SCS ENGINEERS

Quality Assurance Project Plan
Operation and Maintenance

Stoughton City Landfill
Stoughton, Wisconsin

Prepared for:

Wisconsin Department of Natural Resources
101 South Webster Street
Madison, Wisconsin 53703

Prepared by:

SCS ENGINEERS
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(608) 224-2830

March 2016, Revision 2
File No. 25216022

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SIGNATURES

Quality Assurance Project Plan
For Operation and Maintenance at
Stoughton City Landfill

Revision 2
March 2016

Prepared by: SCS Engineers

Leslie Busse Leslie A. Busse, SCS Project Manager	3/31/16 Date
 Sherren Clark, SCS Quality Assurance/ Senior Review Officer	3-30-16 Date
Steven B. Smith Steven B. Smith, SCS Field Team Manager	3/31/16 Date
 Gary A Edelstein, WDNR Project Manager	 Date
 WDNR Quality Assurance Reviewer	 Date

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 5 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 storm water 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, photoionization 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 SCS'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 photoionization 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, SCS, 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.

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

The SCS Project Manager will coordinate with the SCS Quality Assurance/Senior Review Officer, the SCS 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 SCS Project Manager is Leslie Busse.

SCS Quality Assurance/Senior Review Officer
The SCS Quality Assurance/Senior Review Officer will be responsible for data assessment and overall quality of the O&M phase of this project. The SCS Quality Assurance/Senior Review

Officer will also provide final review and approval of all analytical reports to be submitted to the WDNR. The SCS Quality Assurance/Senior Review Officer is Sherren Clark.

2.2 FIELD RESPONSIBILITIES

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

- Day-to-day coordination with the SCS 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 SCS 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 SCS and WDNR Project Managers, and implement and document corrective actions.
- Prepare drafts of all required reports and site facility inspection logs.

The SCS 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 Sandie Fredrick. The Quality Assurance Manager is Terese Preston.

2.3.1 Laboratory Project Manager

The Laboratory Project Manager will report to the SCS 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 SCS.

2.3.2 Laboratory Quality Assurance Manager

The Laboratory QA Manager has the overall responsibility for data after it leaves the laboratory. The Laboratory QA Manager 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 10 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

This Field Sampling Plan (FSP) 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 5 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, 9B, 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 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.

4.2 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 SCS's standard procedures, which are included as **Appendix B**.

4.2.1 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.

4.2.2 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, ± 0.5oC 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 SCS 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.

4.2.3 Gas Probe Monitoring Procedures

The gas probes will be measured by using the direct reading GEM2000 Landfill Gas Meter and a 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).

4.3 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.

4.4 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.

4.4.1 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.

4.4.2 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.

4.4.3 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 SCS 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
- SCS sample location number.

5.4 FINAL EVIDENCE FILES

Per the O&M Bid Package dated October 2015, 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 5 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 Photoionization Detector (PID)

The PID will be calibrated daily with 100 parts per million (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 SCS. 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 5 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

SCS 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 SCS 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 SCS 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 SCS 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 photoionization 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 SCS Project Manager, or the SCS Field Team Manager. If immediate corrective action is required, approvals secured by telephone from the SCS 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 SCS 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 SCS 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 SCS Project Manager or designee. This manager will be responsible for assessing the suspect problems in consultation with the SCS 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 SCS Field Team Manager.

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

- Evaluating all reported nonconformances
- Controlling additional work on nonconforming items
- Determining disposition or action to be taken
- Maintaining a log of nonconformances
- Reviewing nonconformance reports and corrective actions taken
- Ensuring 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 SCS 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 SCS 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 / SCS Engineers Project #25216022

O&M Task	Sample Matrix	Field Parameters	Laboratory Parameters	Investigative[1]			Field Duplicate			Field Blank			MS/MSD[3]			Motrix Total[4]
				No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
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, 9B, 10S, 10I, 13I, 14S, 14I.
2. Full list VOCs to be analyzed on samples from wells: 9S, 9I, 9B, 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
Revised: 3/20/16
Checked by: L. Busse

I:\25216022.00\Deliverables\QAPP\[Table 1_Summary of Annual O&M Sampling and Analysis Program.xlsx]Table 1

Table 2. Summary of Data Quality Objectives
Stoughton City Landfill, Stoughton, Wisconsin / SCS Engineers Project #25216022

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
Chloroethane	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-Trirnethylbenzene	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 umhos/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 µg/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

I:\25216022.00\Deliverables\QAPP\[Table 2