
| pH | 0.01 units | - | - | - | - | - |
| Conductivity | 10 µmhos/cm | - | - | - | - | - |
| Temperature | 0.5 deg. Celsius | - | - | - | - | - |
| Dissolved Oxygen | 1 ppm | - | - | - | - | - |

Notes:
1. The State Required Quantitation Limits for all VOCs including DCDFM and THF are based on the State of Wisconsin Operation and Maintenance Plan updated and revised in July 2005. The limit of quantification must be 10 ug/L or lower.
2. The Preventive Action Limit and the Enforcement Standard are from the Wisconsin Department of Natural Resources NR 140.10 Table 1.

By: S. Smith
Date: 4/5/06
Checked by: L. Reeves

**Table 3**
**Historical Target Compound Detections**
**Stoughton City Landfill**
**BT² Project #1764**

| Shallow Monitoring Wells | | | | |
|---|---|---|---|---|
| | Current Event Concentration (µg/l) | | Historical Range (µg/l) | |
| Well | DCDFM | THF | DCDFM | THF |
| MW3S | NA | NA | ND | ND |
| MW4S | NA | NA | ND | ND-0.84 |
| MW5S | NA | NA | ND-5.2 | ND |
| MW7S | NA | NA | ND | ND-0.87 |
| MW8S | NA | NA | ND | ND |
| MW9S | 220 | ND | 33-400 | 4.4-22 |
| MW10S | 1.3 | ND | ND-20 | ND-20 |
| MW13S | NA | NA | ND | ND |
| MW14S | 120 | ND | 18-710 | ND-50 |
| MW15S | NA | NA | ND | ND-0.76 |

| Intermediate and Deep Monitoring Wells | | | | |
|---|---|---|---|---|
| | Current Event Concentration (µg/l) | | Historical Range (µg/l) | |
| Well | DCDFM | THF | DCDFM | THF |
| MW3D | ND | 11 | ND | 53-310 |
| MW3B | NA | NA | ND | ND-1.9 |
| MW4D | ND | ND | ND | ND-2.2 |
| MW5D | 6.2 | ND | 0.92-10 | 1.2-4.0 |
| MW7I | ND | ND | ND | ND-1.6 |
| MW7B | NA | NA | ND | ND-1.7 |
| MW8I | ND | ND | ND | 1.3-20 |
| MW8B | NA | NA | ND | ND |
| MW9I | 120 | ND | 12-340 | 5.3-12 |
| MW9B | 16 | ND | 3.1-8.4 | ND-2.4 |
| MW10I | 120 | ND | 91-280 | 4.6-21 |
| MW10D | NA | NA | ND | ND |
| MW13I | 3.3 | 17 | ND-2.0 | 9.2-22 |
| MW13D | NA | NA | ND-0.61 | ND-9.3 |
| MW14I | 210 | 1.3 | 96-590 | ND-2.4 |
| MW14D | NA | NA | ND-1.5 | ND-0.47 |
| MW15I | NA | NA | ND | ND |
| MW15D | NA | NA | ND | ND |

NOTES:
1. DCDFM is dichlorodifluoromethane; THF is tetrahydrofuran.
2. ND = No detections.
3. NA = Not analyzed.
4. DCDFM PAL = 200 µg/l, ES = 1,000 µg/l; THF PAL = 10 µg/l, ES = 50 µg/l.
5. Historical range includes 9 rounds of sampling performed by BT² (8/00, 4/01, 11/01, 4/02, 11/02, 4/03, 11/03, 4/04, 11/04) and two rounds performed by Roy F. Weston in April 1998 and April 1999.
6. Data from Roy F. Weston is summarized on Table 3 of the QAPP submitted September 2000.

By: SS 6/20/05
Checked: JSN 6/30/05

**Table 4**
**Project Schedule**
**Stoughton City Landfill, Stoughton, Wisconsin - BT² Project #1764**

| Bid Item | Task | Anticipated Date | Actual Date |
|---|---|---|---|
| | **Year 1** | | |
| 3 | Bimonthly gas probe monitoring and testing | December 2005 | 12/28/05 |
| 3 | Bimonthly gas probe monitoring and testing | February 2006 | 02/23/06 |
| 9 | QAPP Submittal | March 2006 | 04/05/06 |
| 9 | Health & Safety Plan Submittal | March 2006 | 03/30/06 |
| 3 | Bimonthly gas probe monitoring and testing | April 2006 | |
| 1 | Semiannual Facility Inspection | April 2006 | |
| 4 | Annual Groundwater Monitoring | April 2006 | |
| 6 | Monitoring well sampling purge water containerizing/disposal | April 2006 | |
| 2 | Semiannual Facility Inspection Report | May 2006 | |
| 3 | Bimonthly gas probe monitoring and testing | June 2006 | |
| 5 | Annual Preparation and Submission of the Groundwater Monitoring Report | June 2006 | |
| 7 | Electronic submission of analytical results to GEMS System | June 2006 | |
| 8 | Annual Mowing of the Landfill Cap vegetation | June 2006 | |
| 3 | Bimonthly gas probe monitoring and testing | August 2006 | |
| 1 | Semiannual Facility Inspection | October 2006 | |
| 3 | Bimonthly gas probe monitoring and testing | October 2006 | |
| 2 | Semiannual Facility Inspection Report | November 2006 | |
| 3 | Bimonthly gas probe monitoring and testing | December 2006 | |
| | | | |
| | **Year 2** | | |
| 3 | Bimonthly gas probe monitoring and testing | February 2007 | |
| 3 | Bimonthly gas probe monitoring and testing | April 2007 | |
| 1 | Semiannual Facility Inspection | April 2007 | |
| 4 | Annual Groundwater Monitoring | April 2007 | |
| 6 | Monitoring well sampling purge water containerizing/disposal | April 2007 | |
| 2 | Semiannual Facility Inspection Report | May 2007 | |
| 3 | Bimonthly gas probe monitoring and testing | June 2007 | |
| 5 | Annual Preparation and Submission of the Groundwater Monitoring Report | June 2007 | |
| 7 | Electronic submission of analytical results to GEMS System | June 2007 | |
| 8 | Annual Mowing of the Landfill Cap vegetation | June 2007 | |
| 3 | Bimonthly gas probe monitoring and testing | August 2007 | |
| 1 | Semiannual Facility Inspection | October 2007 | |
| 3 | Bimonthly gas probe monitoring and testing | October 2007 | |
| 2 | Semiannual Facility Inspection Report | November 2007 | |
| | | | |

By: S. Smith
Date: 3/10/06
Checked by: E. Nelson

**Table 5**
**Sample Container, Preservation, and Hold Time Requirements**
**Stoughton City Landfill, Stoughton, Wisconsin - BT² Project #1764**

| Matrix | Analysis | Container | Preservation | Hold Time (Max.) |
|---|---|---|---|---|
| Groundwater | Volatile Organic Compounds | Two 40-ml septum cap vials | HCl to pH <2, cool to 4°C | 14 days |

By: S. Smith
Date: 3/10/06
Checked by: E. Nelson

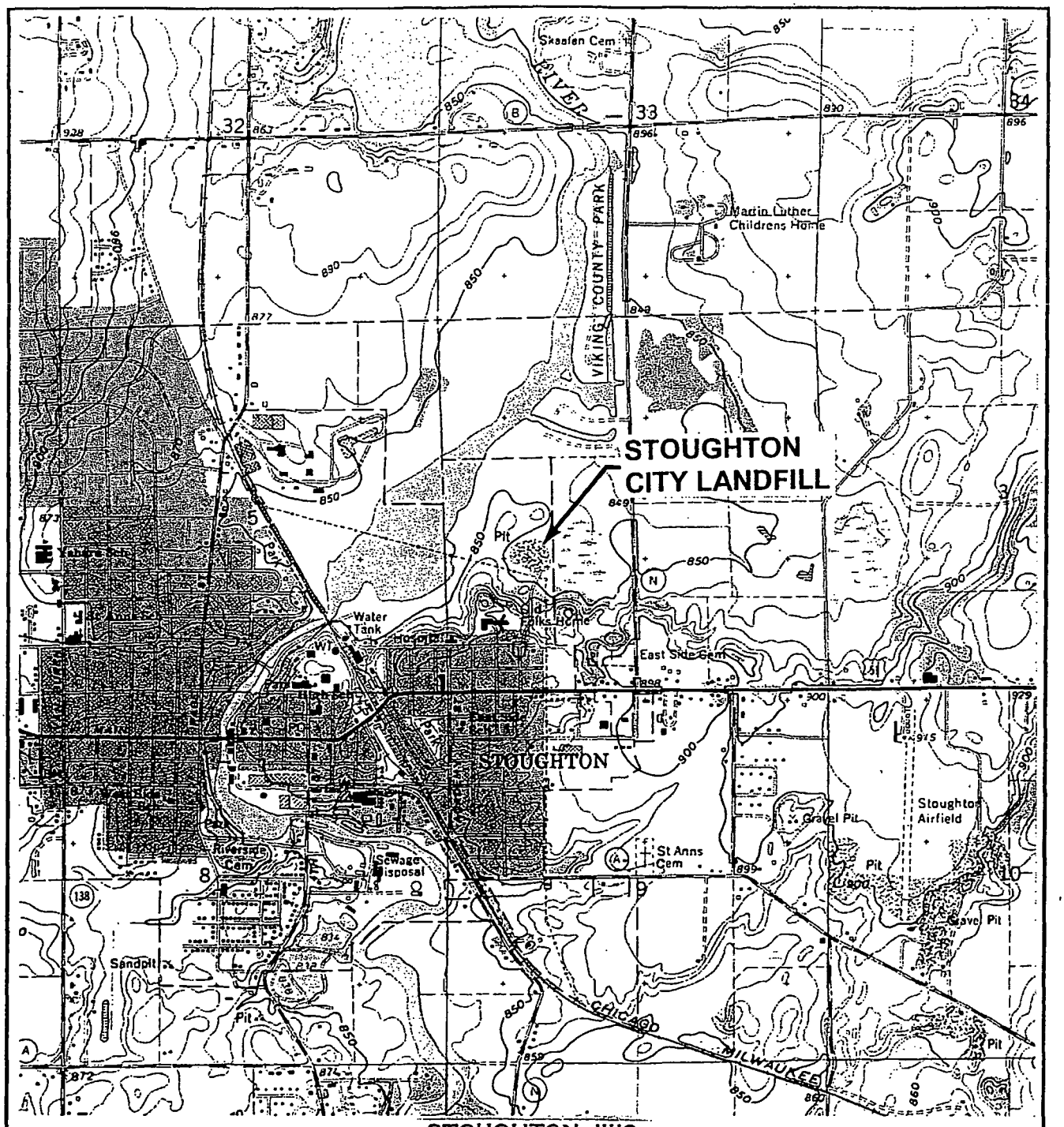
**Table 6**
**Standard Decontamination Protocol for Sampling Equipment**
**Stoughton City Landfill, Stoughton, Wisconsin - BT² Project #1764**

| Step | Procedure |
|---|---|
| 1 | Scrub equipment thoroughly with soft-bristle brushes in a phosphate-free, low-sudsing detergent solution. |
| 2 | Rinse equipment with tap water by submerging and/or spraying. (See note below.) |
| 3 | Rinse equipment with distilled/deionized water until dripping and allow to air dry for 1 to 2 minutes. |
| 4 | Rinse equipment a second time with deionized water by spraying until dripping. |
| 5 | Place equipment on polypropylene or aluminum foil and allow to air-dry for 5 to 10 minutes. |
| 6 | Wrap equipment in polypropylene or aluminum foil for handling and/or storage until next use. |

Note: The decontamination liquids will be managed as described in Section 9. If sampling equipment was used to collect oily or adhesive types of contaminated media, or the presence of organic compound residue is suspected, a rinse via spraying with isopropanol will be included after Step 2. For the Grundfos Submersible pump, pump soapy water through for several minutes followed by pumping tap water for several minutes. Continue at Step 3.

By: S. Smith
Date: 3/10/06
Checked by: E. Nelson

## FIGURES




STOUGHTON, WIS.

NW/4 STOUGHTON 15' QUADRANGLE

N4252.5—W8907.5/7.5

1961

• PHOTOREVISED 1982

DMA 3169 I NW—SERIES V861

UTM GRID AND 1982 MAGNETIC NORTH DECLINATION AT CENTER OF SHEET

WISCONSIN

QUADRANGLE LOCATION

| | |
|---|---|
| PROJECT NO. | 1764 |
| DRAWN BY: | KP |
| CHECKED BY: | SS |
| DRAWN: | 08/02/00 |
| SCALE: | 1" = 2,000' |

FIGURE 1
SITE LOCATION MAP
STOUGHTON CITY LANDFILL
STOUGHTON, WISCONSIN

BT² Inc.

J:\1764\FIG1.DWG

# APPENDIX A

TestAmerica Laboratory Standard Operating Practices

MW-12 CLUSTER LOCATED APPROXIMATELY 1,000' EAST

EAST-WEST 1/4 LINE

APPROX. WETLAND BOUNDARY

EXISTING WETLANDS

APPROXIMATE WETLAND BOUNDARY

CAPPING LIMITS

N02°54'40"W 667.82'

N02°04'20"W 1143.57'

EXISTING WETLANDS

CITY LIMITS

RIP-RAP

24" RCP

AMUNDSON PARKWAY

42" STORM SWR 8/22/00

GV-1

INV. ELEV.= 844.98

MAIN GATE

MW-22S

MW-2D

GMP-1

EX. GARAGE

INV. ELEV.= 845.52

RETAINING WALL

INV. ELEV.= 846.35

36" STORM SWR

INV. ELEV.= 847.07

FIRE HYDRANT

RIP-RAP

GV-11 8/22/00

EXISTING 21" STORM SWR

INV. ELEV.= 850.95

GMP-2

GRAVEL DRIVE

MW-1D

MW-1S

GV-21 8/22/00

EXISTING 15" STORM SWR

INV. ELEV.= 850.80

GMP-3

EXISTING CONDOMINIUM COMPLEX

PROPERTY OWNER: SHALON SUNSET HOME INC 400 NORTH MORRIS STREET STOUGHTON, WI 53589

OVERHEAD ELECTRIC EASEMENT WIDTH NOT DEFINED

EAST GATE

LANDS OWNED BY THE CITY OF STOUGHTON

GV-4 9/29/02

GV-3 8/22/00

GV-2 8/22/00

GV-5 9/25/03

GV-8 9/26/02

GV-6 9/25/03

GV-7 9/25/03

GV-9 9/27/01

GV-10 9/27/01

GV-14 9/26/02

GV-18 9/25/03

GV-13 9/27/01

GV-12 9/27/01

GV-15 9/26/02

GV-17 9/25/03

9/25/03 GV-16

GV-19 9/26/02

GV-20 9/27/01

CP-19

CP-21

CP-1

CP-4

572.63

MW-11D MW-11I MW-11S

MW-6S MW-6D

MW-5S MW-5D

MW-4S MW-4D

MW-14S MW-14D

MW-13S MW-13I MW-13D

MW-10S MW-10I MW-10D

MW-7S MW-7D

MW-8S MW-8D

MW-9S MW-9I MW-9D

MW-15S MW-15I MW-15D

MW-7S MW-7D MW-7I

OW-04 OW-01 OW-03 OW-02 EW-01

LEGEND

- BENCHMARK
- POWER POLE
- SEWER MANHOLE
- WATER VALVE
- CHAIN LINK FENCE
- PROPERTY LINE
- WETLAND BOUNDARY
- SANITARY SEWER LINE
- WATER LINE
- TELEPHONE LINE
- OVERHEAD UTILITY LINE
- CAPPING LIMITS
- TOPSOIL CONTOUR (1' INTERVAL)
- DECIDUOUS TREE
- TREE LINE
- WOOD FENCE
- CHAINLINK FENCE
- GAS VENT RISER
- MONITORING WELL
- FIRE HYDRANT
- CONTROL POINT

NOTES:

1. ORIGINAL MAP BASED ON RDI SURVEYING MAP DRAWN 02/01/99 BY THOMAS A. RADENZ.
2. THE LOCATION OF UNDERGROUND UTILITIES ARE OBTAINED FROM FIELD MEASUREMENTS AND CITY OF STOUGHTON RECORDS - FIELD VERIFY.

ANNUAL GAS VENT WELL SAMPLING

YEAR 1 (2000) GV-1,2,3,11,21
YEAR 2 (2001) GV-8,10,12,13,20
YEAR 3 (2002) GV-4,6,14,15,19
YEAR 4 (2003) GV-5,6,7,16,17,18

130+00 129+00 128+00 127+00 126+00 125+00 124+00 123+00 122+00 121+00 120+00

372+00 373+00

391+00 390+00 389+00 388+00 387+00 386+00 385+00 384+00 383+00 382+00 381+00 380+00 379+00 378+00 377+00 375+00 374+00

60 0 60

SCALE: 1" = 60'

FIGURE 2
SITE MAP
STOUGHTON CITY LANDFILL
STOUGHTON, WISCONSIN

| | |
|---|---|
| PROJECT NO. 1784 | |
| DRAWN BY: RR/PB | |
| CHECKED BY: BB | |
| DRAWN: 08/08/03 | |
| REVISED: 04/04/05 | |

SHEET 1 OF 1

I:\1784\Figures General\1784-2.dwg, 4/5/2005 10:22:48 AM

# STANDARD OPERATING PROCEDURE

TestAmerica
Watertown, WI Division

**Title:** Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry

**SOP No.:** WT05-01.3
**Revision:** 1
**Effective:** 05/03/2004

Page 1 of 22

**Computer File Name:** c:\qc\sop\final\VOCGCMS.doc

| | | | |
|---|---|---|---|
| Division/Lab Manager Approval | 5/3/04 Date | Quality Assurance Approval | 05/03/04 Date |
| Technical Approval | 05/03/04 Date | | |

This method may involve hazardous materials, operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed toe, nonabsorbent shoes are a minimum. For specific hazard(s) see reagents, materials and procedure sections of this SOP.

**Method Reference:**
METHOD #:8260B (SW-846 Third Edition, November 1986)
METHOD # 624

Modifications: SW846 methods explicitly allow modifications to items that are not mandated by the words "shall", "must", or "require". Method 624 allows modifications as long as they meet the performance criteria of Section 8.2 of Method 624.

| Item | Method | Modification |
|---|---|---|
| IS and Surr | 8260B and 624 | Internal Standards and surrogates are chosen that are representative of all compounds analyzed. Internal Standards used are Pentaflurobenzene, 1,4-Difluorobenzene, Chlorobenzene-d5, and 1,4-Dichlorobenzene-d4. Surrogates used are Dibromofluoromethane, Toluene-d8, and Bromofluorobenzene. All are prepared at a concentration of 50 ug/L. |

# STANDARD OPERATING PROCEDURE

TestAmerica
Watertown, WI Division

**Title:** Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry

**SOP No.:** WT05-01.3
**Revision:** 1
**Effective:** 05/03/2004

Page 1 of 22

**Computer File Name:** c:\qc\sop\final\VOCGCMS.doc

| | | | |
|---|---|---|---|
| Division/Lab Manager Approval | Date | Quality Assurance Approval | Date |
| Technical Approval | Date | | |

This method may involve hazardous materials, operations and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed toe, nonabsorbent shoes are a minimum. For specific hazard(s) see reagents, materials and procedure sections of this SOP.

**Method Reference:**
METHOD #:8260B (SW-846 Third Edition, November 1986)
METHOD # 624

Modifications: SW846 methods explicitly allow modifications to items that are not mandated by the words "shall", "must", or "require". Method 624 allows modifications as long as they meet the performance criteria of Section 8.2 of Method 624.

| Item | Method | Modification |
|---|---|---|
| IS and Surr | 8260B and 624 | Internal Standards and surrogates are chosen that are representative of all compounds analyzed. Internal Standards used are Pentaflurobenzene, 1,4-Difluorobenzene, Chlorobenzene-d5, and 1,4-Dichlorobenzene-d4. Surrogates used are Dibromofluoromethane, Toluene-d8, and Bromofluorobenzene. All are prepared at a concentration of 50 ug/L. |
| Preservation | 624 | All analytes preserved with HCl to pH<2 |

| | | |
|---|---|---|
| Storage | 8260B and 624 | The stricter Wisconsin requirement of ≤4°C is maintained. |
| LCS | 8260B and 624 | LCS and CCV are equivalent samples for the analysis of aqueous samples |
| Apparatus | 8260B and 624 | Apparatus are as described in the SOP, which may be different from the method, as is allowed. |

## 1.0 Scope and Application

1.1 This method is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds are reported by this method:

| Compound | CAS No. | Standard List | WDNR LUST | EPA 624 | MDH 465 | Optional List |
|---|---|---|---|---|---|---|
| Acetone | 67-64-1 | | | | | X |
| Acrolein | 107-02-8 | | | | | X |
| Acrylonitrile | 107-13-1 | | | | | X |
| Allyl chloride | 107-05-1 | | | | X | |
| Benzene | 71-43-2 | X | X | X | X | |
| Bromobenzene | 108-86-1 | X | X | | X | |
| Bromochloromethane | 74-97-5 | X | | | X | |
| Bromodichloromethane | 75-27-4 | X | X | X | X | |
| Bromoform | 75-25-2 | X | | X | X | |
| Bromomethane | 74-83-9 | X | | X | X | |
| 2-Butanone (MEK) | 78-93-3 | | | | X | X |
| n-Butylbenzene | 104-51-8 | X | X | | X | |
| sec-Butylbenzene | 135-98-8 | X | X | | X | |
| tert-Butylbenzene | 98-06-6 | X | X | | X | |
| Carbon disulfide | 75-15-0 | | | | | X |
| Carbon tetrachloride | 56-23-5 | X | X | X | X | |
| Chlorobenzene | 108-90-7 | X | X | X | X | |
| Chlorodibromomethane | 124-48-1 | X | X | X | X | |
| Chloroethane | 75-00-3 | X | X | X | X | |
| Chloroform | 67-66-3 | X | X | X | X | |
| Chloromethane | 74-87-3 | X | X | X | X | |
| 2-Chlorotoluene | 95-49-8 | X | X | | X | |
| 4-Chlorotoluene | 106-43-4 | X | X | | X | |
| 1,2-Dibromo-3-chloropropane | 96-12-8 | X | X | | X | |
| 1,2-Dibromoethane | 106-93-4 | X | X | | X | |
| Dibromomethane | 74-95-3 | X | | | X | |
| 1,2-Dichlorobenzene | 95-50-1 | X | X | X | X | |
| 1,3-Dichlorobenzene | 541-73-1 | X | X | X | X | |
| 1,4-Dichlorobenzene | 106-46-7 | X | X | X | X | |
| Dichlorodifluoromethane | 75-71-8 | X | X | | X | |
| 1,1-Dichloroethane | 75-34-3 | X | X | X | X | |
| Compound | CAS No. | Standard | WDNR | EPA | MDH | Optional |

| | | List | LUST | 624 | 465 | List |
|---|---|---|---|---|---|---|
| 1,2-Dichloroethane | 107-06-2 | X | X | X | X | |
| 1,1-Dichloroethene | 75-35-4 | X | X | X | X | |
| cis-1,2-Dichloroethene | 156-59-2 | X | X | | X | |
| trans-1,2-Dichloroethene | 156-60-5 | X | X | X | X | |
| Dichlorofluoromethane | 75-43-4 | | | | X | |
| 1,2-Dichloropropane | 78-87-5 | X | X | X | X | |
| 1,3-Dichloropropane | 142-28-9 | X | X | | X | |
| 2,2-Dichloropropane | 594-20-7 | X | X | | X | |
| 1,1-Dichloropropene | 563-58-6 | X | | | X | |
| cis-1,3-Dichloropropene | 10061-01-5 | X | | X | X | |
| trans-1,3-Dichloropropene | 10061-02-6 | X | | X | X | |
| 2,3-Dichloropropene | 78-88-6 | | | | | X |
| Diethyl ether | 60-29-7 | | | | X | |
| Di-isopropyl ether | 108-20-3 | X | X | | | |
| Ethylbenzene | 100-41-4 | X | X | X | X | |
| Hexachlorobutadiene | 87-68-3 | X | X | | X | |
| Hexane | 110-54-3 | | | | | X |
| Isopropylbenzene | 98-82-8 | X | X | | X | |
| p-Isopropyltoluene | 99-87-6 | X | X | | X | |
| Methylene chloride | 75-09-2 | X | X | X | X | |
| 4-Methyl-2-pentanone (MIBK) | 108-10-1 | | | | X | X |
| Methyl-t-butyl ether | 1634-04-4 | X | X | | X | |
| Naphthalene | 91-20-3 | X | X | | X | |
| n-Propylbenzene | 103-65-1 | X | X | | X | |
| Styrene | 100-42-5 | X | | | X | |
| 1,1,1,2-Tetrachloroethane | 630-20-6 | X | | | X | |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | X | X | X | X | |
| Tetrachloroethene | 127-18-4 | X | X | X | X | |
| Tetrahydrofuran | 109-99-9 | | | | X | X |
| Toluene | 108-88-3 | X | X | X | X | |
| 1,2,3-Trichlorobenzene | 87-61-6 | X | X | | X | |
| 1,2,4-Trichlorobenzene | 120-82-1 | X | X | | X | |
| 1,1,1-Trichloroethane | 71-55-6 | X | X | X | X | |
| 1,1,2-Trichloroethane | 79-00-5 | X | X | X | X | |
| Trichloroethene | 79-01-6 | X | X | X | X | |
| Trichlorofluoromethane | 75-69-4 | X | X | X | X | |
| Trichlorotrifluoroethane | 76-13-1 | | | | X | X |
| 1,2,3-Trichloropropane | 96-18-4 | X | | | X | |
| 1,2,4-Trimethylbenzene | 120-82-1 | X | X | | X | |
| 1,3,5-Trimethylbenzene | 108-67-8 | X | X | | X | |
| Vinyl chloride | 75-01-4 | X | X | X | X | |
| o-Xylene | 95-47-6 | | | | X | X |
| m- & p-Xylenes | | | | | X | X |
| Xylenes, total | 1330-20-7 | X | X | | | |

1.2 There are various techniques by which these compounds may be introduced into the GC/MS system. Purge-and-trap, by Method 5030, is the approved technique for aqueous samples and

methanolic extracts. Closed system Purge-and-Trap and Extraction, by Method 5035, is the approved technique for soil samples that are collected outside of Wisconsin.
1.3 This method can be used to quantitate most volatile organic compounds that have boiling points below 200°C.
1.4 Typical reporting limits are 0.5 ug/L for ground water, 25 ug/kg (wet weight) for methanol preserved samples, 5 ug/kg (wet weight) for Method 5035 soil samples, and 0.5 mg/kg (wet weight) for wastes. Reporting limits are based on the low standard in the calibration curve, and have been justified as being above the MDL. Data will be flagged where the reporting limit is less than the lowest standard in the calibration curve. Achievable limits will be proportionately higher for samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.
1.5 Analysts are advised that flexibility is allowed in the choice of apparatus, reagents, and supplies. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgements necessary to meet the data quality objectives or needs for the intended use of the data.
1.6 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

**2.0 Summary of Method**

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method. The analytes are introduced directly to a wide-bore capillary column. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC).
*NOTE: HP Model 5973 GC/MS does not use wide-bore column.*
2.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.
*NOTE: HP Model 5973 GC/MS does not use jet separator.*

**3.0 Interferences/Comments/Definitions**

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If the laboratory feels that contamination will yield a false positive result for a sample, the laboratory should fully explain this in text accompanying the uncorrected data.
3.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After the analysis of a sample known to contain high concentrations of volatile organic compounds, one or more blanks should be analyzed

to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

3.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil samples. Screening may be accomplished by referring to GRO analyses, where applicable.

3.4 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.5 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water (or methanol for soil samples) and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

3.6 Use of sensitive mass spectrometers to achieve lower detection levels and the requirement to report data to the MDL will increase the potential to detect laboratory contaminants as interferences.

3.7 Definitions are as listed in the Quality Manual, Section 11, and Appendix 5, unless otherwise defined herein.

**4.0 Equipment and Supplies**

4.1 Sample introduction device (1) - The purge-and-trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber. Several complete devices are commercially available.

4.1.1 The recommended purging chamber is designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column.

4.1.2 The traps used are the Supelco K trap for the analysis of aqueous samples, and the Supelco H trap for the analysis of methanol extracts. Before initial use, the trap should be conditioned per manufacturer's instructions. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

4.1.3 The desorber must be capable of rapidly heating the trap to 180°C for desorption. The polymer section of the trap should not be heated higher than 180°C, and the remaining sections should not exceed 220°C during bake-out mode.

4.2 Sample introduction device (2) - The purge-and-trap system consists of a unit that automatically adds surrogates and internal standards to a vial containing the sample, purges the VOCs using an inert gas stream, and also traps the released VOCs for subsequent desorption into the gas chromatograph.

Such systems are commercially available from several sources and shall meet the following specifications.

4.2.1 The purging device should be capable of accepting a 40 mL VOC vial. The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 4.2.2).

4.2.2 The Archon uses the same traps (Supelco K & H) as those described in section 4.1.2.

4.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers are available.

4.3 Injection port liners - A 0.53-mm ID column is mounted 1 cm into the liner from the oven side of the injection port, according to manufacturer's specifications.

4.4 Gas chromatography/mass spectrometer/data system

4.4.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

4.4.1.1 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

4.4.1.2 For some column configurations, the column oven must be cooled to less than 30°C, therefore, a subambient oven controller may be necessary.

4.4.1.3 The capillary column is interfaced through a jet separator.

*NOTE: HP Model 5973 GC/MS does not use a jet separator.*

4.4.2 Gas chromatographic column - 60 m x 0.53 mm ID capillary column coated with DB-624 (J&W Scientific), Rt -502.2 (RESTEK), 3-um film thickness.

4.4.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria in Table 1 when 5-50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

**TABLE 1 BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA**

| m/z | Required Intensity (relative abundance) |
|---|---|
| 50 | 15 to 40% of m/z 95 |
| 75 | 30 to 60% of m/z 95 |
| 95 | Base peak, 100% relative abundance |
| 96 | 5 to 9% of m/z 95 |
| 173 | Less than 2% of m/z 174 |
| 174 | Greater than 50% of m/z 95 |
| 175 | 5 to 9% of m/z 174 |
| 176 | Greater than 95% but less than 101% of m/z 174 |
| 177 | 5 to 9% of m/z 176 |

4.4.4 GC/MS interface - A jet separator is used to interface the GC to the mass spectrometer, including an all-glass transfer line and glass enrichment device or split interface, is used with a 0.53 mm column.

*NOTE: Any enrichment device or transfer line may be used, if all of the performance specifications described in Sec. 8.0 (including acceptable calibration at 50 ng or less) can be achieved. GC/MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass may be deactivated by silanizing with dichlorodimethylsilane.*

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

4.5 Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1,000-uL.

4.6 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

4.7 Syringes - 5-, 10-, or 25-mL, gas-tight with shutoff valve.

4.8 Balance - Analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1 g.

4.9 Glass scintillation vials - 20-mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps.

4.10 Disposable pipets - Pasteur.

**5.0 Reagents and Standards**

The user of the method should read all relevant material safety data sheets (MSDS).

5.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - water in which an interferant is not observed at the method detection limit of the compounds of interest. Organic-free reagent water is prepared by bubbling a contaminant-free inert gas through laboratory DI water for 1 hour prior to its use, and constantly during use.

5.3 Methanol, $CH_3OH$ - Purge and trap grade, demonstrated to be free of analytes. Store apart from other solvents.

5.4 Hydrochloric acid (1:1 v/v), HCl - Carefully add a measured volume of concentrated HCl to an equal volume of organic-free reagent water.

5.5 Sodium Bisulfate, $NaHSO_4$ – ACS reagent grade.

5.6 Stock solutions - Stock solutions are purchased as certified solutions. Transfer the stock standard solutions into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10°C or less. Standards should be returned to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds. Stock solutions msut be recorded in the standard logbook, assigned a unique ID, and be traceable (by way of the unique ID) from any container to any raw data.

5.7 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. The analyst should also handle and store standards as stated in Sec. 5.6 and return them to the freezer as soon as standard

mixing or diluting is completed to prevent the evaporation of volatile target compounds. Secondary dilution standards must be recorded in the standards logbook, and reference the stock solution used for their creation.

5.8 Surrogate and internal standards are prepared in one mix. They are prepared as follows:

5.8.1 Surrogate standards - Surrogates used are toluene-$d_8$, bromofluorobenzene, and dibromofluoromethane. A stock surrogate solution in methanol should be prepared as described above, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 500 ug/10 mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with 5 uL of the surrogate spiking solution prior to analysis.

5.8.2 Internal standards - The internal standards used are 1,4-difluorobenzene, chlorobenzene-$d_5$, 1,4-dichlorobenzene-$d_4$, and pentafluorobenzene. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described in Secs. 5.6 and 5.7. It is recommended that the secondary dilution standard be prepared at a concentration of 50 mg/L of each internal standard compound. Addition of 5 uL of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50 ug/L. Area counts of the internal standard peaks should be between 50-200% of the areas of the target analytes in the mid-point calibration analysis.

5.9 Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/uL of BFB in methanol should be prepared.

5.10 Calibration standards -There are two types of calibration standards used for this method: initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

5.10.1 Initial calibration standards should be prepared at a minimum of five different concentrations from a premixed certified solution. Prepare these solutions in organic-free reagent water. Concentrations should be (in ug/L) 2, 5, 10, 20, 50, 100, and 150. Initial calibration standards should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

5.10.2 Calibration verification standards should be prepared at a concentration near the mid-point of the initial calibration range (50 ug/L) from a premixed certified solution. Prepare these solutions in organic-free reagent water. See Sec. 7.4 for guidance on calibration verification.

5.10.3 It is the intent of EPA that all target analytes for a particular analysis be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

5.10.4 The calibration standards must also contain the internal standards chosen for the analysis.

5.11 Matrix spiking and laboratory control sample (LCS) standards - Matrix spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. In practice, all compounds are spiked, but not all are reported. For purposes of calculating control limits, all analytes must be reviewed in the raw data. The matrix spike samples must be spiked among all potential matrices at a rate of 1 in 20 samples over time. QC samples (blanks, CCVs, LCSs, MS/MSDs) are not included in the count for purposes of maintaining the proper ratio.

5.11.1 The standard should be prepared in methanol, with each compound present at a concentration of 50 ug/1.0 mL.

5.11.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike is used for the LCS.

5.12 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C or less, in amber bottles with PTFE-lined screw-caps. Standards should be used within 6 months of opening.

**6.0 Sample Collection, Preservation, Shipment and Storage**

6.1 Introduction - Once the sample has been collected it must be stored and preserved to maintain the chemical and physical properties that it possessed at the time of collection. The sample type, type of containers and their preparation, possible forms of contamination, and preservation methods are all items which must be thoroughly examined in order to maintain the integrity of the samples. This section highlights considerations that must be addressed in order to maintain a sample's integrity and representativeness. This section is, however, applicable only to trace analyses. Quality Control (QC) requirements need not be met for all compounds presented in the Table of Analytes for the method in use, rather, they must be met for all compounds reported. A result of less than detection limit is considered a quantitative report, and must meet all applicable QC requirements for that compound and method.

6.2 Sample Handling - 40 mL glass screw-cap VOC vials with Teflon lined silicone septa are used for liquid matrices and for solid matrices collected in compliance with Method 5035 protocols. 60 mL VOC vials are used for methanol preserved solid matrices. When collecting the samples, liquids and solids should be introduced into the vials gently to reduce agitation that might drive off volatile compounds. Vials for liquid matrices should be completely filled at the time of sampling, so that when the septum cap is fitted and sealed, and the vial inverted, no headspace is visible. The sample should be hermetically sealed in the vial at the time of sampling, and must not be opened prior to analysis to preserve its integrity.

Due to differing solubility and diffusion properties of gases in liquid matrices at different temperatures, it is possible for the sample to generate some headspace during storage. This headspace will appear in the form of micro bubbles, and should not invalidate a sample for volatiles analysis.

The presence of a macro bubble in a sample vial generally indicates either improper sampling technique or a source of gas evolution within the sample. The latter case is usually accompanied by a buildup of pressure within the vial, (e.g. carbonate-containing samples preserved with acid). Studies conducted by the USEPA indicate bubbles not exceeding 6 mm in diameter did not adversely affect volatiles data. Immediately prior to analysis of liquid samples, the aliquot to be analyzed should be taken from the vial. The sample may be carefully poured into the syringe barrel. Opening a volatile sample to pour a sample into a syringe destroys the validity of the sample for future analysis. Therefore, if there is only one VOC vial, it is strongly recommended that the analyst fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time as the analyst has determined that the first sample has been analyzed properly.

If these guidelines are not followed, the validity of the data generated from the samples is suspect.

Soil can be collected using a variety of appropriate devices. If soil samples are collected for reporting to the WDNR, they must be preserved with methanol at the time of collection. If soil samples are collected in compliance with Method 5035 protocols, take replicate samples and dispense into a vial with Sodium Bilsulfate. One container must be collected for dry weight determination (without methanol). A methanol trip blank must accompany each batch of samples (for each site and each day that samples are collected).

VOC samples may be contaminated by diffusion of volatile organics through the septum during shipment and storage. To monitor possible contamination, a trip blank should be carried throughout the sampling, storage, and shipping process.

6.3 Sample Preservation and Holding Time

| Matrix | Sample Container | Preservation | Storage | Holding Time |
|---|---|---|---|---|
| Aqueous | (2) 40 mL VOC vials | HCl | ≤4°C | 14 days |
| Solids (Wisconsin) | (1) 60 mL VOC vial | Methanol | ≤4°C | 21 days initial analysis<br>28 days for confirmation |
| Solids (5035) | (2) 40 mL VOC vials<br>(1) 40 mL VOC vial | (2) Sodium Bisulfate and<br>(1) Methanol | ≤6°C | 14 days |

Aqueous is equivalent to non-Potable water.

6.4 Cleaning of Glassware - In the analysis of samples containing components in the parts per billion range, the preparation of scrupulously clean glassware is necessary. Failure to do so can lead to a myriad of problems in the interpretation of the final chromatograms due to the presence of extraneous peaks resulting from contamination.

**7.0 Procedure**

7.1 Purge-and-trap sample introduction- All internal standards, surrogates, and matrix spiking compounds must be added to the samples before introduction into the GC/MS. Aqueous and soil/solid samples are purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purged at the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40°C) improves the purging performance of many of the water soluble compounds which have poor purging efficiencies at ambient temperatures.

7.2 Recommended chromatographic conditions

Injector temperature: 175-220°C
Transfer line temperature: 170°C
Carrier gas (He) flow rate: 8-10 mL/min
Initial temperature: 40°C, hold for 4 minutes
Temperature program: 12°C/min to 220°C, hold for 2 minutes

7.3 Initial calibration

Establish the GC/MS operating conditions, using the following as guidance:
Mass range: 35 - 260 amu
Scan time: 0.6 - 2 sec/scan
Source temperature: According to manufacturer's specifications

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 1 for a 5-50 ng injection or purging of bromofluorobenzene (1-uL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, the mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak.

7.3.1.2 Use the BFB mass intensity criteria in Table 1 as tuning acceptance criteria.

*NOTE: All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.*

7.3.2 Set up the sample introduction system. A set of at least five different calibration standards is necessary. Calibration must be performed using the sample introduction technique that will be used for samples. Therefore, develop the standard curve with whichever volume of methanol that will be present in the sample (i.e., none for waters and 100 uL for methanol preserved soils).

7.3.2.1 To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL of each standard to a gas tight syringe along with 5 uL of internal standard. Then transfer the contents to the appropriate device or syringe.

7.3.2.2 The internal standards selected in Sec. 5.8 should permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion.

7.3.3 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. The injection volume will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water.

7.3.4 Tabulate the area response of the characteristic ions against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (Sec. 7.6.2).

7.3.5 System performance check compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (5-point) calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; chlorobenzene; and 1,1,2,2-tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

7.3.5.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

7.3.5.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

7.3.5.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.3.5.4 The minimum mean response factors for the volatile SPCCs are as follows:

| | |
|---|---|
| Chloromethane | 0.10 |
| 1,1-Dichloroethane | 0.10 |
| Bromoform | 0.10 |

| | |
|---|---|
| Chlorobenzene | 0.30 |
| 1,1,2,2-Tetrachloroethane | 0.30 |

7.3.6 Calibration check compounds (CCCs)

7.3.6.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. The CCCs are 1,1-Dichloroethene, Toluene, Chloroform, Ethylbenzene, 1,2-Dichloropropane and Vinyl chloride.

7.3.6.2 Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration.

7.3.6.3 The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all target analytes must meet the 15% criteria for the initial calibration to remain valid.

7.3.6.4 If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before reattempting calibration.

7.3.7 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

7.3.8 Linearity of target analytes

7.3.8.1 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation (Sec. 7.7.2).

7.3.8.2 If the RSD of any target analyte is greater than 15%, either a new calibration curve must be analyzed, or the data for the analyte with an unacceptable RSD must be quantified via linear regression. Linear regression requires a correlation coefficient of at least 0.99 to be valid. When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

7.3.9 Independent Calibration Verification (ICV) – Immediately following the analysis of a calibartion curve, an independent source standard (either from a different manufacturer, or a different lot of the same manufacturer) must be analyzed. Acceptance criteria are 80-120% recovery.

7.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

7.4.1 Prior to the analysis of samples or calibration standards, introduce 1 uL of the 25 ng/uL bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 1 before sample analysis begins. These criteria must be demonstrated at the beginning of each 12-hour shift during which samples are analyzed, prior to the analysis of samples. Tune failure requires instrument maintenance, after which a passing tune must occur prior to any sample analysis.

7.4.2 The initial calibration curve (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration

standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

*NOTE: The BFB and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.*

7.4.3 A method blank should be analyzed to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. The results of the method blank should be:

7.4.3.1 Less than the MDL for the analyte or less than the level of acceptable blank contamination specified in the approved quality assurance project plan.

7.4.3.2 Less than 5% of the regulatory limit associated with an analyte.

7.4.3.3 Or less than 5% of the sample result for the same analyte, whichever is greater.

7.4.3.4 If the method blank results do not meet the acceptance criteria above, then corrective action should be taken to locate and reduce the source of the contamination and to reanalyze any samples associated with the contaminated method blank. If this is not possible, data must be reported with the applicable flag (see Sec. 14).

7.4.4 System Performance Check Compounds (SPCCs)

7.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard must meet its minimum response factor (see Sec. 7.3.5.4). This is the same check that is applied during the initial calibration.

7.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

7.4.5 Calibration Check Compounds (CCCs)

7.4.5.1 After the system performance check is met, the CCCs listed in Sec. 7.3.6 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model.

7.4.5.2 If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift), for any one CCC, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the calibration verification standard prior to the analysis of samples. If the response for the analyte is still not within 20%, then a new initial calibration must be prepared. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion.

7.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

7.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as

required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 GC/MS analysis of samples

7.5.1 It is highly recommended that the sample be screened to minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds. Available options are data from GRO analyses of the same sample or historical data from the same site that is available in LABSYS.

7.5.2 BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

7.5.3 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in Sec 7.2.

7.5.4 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one intact VOC vial remains, the analyst should proceed with caution to protect against possible loss of sample integrity. The remaining sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. A 20-mL, Teflon lined vial should be filled without headspace, labeled, and stored appropriately. If the second aliquot is to be taken from this vial, it must be analyzed within 24 hours.

7.5.5 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL.

7.5.6 The following procedure may be used to dilute aqueous samples. All steps must be performed without delays, until the diluted sample is in a gas-tight syringe.

7.5.6.1 Dilutions are made in gas tight syringes. Intermediate dilution steps may be necessary for extremely large dilutions.

7.5.6.2 Calculate the approximate volume of organic-free reagent water to be added to the syringe, and add slightly less than this quantity of organic-free reagent water to the flask.

7.5.6.3 Inject the appropriate volume of the original sample into the syringe. Dilute the sample to the mark with organic-free reagent water. Repeat above procedure for additional dilutions.

7.5.6.4 Fill the syringe with the diluted sample, as described in Sec. 7.5.5.

7.5.7 Add 5 uL of the solution containing surrogates and internal standards to each sample either manually or by autosampler. The addition of 5 uL of the surrogate spiking solution to 5 mL of aqueous sample will yield a concentration of 50 ug/L of each surrogate standard. The addition of 5 uL of the surrogate spiking solution to 5 g of a non-aqueous sample or 100 uL of a methanolic extract will yield a concentration of 50 ug/kg of each standard. If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute surrogate and internal standard solutions may be required.

7.5.8 Add 5 uL of the matrix spike solution (Sec. 5.11) to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 ug/L of each matrix spike standard.

*NOTE: For the Archon sample introduction device, add 10 uL of a 200 ug/mL standard.*

7.5.8.1 Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix.

7.5.9 Analyze the sample following the procedure in the introduction method of choice.

*NOTE: It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.*

7.5.10 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.

7.5.10.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

7.5.10.2 Where not limited by potential contamination of non-target analytes or other contamination, dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.6 Qualitative analysis

7.6.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

7.6.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.6.1.2 The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

7.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

7.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.6.2 If a client requests the reporting of compounds that are not included in a calibration, a library search may be made for the purpose of tentative identification. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:

7.6.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

7.6.2.2 The relative intensities of the major ions should agree within 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

7.6.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

7.6.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

7.6.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

7.7 Quantitative analysis

7.7.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of that of a given analyte.

7.7.2 If the RSD of a compound's response factors is 15% or less, then the concentration in the extract may be determined using the average response factor from initial calibration data (7.3.6). If the RSD of a compound's response factors is greater than 15%, then the concentration in the extract must be determined using an acceptable linear calibration.

**8.0 Quality Control**

8.1 The laboratory should maintain records to document the quality of the data generated. Examples are as follows through the remainder of this section.

8.2 Quality control procedures necessary to evaluate the GC system operation include evaluation of retention time windows, calibration verification, and chromatographic analysis of samples.

8.2.1 The GC/MS system must be tuned to meet the BFB specifications in Sec. 4.4.3.

8.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

8.2.3 The GC/MS system must meet the SPCC criteria described in Sec. 7.4.4 and the CCC criteria in Sec. 7.4.5, each 12 hours of analysis.

8.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made

8.3.1 The reference samples are prepared from a spiking solution containing each analyte of interest. The reference sample concentrate (spiking solution) may be prepared from pure standard materials, or purchased as certified solutions. If prepared by the laboratory, the reference sample concentrate must be made using stock standards prepared independently from those used for calibration. Prepare a reference sample concentrate in methanol at a concentration such that the spike will provide a concentration in the clean matrix that is 50 ug/L for each analyte in water.

8.3.2 To evaluate the performance of the total analytical process, the reference samples must be handled in exactly the same manner as actual samples. Use a clean matrix for spiking purposes (one that does not have any target or interference compounds), e.g., organic-free reagent water for the aqueous matrix and organic-free sand or soil for the solid matrix.

8.3.3 Prepare the reference sample by adding the appropriate volume of the reference sample concentrate (Sec. 8.3.1) to 100 mL of organic-free reagent water. Transfer this solution immediately to four 5-mL gas-tight syringes.

8.3.4 Analyze at least four replicate aliquots of the well-mixed reference samples. This will include a combination of the sample preparation method and the determinative method.

8.3.5 Calculate the average recovery in ug/L, and the standard deviation of the recovery in ug/L, for each analyte of interest using the four results.

8.3.6 If all analytes of interest meet 80 - 120 percent recovery and <20% RSD, then the system performance is acceptable and analysis of actual samples can begin. If any individual value exceeds the precision limit or any individual value falls outside the range for accuracy, then the system performance may be unacceptable for that analyte. When one or more of the analytes fail at least one of the performance criteria, the analyst should proceed according to Sec. 8.3.6.1 or 8.3.6.2.

*NOTE: The large number of analytes in each of the methods presents a substantial probability that one or more analyte will fail at least one of the performance criteria when all analytes of a given method are determined.*

8.3.6.1 Locate and correct the source of the problem and repeat the test for all analytes of interest, beginning at Sec. 8.3.2.

8.3.6.2 Beginning at Sec. 8.3.2, repeat the test only for those analytes that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning at Sec. 8.3.2.

8.4 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate (matrix spike duplicate is generally analyzed, rather than a sample duplicate), and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.4.1 Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed, a method blank should be analyzed as a

safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

8.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

8.4.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean matrix (Ottawa sand in the case of soils) similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. When analyzing aqueous samples, the calibration verification standard serves the same purpose as the LCS.

8.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory.

8.5.1 If recovery is not within in-house surrogate recovery limits, the following procedures are necessary.

8.5.1.1 Check to be sure that there are no errors in the calculations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas.

8.5.1.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the sample.

8.5.1.3 If no instrument problem is found, the sample should be re-analyzed.

8.5.1.4 If, upon re-analysis (in either 8.5.1.2 or 8.5.1.3), the recovery is again not within limits, report the data as an "estimated concentration." If the recovery is within the limits in the re-analysis, provide the re-analysis data to the data user. If the holding time for the method has expired prior to the re-analysis, provide both the original and re-analysis results to the data user, and note the holding time problem.

8.5.2 The procedures for the calculation of in-house performance criteria for matrix spike recovery and surrogate recovery are provided below. These procedures may also be applied to the development of in-house criteria for the initial demonstration of proficiency and for LCS recoveries.

8.5.2.1 For each matrix spike sample analyzed, calculate the percent recovery of each matrix spike compound added to the sample. For each field sample, calculate the percent recovery of each surrogate.

8.5.2.2 Calculate the average percent recovery (p) and the standard deviation (s) for each of the matrix spike compounds after analysis of 15-20 matrix spike samples of the same matrix. Calculate the average percent recovery (p) and the standard deviation (s) for each of the surrogates after analysis of 15-20 field samples of the same matrix.

8.5.2.3 After the analysis of 15-20 matrix spike samples of a particular matrix (for matrix spike limits) or 15-20 field samples (for surrogate limits), calculate control and warning limits for each matrix spike or surrogate compound. The control limits ($p \pm 3s$) approximate a 99% confidence interval around the mean recovery, while the warning limits ($p \pm 2s$) approximate a 95% confidence interval.

8.5.2.4 Any matrix spike, surrogate, or LCS results outside of the control limits require evaluation by the laboratory. Such actions should begin with a comparison of the results from the samples or matrix spike samples with the LCS results. If the recoveries of the analytes in the LCS are outside of the control limits, then the problem may lie with the application of the extraction and/or cleanup procedures applied to the sample matrix or with the chromatographic procedures. Once the problem has been identified and addressed, corrective action may include the reanalysis of samples, or the extraction and analysis of new sample aliquots, including new matrix spike samples and LCS.
When the LCS results are within the control limits, the problem may either be related to the specific sample matrix or to an inappropriate choice of extraction, cleanup, and determinative methods. If the results are to be used for regulatory compliance monitoring, then the analyst must take steps to demonstrate that the analytes of concern can be determined in the sample matrix at the levels of interest.
See Appendix 4 of the TestAmerica Watertown Quality Manual for additional information.
8.5.2.5 Once established, control limits and warning limits for matrix spike compounds and surrogates should be updated at least semi-annually. The control and warning limits used to evaluate the sample results should be those in place at the time that the sample was analyzed. Once limits are updated, those limits should apply to all subsequent analyses of new samples.
8.5.2.6 For methods and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits should be established using available data or by analogy to similar methods or matrices.
8.5.2.7 Results used to develop acceptance criteria should meet all other QC criteria associated with the determinative method.
8.5.2.8 Laboratories are advised to consider the effects of the spiking concentration on matrix spike performance criteria, and to avoid censoring of data. The acceptance criteria for matrix spike recovery and precision are often a function of the spike concentration used. Therefore, use caution when pooling matrix spike/matrix spike duplicate data for use in establishing acceptance criteria. Not only should the results all be from the same (or very similar) matrix, but the spiking levels should also be approximately the same (within a factor of 2).
8.5.2.9 While professional judgement is important in evaluating data to be used to develop acceptance criteria, do not discard specific results simply because they do not meet one's expectations. Rather, employ a statistical test for outlier values, or at least calculate the acceptance limits both with and without the results that are considered suspect and observe the effect of deleting suspect data. Remember that for a 95% confidence interval, 1 out of every 20 observations likely will still fall outside the limits.
8.5.2.10 In-house QC limits must be examined for reasonableness. It is not EPA's intent to legitimize poor recoveries that are due to the incorrect choice of methods or spiking levels. In-house limits also should be compared with the objectives of specific analyses. It may be useful to compare QC limits generated in the laboratory to the performance data that may be listed in specific determinative methods. However, the analyst must be aware that performance data generated from multiple-laboratory data tend to be significantly wider than those generated from single-laboratory data. In addition, comparisons between in-house limits and those from other sources should generally focus more on the recovery limits of single analyses rather than the precision limits. For example, a mean recovery closer to 100% is generally preferred, even if the ±3 standard

deviation range is slightly wider, because those limits indicate that the result is likely closer to the true value.

8.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

8.7 Control limits are maintained and updated based on input in LABSYS. Current limits are as stated in LABSYS. Any out of control QC data will cause a warning window to appear during data entry.

**9.0 Calculations/Data Reduction and Interpretation**

See Section 12.0, TestAmerica Watertown Quality Manual.

**10.0 Method Performance**

10.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. See Section 11.3 of the Quality Manual for additional information.

10.2 Current MDLs and LOQs for this method are maintained in the LIMS for reporting purposes.

10.3 This method was initially validated by NET Watertown on 11/08/93. That data is available on file.

**11.0 Pollution Control**

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

**12.0 Waste Management**

Laboratory waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WT07-02.0, Waste Disposal.

**13.0 References/Cross References**

13.1 SOP WT05-03 – Method 5035: Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

13.2 Quality Manual – TestAmerica Watertown

13.3 SOP WT07-02 – Waste Disposal

13.4 SOP CP01-01 – Writing a Standard Operating Procedure (SOP)

**14.0 Attachments**

14.1 The following are the flags that could routinely be expected to be used for this analysis. The usages that are listed are not meant to be all-inclusive, they are only to serve as examples.

| FLAG | DESCRIPTION | USAGE |
|---|---|---|
| A | Sample analyzed past holding time | Sample is analyzed past hold time |
| B | Blank is contaminated | Analyte of concern is found in the blank at a level higher than is acceptable in the method |
| C | Standard outside of control limits | Percent recovery of a CCV or LCS is outside of control limits |
| G | Sample received past holding time | Sample is received past hold time |
| H | Late eluting hydrocarbons present | Sample contains hydrocarbons outside of the calculation window |
| I | Improperly handled sample | Sample is not submitted in accordance with method requirements (i.e., overweight for the size container used, submitted in a non-tared container, ...) |
| J | Estimated Concentration | Sample concentration is over calibration or in doubt for some reason (i.e., potential loss in transfer, leakage, ...) |
| L | Common lab solvent and contaminant | Applies to any sample which contains Methylene chloride hits |
| M | Matrix Interference | Sample quantitation is difficult due to matrix interference (i.e., peak shape is not normal, recovery of matrix spike is out of control, ...) |
| P | Improperly preserved sample | Sample is not correctly preserved, whether chemically or by temperature |
| Q | Results confirmed via re-analysis | Sample result is confirmed via re-analysis due to unexpected initial result; both analyses are in agreement with each other |
| S | Sediment present | Sample contains sediment which may interfere with results |
| X | Unidentified compound(s) present | Sample result is due to a small number of unidentified compounds; Non-target compound(s) make analysis at a lesser dilution impossible |
| Z | Internal Standard outside limits | Sample result reported even though internal standard recovery is out of control for this compound |

**15.0 Contigencies**

15.1 Any deviations from this SOP must be brought to the attention of the appropriate Operations Manager, the Laboratory Manager, or the QA Coordinator. This deviation must be documented on a Corrective Action Report and submitted to the QA Coordinator.

15.2 This SOP was written on the basis of existing EPA Methods. It does not follow the section numbering format of SOP CP01-01 – Writing a Standard Operating Procedure, in that Sections 7 and 8 should be reversed per SOP CP01-01, and references to specific EPA Methods are avoided where

ever possible. Also, a section relating to tared sample containers has been added. This addition was at the request of Wisconsin DNR, and does not follow the format described above.

**16.0 Tared Sample Containers**

We have made a label that is applied to all of our 2 and 4 ounce soil jars. That label has spaces for Client Name, Date and Time of sampling, Sample ID, Analysis, and our tracking code. Our tracking code contains the date that the label was printed, a number to track each bottle from that day's lot, and a letter code. The tracking code is automatically generated by computer. No other labels are to be supplied to our clients for these jar sizes. The process of recording the weights is as follows:
16.1 Labels are applied to the jars before they are weighed.
16.2 Weights are recorded (to two decimal places) on the tracking form for the appropriate bottle tracking code.
16.3 The date the bottles are weighed is recorded at the top of the tracking form.
16.4 The coded weight of the bottle is recorded on the label for instant tracking on return to the lab. See the weight codes recorded in the "Soil Jar Tacking" log for further details.
16.5 This process has been deemed acceptable per Rick Mealy, WDNR.

# TestAmerica Analytical - Watertown
## Analytical Method Information

**8260B WI VOCs**

## VOCs by SW8260B

**Water - SW 8260B**

**Sampling Info:**

**Preservation:** Add HCl to pH<2; Store cool at 4°C
**Container:** VOC Vial HCl
**Amount Required:** 3x40ml
**Hold Times:**

**Prep Info:**

**Extraction Method:** SW 5030B
**Initial Units:** ug/L
**Final Units:** ug/L
**Default Initial Amt:** 5.00 mL
**Default Final Amt:** 5.00 mL
**Default Dilution:** 1

| | Analyte | MDL (ug/L) | Reporting Limit (ug/L) | Surrogate %R | Dup RPD | Matrix Spike %R | Matrix Spike RPD | Blank Spike (LCS) %R | Blank Spike (LCS) RPD | RL Date | CL Date |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ☐ | Acetone | 2.0 | 6.6 | | | | 20 | | | 4/1/05 | |
| ☐ | Acrolein | 5.0 | 17 | | | | 20 | | | 4/1/05 | |
| ☐ | Acrylonitrile | 5.0 | 17 | | | | 20 | | | 4/1/05 | |
| ☐ | Allyl chloride | 0.50 | 1.7 | | | | 20 | | | 4/1/05 | |
| ☒ | Benzene | 0.20 | 0.67 | | | 80 - 121 | 11 | | | 4/1/05 | 4/1/05 |
| ☒ | Bromobenzene | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | Bromochloromethane | 0.50 | 1.7 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | Bromodichloromethane | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | Bromoform | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | Bromomethane | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☐ | 2-Butanone (MEK) | 0.50 | 1.7 | | | | 20 | | | 4/1/05 | |
| ☒ | n-Butylbenzene | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | sec-Butylbenzene | 0.25 | 0.83 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | tert-Butylbenzene | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☐ | Carbon disulfide | 0.25 | 0.83 | | | | 20 | | | 4/1/05 | |
| ☒ | Carbon Tetrachloride | 0.50 | 1.7 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | Chlorobenzene | 0.20 | 0.67 | | | 85 - 116 | 9 | | | 4/1/05 | 4/1/05 |
| ☒ | Chlorodibromomethane | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | Chloroethane | 1.0 | 3.3 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | Chloroform | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | Chloromethane | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | 2-Chlorotoluene | 0.50 | 1.7 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | 4-Chlorotoluene | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | 1,2-Dibromo-3-chloropropane | 0.50 | 1.7 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | 1,2-Dibromoethane (EDB) | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | Dibromomethane | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | 1,2-Dichlorobenzene | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | 1,3-Dichlorobenzene | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | 1,4-Dichlorobenzene | 0.20 | 0.67 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | Dichlorodifluoromethane | 0.50 | 1.7 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |
| ☒ | 1,1-Dichloroethane | 0.50 | 1.7 | | | 70 - 130 | 20 | | | 4/1/05 | 4/1/05 |

# TestAmerica Analytical - Watertown

## Analytical Method Information

### 8260B WI VOCs

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| [X] | 1,2-Dichloroethane | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 1,1-Dichloroethene | 0.50 | 1.7 | 72 - 131 | 17 | 4/1/05 | 4/1/05 |
| [X] | cis-1,2-Dichloroethene | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | trans-1,2-Dichloroethene | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 1,2-Dichloropropane | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [ ] | Dichlorofluoromethane | 0.25 | 0.83 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 1,3-Dichloropropane | 0.25 | 0.83 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 2,2-Dichloropropane | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 1,1-Dichloropropene | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | cis-1,3-Dichloropropene | 0.20 | 0.67 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | trans-1,3-Dichloropropene | 0.20 | 0.67 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [ ] | 2,3-Dichloropropene | 0.25 | 0.83 | | 20 | 4/1/05 | |
| [ ] | Diethyl ether | 0.50 | 1.7 | | 20 | 4/1/05 | |
| [X] | Isopropyl Ether | 0.50 | 1.7 | 68 - 128 | 16 | 4/1/05 | 4/1/05 |
| [X] | Ethylbenzene | 0.50 | 1.7 | 83 - 118 | 13 | 4/1/05 | 4/1/05 |
| [X] | Hexachlorobutadiene | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [ ] | Hexane | 1.0 | 3.3 | | 20 | 4/1/05 | |
| [ ] | 2-Hexanone | 0.50 | 1.7 | | 20 | 4/1/05 | |
| [X] | Isopropylbenzene | 0.20 | 0.67 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | p-Isopropyltoluene | 0.20 | 0.67 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | Methylene Chloride | 1.0 | 3.3 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [ ] | 4-Methyl-2-pentanone (MIBK | 0.50 | 1.7 | | 20 | 4/1/05 | |
| [X] | Methyl tert-Butyl Ether | 0.50 | 1.7 | 71 - 127 | 22 | 4/1/05 | 4/1/05 |
| [X] | Naphthalene | 0.25 | 0.83 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | n-Propylbenzene | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | Styrene | 0.20 | 0.67 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 1,1,1,2-Tetrachloroethane | 0.25 | 0.83 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 1,1,2,2-Tetrachloroethane | 0.20 | 0.67 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | Tetrachloroethene | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [ ] | Tetrahydrofuran | 0.50 | 1.7 | | 20 | 4/1/05 | |
| [X] | Toluene | 0.20 | 0.67 | 82 - 116 | 11 | 4/1/05 | 4/1/05 |
| [X] | 1,2,3-Trichlorobenzene | 0.25 | 0.83 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 1,2,4-Trichlorobenzene | 0.25 | 0.83 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 1,1,1-Trichloroethane | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 1,1,2-Trichloroethane | 0.25 | 0.83 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | Trichloroethene | 0.20 | 0.67 | 80 - 117 | 13 | 4/1/05 | 4/1/05 |
| [X] | Trichlorofluoromethane | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | 1,2,3-Trichloropropane | 0.50 | 1.7 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [ ] | 1,1,2-Trichlorotrifluoroethane | 1.0 | 3.3 | | 20 | 4/1/05 | |
| [X] | 1,2,4-Trimethylbenzene | 0.20 | 0.67 | 80 - 122 | 14 | 4/1/05 | 4/1/05 |
| [X] | 1,3,5-Trimethylbenzene | 0.20 | 0.67 | 83 - 122 | 12 | 4/1/05 | 4/1/05 |
| [ ] | Vinyl Acetate | 0.50 | 1.7 | | 20 | 4/1/05 | |
| [X] | Vinyl chloride | 0.20 | 0.67 | 70 - 130 | 20 | 4/1/05 | 4/1/05 |

# TestAmerica Analytical - Watertown

**Analytical Method Information**

## 8260B WI VOCs

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| [X] | Xylenes, Total | 0.50 | 1.7 | | 84 - 119 | 12 | 4/1/05 | 4/1/05 |
| [ ] | m,p-Xylene | 0.25 | 0.83 | | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [ ] | o-Xylene | 0.50 | 1.7 | | 70 - 130 | 20 | 4/1/05 | 4/1/05 |
| [X] | Surr: Dibromofluoromethane | | | 89 - 119 | | | 4/1/05 | 4/1/05 |
| [X] | Surr: Toluene-d8 | | | 91 - 109 | | | 4/1/05 | 4/1/05 |
| [X] | Surr: 4-Bromofluorobenzene | | | 89 - 114 | | | 4/1/05 | 4/1/05 |
| [ ] | Pentafluorobenzene | | | | | | 1/1/80 | |
| [ ] | 1,4-Difluorobenzene | | | | | | 1/1/80 | |
| [ ] | Chlorobenzene-d5 | | | | | | 1/1/80 | |
| [ ] | 1,4-Dichlorobenzene-d4 | | | | | | 1/1/80 | |

# APPENDIX B

$BT^2$ Standard Procedures

# Appendix B
# BT² Standard Procedures

## TABLE OF CONTENTS

*This page intentionally blank.*

# 1.0 LABELING OF MONITORING WELLS, BORINGS, AND OTHER SAMPLING AND REFERENCE POINTS

## 1.1. Scope

This standard operating procedure (SOP) describes standard procedures for labeling monitoring wells, soil borings, and other common sampling and reference points.

## 1.2 Background

The objectives of standardizing the designation of sampling and reference points are:

- To provide consistency in documentation of field sampling and reference points.
- To communicate information about the type of sampling point by using standard designations that represents the general types of sampling points.
- To avoid confusion resulting from non-unique, vague, or misleading labeling of sampling points.

The purpose of the standard procedure is to provide a labeling scheme for common sampling and reference points, and serve as the basis for devising labeling schemes needed for complex or unusual sites, or for sites on which sampling and reference points have previously been designated by site owners or other consultants.

## 1.3 Personnel Training and Qualifications

For labeling sampling and reference points, the following personnel descriptions shall apply:

| | |
|---|---|
| Technical Expert: | A technical staff member, generally the technical coordinator for the project, with significant training and experience in the technical area in which the sampling or reference point is used. Technical experts will typically be senior engineers, senior scientists, senior field technicians, or surveyors. |

Reviewer: The reviewer of the labeling scheme will be the project manager who should be aware of pre-existing sampling and reference points, and the potential for creation of additional sampling and reference points for the project in the future.

## 1.4 Procedures and Documentation

### *1.4.1 General Guidelines*

When developing a labeling scheme the following general guidelines apply:

#### *1.4.1.1 Numbering*

A number is to be used to identify a location at a site. Generally, a location has a radius of about 10 feet. The following is an example.

> A monitoring well, installed within 10 feet of a previously installed boring, B**2**, is designated MW**2**. A piezometer, installed within 10 feet of the monitoring well, is designated MW**2**P. A test pit excavated at the location is designated TP**2**.
>
> Numbering at a site should begin at **1** unless sampling and reference points have already been established at the site. To avoid nonspecific numbering at a site when you are unsure of what numbers have been already used, or if sampling or reference points designated for purposes other than yours exist at the site, start with number 100 or 200. The following are examples.
>
> > At an existing landfill, monitoring wells were designated MW1 through MW135, with some ambiguity if wells with numbers greater than 135 had previously existed at the site. The new series of wells installed for the landfill expansion were designated MW201 through MW212.
> >
> > At an industrial site, an ongoing geotechnical investigation is using a labeling scheme of B1 through B55. The environmental investigation used a designation system starting with B201 to avoid possible overlap with the geotechnical borings, they might eventually exceed 100 in number.

At a petroleum release site, a monitoring well, MW7, was located in the road right-of-way and was part of the monitoring network for an adjacent petroleum release investigation. Access to MW7 could not be obtained, so a well was installed within 10 feet of MW7 and was designated MW7BT (BT for BT²).

*1.4.1.2 Replacements*

If a sampling or reference point is removed and then replaced within 10 feet of the original point, it is designated with an **R**. The following is an example.

MW2**R** is a replacement well installed within 10 feet of the original MW2, which was removed.

*1.4.1.3 Unsuccessful Installations*

If the installation at a sampling point is unsuccessful, the unsuccessful attempts at the location should be designated with an **X**. The following is an example.

A boring could not be advanced to its target depth because of refusal on boulders. Two attempts were made within 10 feet of the original location before the boring was advanced to its target depth. The unsuccessful borings were designated B14**X**, and B14**XX**. The successful boring was designated B14.

*1.4.2 General Designations*

The following designations should be used unless site activities require a unique labeling scheme.

*1.4.2.1 Borings*

| | |
|---|---|
| **B** | A boring not converted to a permanent sampling point. Example **B2**. |
| **GB** | A boring installed using direct-push technology and not converted to a permanent sampling point. Example **GB2**. |
| **HA** | A hand-auger boring not converted to a permanent sampling point. Example **HA2**. |

*1.4.2.2 Wells*

| | |
|---|---|
| **MW** | A monitoring well used to measure water levels and collect groundwater samples for field or laboratory analysis. Constructed such that the water table intersects the screen. Example **MW2**. |

**MW P** A monitoring well used to measure water levels and collect groundwater samples for field or laboratory analysis. Constructed such that the screen is below the water table. Example **MW2P**.

**MW PP** A monitoring well used to measure water levels and collect groundwater samples for field or laboratory analysis. Constructed such that the screen is below the water table and is deeper than the next deepest well. Example **MW2PP**.

**MW Q** A monitoring well used to measure water levels and collect groundwater samples for field or laboratory analysis. Constructed in a perched aquifer. Example **MW2Q**.

**MW T** A temporary well. Examples **MW2T, MW2PT**.

**PZ** Small diameter well used only for measuring water levels. Example **PZ4**.

**EXT** Groundwater extraction well. Example **EXT2**.

**SV** Soil vapor extraction well. Example **SV7**.

**TW** Groundwater pump test well. Example **TW2**.

**PW** Private drinking water supply well. Example **PW13**.

*1.4.2.3 Other Monitoring Well Designations*

Sites with extensive monitoring systems may require monitoring well designations that reflect the site stratigraphy. (Avoid the use of A, B, C designations as these convey little or no information.) Select letters designations that provide stratigraphic information. The following is an example for nested wells at a site with four stratigraphic units.

**MW2SG** Monitoring well installed in the sand and gravel aquifer. The screen intersects the water table.

**MW2D** Monitoring well installed in the dolomite underlying the sand and gravel. The screen is below the water table.

**MW2SS** Monitoring well installed in the sandstone underlying the dolomite.

**MW2PC** Monitoring well installed in the PreCambrian rock underlying the sandstone.

*1.4.2.4 Other Sampling Points*

**TP** Test pit excavated with a backhoe or by hand. Example **TP5.**

**SW** Surface water sampling point. Example **SW5.**

*1.4.2.5 Reference Points*

**SG** Staff gauge, surface water level measuring point. Example **SG5.**

**M** Survey control monument. Example **M5.**

**MH** Manhole. Example **MH7.**

**UTR** Underground storage tank riser. Example UTR5.

*1.4.3 Creation of Numbering System*

Each numbering system shall be prepared by a technical expert in the area to be covered by the sampling or reference points.

*1.4.4 Numbering System Review and Approval*

Before beginning field or office work (workplan preparation, bid specification preparation, etc.) on the project, the numbering system should be approved by the Project Manager.

*1.4.5 Revision of Numbering System or Individual Sampling or Reference Point Designation*

A revision to the numbering system or to an individual sampling or reference point designation must be approved by the Field Team Manager and the Project Manager. After approval is obtained, the change to the numbering system should be documented by a memo to the project team and file. Changes made to an individual sampling or reference point designation must be documented on all existing original forms including field notes, field forms such as boring logs, monitoring well diagrams, etc., and finalized report ready reversion of these and similar form. The changes are to be indicated by drawing a single line through the original designation, then writing the revised designation beneath it, initialing and dating the revision.

1.5 Limitations on Standard Procedure Application

A project may require a labeling scheme for sampling and reference points that serves special need and is not compatible with the general scheme for common sampling and reference points described in the standard procedure.

## 2.0 GROUNDWATER SAMPLE COLLECTION

2.1 Well Construction and Development

- Construct and develop all groundwater-monitoring wells in accordance with 77 IAC Part 920.170 (Monitoring Wells) when working in Illinois or NR 141 when working in Wisconsin.
- Develop wells by bailing them dry, if possible. Develop wells that cannot be bailed dry by alternately surging and purging with a PVC bailer, B-K pump or a Grundfos submersible pump.

Surge and purge each well for 30 minutes, and then purge the well continuously until ten well volumes of water are removed or the water is clear.

## 2.2 Well Purging

- Purge and sample monitoring wells in accordance with Wisconsin Department of Natural Resources (WDNR) guidelines (Groundwater Sampling Field Manual, WDNR Pub #DG-03896, Sect. 2.4.A.6b and Groundwater Sampling Desk Reference, WDNR Pub #DG037).
- Proceed with groundwater sampling from the least contaminated well (based upon observations and field instrument readings during drilling) to the most contaminated well.
- Purge each well immediately prior to sampling using a PVC, Teflon, or a stainless steel bailer attached to a dedicated sampling rope, or a Grundfos submersible pump.
- Measure the volume of water removed from the wells so that if the well cannot be purged dry, three to five volumes of water will be removed.
- Measure the total depth of the groundwater monitoring well and the depth to the groundwater using the methods detailed in **Section 3.0** Water Level and Well Depth Measurements.
- Subtracting the depth to the groundwater from the total depth of the monitoring well will give you the height of the water column within the well.
- The well volumes can be determined using the following conversion factors:
  - Each foot of water in a 2-inch diameter well equals 0.16 gallons
  - Each foot of water in a 4-inch diameter well equals 0.66 gallons
  - Each foot of water in a 6-inch diameter well equals 1.5 gallons
- Multiply the well volume based on the height of the water column by three. Measure the volume of water removed from the well. Pump or bail water from the well until three to five well volumes have been removed.

## 2.3 Low Flow Groundwater Sampling

### *2.3.1 Scope*

The following SOP describes the procedures for collection of representative groundwater samples using low flow sampling (Groundwater Sampling Field Manual, WDNR Pub #DG-03896, Sect. 2.5).

### *2.3.2 Procedure*

The following procedures will be followed when collecting groundwater samples using low flow methods:

1. Determine the order of sampling. This should be determined before fieldwork commences. The order in which monitoring wells are purged and sampled should proceed from the cleanest wells to the most contaminated wells. When no historical water quality data are available, sample background wells will be sampled first followed by the furthest downgradient wells and then wells most likely to be significantly contaminated.

2. Determine the following field information before work commences:

    a. Measured well depth, if possible. If not previously measured then determine by subtracting the distance between ground surface and top-of-casing (stick up) and add this distance to the installation screen depth.
    b. Screen length.
    c. Determine the depth to bottom of screen and depth to top of screen from top of casing. From this information, determine the depth to the midpoint of the well screen.

3. Note the condition of the monitoring well and verify the correct well to be sampled. Additional information may be required for documentation before, during, and after groundwater sampling.

4. Determine the static water level. Record in the field notebook. Minimize disturbances of the stagnant water column during groundwater level measurement.

5. Water levels are measured prior to and during a groundwater-sampling event for the following reasons:

    a. To assess whether the static water column length is sufficient to allow purging and sampling to proceed in the normal manner provided that draw down is moderate.
    b. To select the depth to which the pump intake or other purging or sampling device should be lowered.
    c. To monitor the water levels during purging and sampling and determine the optimum pumping rate minimizing draw down.
    d. To determine groundwater flow directions.

Unless stated in the workplan, groundwater from monitoring wells containing free product will not be sampled. If the groundwater has to be sampled, disposal equipment will be utilized.

6. Reusable sampling equipment (pumps, etc.) will be rinsed with deionized water prior to inserting the equipment into the monitoring well.

7. Calibrate field parameter measuring equipment, if required. Otherwise, check standard and record the measurement.

8. Note the depth to the top and bottom of the well screen (if known) from top-of-casing. Depth of the well should not be measured prior to purging and this may cause resuspension of settled solids from the formation and require longer purging times for turbidity equilibration. Measure the well depth after sample collection. Compare the static water level to the depth to the top of the screen. If the water level is above the screen, insert pump intake to the middles or slightly above the middle of the screened interval. Placement of the pump too close to the bottom of the well will cause entrainment of solids collected in the well over time. If the water level is across the well screen, place the pump at the top of the water column.

9. Slowly insert sampler into the well to the desired depth and begin to purge at a rate (0.1 - 0.5 l/min or 0.026 - 0.13 gpm or 100 ml/min-500 ml/min) to minimize draw down (<0.1 m or <0.33 ft.). Monitor draw down during purging using an electric tape. Make the proper adjustments to stabilize the flow rates as soon as possible. During purging of well screened in low-permeability formations (<0.1 l/min recharge), lowering of the water level causes cascading of water into the well if the purged rate is greater than the recovery rate of the well. The cascading of water into the well can accelerate alteration of the water. Cascading should be kept to a minimum by not drawing the water level in the well down below the top of the screen. If the water level is already at the top or within the well screen, then a purging rate that results in minimum draw down while allowing the well to be purged in a reasonable length of time should be selected. If the sampling team knows that pumping the well at the lowest feasible rate will pump the well dry, pump the well dry recording the field parameters during pumping. Allow the well to recover, and measure and record the field parameters. Allow the well to recover again, to provide sufficient water to completely fill the appropriate sample containers, and collect the sample. Passive sample collection is an alternative method of sampling low-permeability formations. Passive sample collection requires insertion of the device into the screened interval for a sufficient time period to allow flow and sample equilibration before extraction for analysis. The limitation of this technique is satisfying sampling volume requirements.

10. Record all field parameters (pH, temperature, specific conductance, turbidity) after stabilization.

11. After field parameters have been stabilized, collect a sample. The data, as well as time of sample collection, are recorded in the field notebook or field data sheets. See the workplan for other types of field parameters to measure and record.

If the monitoring well is sampled repeatedly (quarterly, annually, etc.) for assessment of the temporal variations in water quality with time, it is necessary to set the pump to the same depth, purge at approximately the same rate, and purge the same volume of water during each subsequent sampling event. Thus, the purging criteria for each individual well are set during the first round of a monitoring program. If the same purging criteria do not result in stabilization in subsequent sampling events, then consider the following:

a. Groundwater chemistry has changed over time.
b. The monitoring well may need rehabilitation (redevelopment, replaced, etc.).
c. Errors in field measurements may have been made during one or more sampling events.
d. Collect a set of samples at the normal purging time and also collect time-series samples to compare with changes in field parameters.

It may not be possible in certain situations to reach stabilization due to:

a. on-uniform distribution of chemical and physical parameters in the water yielding zone(s) being monitored.
b. Previously undetected coalescing plumes.
c. Multiple water-yielding zones screened by the monitoring well(s).
d. Leaky confining layers, perched zones, etc., nearby.

If stabilization is not reached, it is important to record that stabilization was not achieved. This does not mean the sampling event was a failure. In fact, the information could be quite accurate and valuable.

12. Samples for various analyses will be collected in the following order:

a. Field parameters
b. Volatile organics
c. Inorganic parameters

2.4 Sample Collection, Preparation, Handling, and Preservation

*2.4.1 General*

- After well purging has been completed, collect samples using a low flow sampling pump.
- Place groundwater samples in a sample container appropriate for the analytical method.
- Place all samples on ice for storage and shipping at approximate 4° Fahrenheit.

*2.4.2 Volatiles*

- Samples for volatile organic compound (VOC) analysis will be collected first.
- Gently fill a tilted 40-milliliter sample VOC preservation vial (preserved with HCl) with as little turbulence as possible.
- Place the Teflon-coated silicone septum carefully into place and screw cap on firmly.
- Invert the vial to check for air bubbles. If any are present, remove cap and refill to the top until a sample is obtained with no trapped air.

## 3.0 WATER LEVEL AND WELL DEPTH MEASUREMENTS

- Open all wells and allow water levels to equilibrate before measure depths to water. Measure water levels several times at 10- to 15-minute intervals to ensure that the water levels have stabilized (Groundwater Sampling Field Manual, WDNR Pub #DG-03896, Sect. 3.3).
- Measure and record the depth to water and depth to the bottom of the well using an electric water level indicator tape.
- Measure the depth to water at least three times to ensure accuracy and precision of measurement.

## 4.0 GROUNDWATER PH, CONDUCTIVITY, AND TEMPERATURE MEASUREMENTS

4.1 Scope

The following procedure outlines the techniques used for the accurate field measurement of pH, specific conductance, and temperature using appropriate meters and electrodes.

4.2 Equipment

The following equipment will be utilized by field personnel during measurement activities:

- pH meter
- pH buffer solution
- Probe preservation solution
- Deionized water
- Conductance meter
- Conductance standards
- Temperature probe
- Temperature simulator
- Field notebook or field data sheets

4.3 Procedures

*4.3.1 Field Measurement of pH /Conductivity/Temperature by Electrode*

Electrode Preparation

All field analytical meters require a pre-field inspection to insure that the equipment components are complete and in proper working order.

Oakton pH/Conductivity 10 Meter

Remove battery after use.

1) Use fresh buffers; check battery before fieldwork; store probe with #4 buffer in boot.

2) pH Calibration:
   A. Connect 6-pin probe to meter: Line up notch, push in and turn the locking ring into place.
   B. Wet the probe in tap water for 10 minutes.
   C. POWER ON (BAT indicates replace battery).
   D. Press "Mode" to select pH.
   E. Place probe into #7 pH buffer, stir gently. Wait until "Ready" is displayed.
   F. Press "Cal/Meas". Primary display indicates measured reading. Secondary display indicates the standard buffer solution.
   G. Wait until "Ready" is displayed.

H. Press "Enter". Display flashes "Con"; secondary display automatically scrolls to the next buffer calibration option.

I. Use scroll up/down to select the next buffer value in the secondary display.

J. Rinse probe, place into next buffer solution.

K. Wait until "Ready" is displayed. Press "Enter". Turn meter off.

L. Meter is now calibrated.

3) Conductivity Calibration

A. Press "Mode" to select conductivity.

B. Rinse probe with tap water, immerse probe into calibration standard (1413uS). Gently tap probe with hand to remove air bubbles.

C. Wait until "Ready" is displayed.

D. Press "Cal/Meas".

E. Scroll up/down to display calibration standard value.

F. Press "Enter". The meter is now calibrated and displays readings corrected to $25^0$ C.

4) Temperature Calibration

A. Press "Mode" to select pH measurement mode.

B. Press "Cal/Meas" to enter pH calibration mode. The "CAL" indicator will appear above the primary display.

C. While in pH calibration mode, press "Mode" to enter the temperature calibration mode. The primary display shows the temperature reading with zero offset and the secondary display shows the initial temperature value.

D. Compare the primary display reading with a NIST-traceable thermometer or another thermometer known to be accurate.

E. Scroll the up/down to adjust the primary display reading to agree with the temperature standard.

F. Press "Enter" to confirm temperature calibration. The meter is now calibrated for temperature.

*4.3.2 Field Measurement of Turbidity*

Per the WDNR Groundwater Sampling Field Plan, Section 2.6, we will measure turbidity by visual description (e.g., slight, moderate, heavy, none).

## 5.0 SAMPLE COLLECTION DOCUMENTATION

- Record field observations and measurements on field record forms. Record information concerning field activities and conditions directly and legibly in the field logbooks in ink. If an entry must be changed, the change will not obscure the original entry. Document the date, weather conditions, site activities, and personnel on site including visitors in the logbook.
- Record sample time, sample location, sample interval depth, sample number, and sample preservation method in field notebook. Identify soil samples by the sampling location and sample depth. For example, a soil sample from soil boring number B3 collected from a depth interval of 7 to 9 feet will be designated as B3 7-9 feet. Identify field samples with sample labels that list the date, sample identification, and BT², Inc. project number.
- Prepare COC forms that include sample number, sampling procedures, analysis required, the signature of the sampler, type of sample (grab or composite), number of containers, and signature blocks for all who handle the sample (with the exception of shipping personnel).

## 6.0 AIR MONITORING FOR VOLATILE ORGANIC CHEMICALS

### 6.1 Photo-Ionization Detector (PID)

A PID will be used to monitor the concentration of volatile organic chemicals in the ambient air and in the passive gas vents.

- Unplug PID in office, pack up in case, and proceed to field site
- Calibrate PID
  - Insert RUN key
  - Turn PID on
  - Press "mode/store"
  - Press "-/CSR"
  - Press "-/CSR"
  - Press "-/CSR"
  - Press "-/CSR" display will say "reset to calibrate"
  - Press "reset"
  - Press "-\CSR" display will read "zero gas reset when ready"
  - Press "reset" display will read "580 zeroing" then "span=0100"

- Press "+" display will read "span gas when ready"
- Attach 100 ppm gas to PID
- Press "reset" display will read "calibrating" then "reset to calibrate"
- DO NOT PRESS RESET
- Unplug the RUN key
- Plug the RUN key back in
- Turn PID on
- Record calibrated reading and background range

- Hold PID inlet pipe in the gas vent port until a stable reading is obtained.
- Record reading on the appropriate field form and include in the Annual Groundwater Monitoring Report.

6.2 GEM2000 Landfill Gas Meter

The following section is designed to be a rapid run through the operation of the unit, and does not detail all possible screen displays and situations that can arise. In particular, it assumes that the unit will be in a factory supplied state, with no stored readings and no site and boreholes identifiers set. Please refer to the reference section below for more complete information.

*Turning the Instrument On*

Press the red On/Off key. A long beep will sound, followed by the manufacturer's logo displayed. The instrument will then perform a predetermined self-test taking approximately 20 seconds.

Calibration Procedures

This SOP is a brief summary for start-up, field calibration, and general monitoring requirements. Review the operations manual (located in instrument case) prior to taking the meter out into the field. Refer to the GEM2000 operations manual for details in which predetermined settings are needed or if you have miscellaneous questions regarding instrument settings, functions maintenance, etc.

- Connect the sample inlet hose and pressure impact hose to the instrument. Connect the clear sample inlet hose with the water trap filter closest to the instrument in the upper of the two available ports at the right side of the meter. The blue colored pressure port hose is installed the lower port.

- In the gas display screen select #1 key to bring up the main menu. Scroll down to "Mode of Operation" and select the enter/store key below the on/off key to select. Two choices must then be picked from, "Landfill Gas Analyser" and "Gas Extraction Monitor." If analyzing only $CH_4$, $CO_2$, and $O_2$ select the "Landfill Gas Analyser" (i.e. for sampling a gas probe). If sampling a gas extraction well or gas probe in which vacuum or pressure readings are needed select the "Gas Extraction Monitor".

- **Field Calibration**: Select the menu or #1 key while in the gas display screen then scroll down to "Field Calibration" and press the enter key. In the "Check Calibration" screen there will be set span gas concentrations. Set the span concentrations if the available calibration gas components are different. To change the span concentrations press the #3 key "Edit target concentrations" and then edit based upon calibration gas components. If the calibration gas does not contain a specific gas re-enter the previous span concentration. Current cylinders contain 2.5% $CH_4$ balance $O_2$, and 50% $CH_4$, 35% $CO_2$, N2 balance. Enter 20.8% for $O_2$. Once appropriate span gas concentrations chosen connect the cylinder/regulator to the inlet hose. REGULATOR ONLY NEEDS TO BE SLIGHTLY OPEN, DO NOT OPEN BEYOND 10%. Allow span gas approximately 1 to 2 minutes to flow into instrument. Readings in the calibration screen will change as gas first enters instrument sensors. Once the readings have stabilized press the enter key to access "Calibration Menu". If residual methane is being displayed in the "R" or reading row in the calibration screen, select "Zero Channel(s) press enter then select "Zero CH4". Next select "Span Channel(s) in the "Calibration Menu" and select the desired span gas concentration (i.e. "Span $CH_4$ @ 2.5%"). ONLY SELECT THE SPAN CONCENTRATION FOR THE CALBIRATION GAS COMPONENETS IN WHICH YOU ARE CURRENTLY READING. IGNORE THOSE WHICH ARE NOT COMPONENTS OF THE CALIBRATION GAS CYLINDER. Following this sequence turn off the calibration gas cylinder and remove inlet hose from regulator. To field calibrate the $O_2$ while in the "Check Calibration" screen press the pump key and allow the $O_2$ reading to stabilize while sampling CLEAN AMBIENT AIR. Press the enter key to access the "Calibration Menu" (pump will automatically turn off) select "Span Channel(s)" press the enter key then select "Span $O_2$ @ 20.8%. When complete press the #1 key to exit calibration menu and begin sampling.

If analyzing probes with concentrations below 10% $CH_4$ use the 2.5% $CH_4$ by volume (50% LEL). If analyzing gas extraction wells or probes with $CH_4$ concentrations greater than 10% use the 50% $CH_4$ by volume calibration cylinder to calibrate the instrument. The instrument can be

calibrated using the higher methane cal gas (50% $CH_4$) when testing gas probes as long as the instrument is checked against the lower calibration gas (2.5% $CH_4$).

Periodically check readings against calibration gas. $O_2$ may have to be calibrated more often due to temperature fluctuations and drift.

If measuring well pressure or flow always zero out transducers prior to connecting hoses to monitoring point to ensure an accurate reading.

*Use of Standard Test Gases*

The calibration of the GEM2000 can be checked against calibration check gases purchased by the user at any time. Care should be taken to NEVER EXCEED PRESSURES OF 300 MBAR when injecting calibration gases into the GEM2000. Use a proprietary pressure regulator or similar. NEVER attempt to flow gas in the REVERSE flow direction as the pump and the oxygen cell would be destroyed.

*Quality Assurance Procedure*

To document calibration checks it is suggested 'dummy' site and borehole names are used to store the gas values displayed during a calibration check. These results can then be printed and filed with the site data as part of an in-house quality control procedure.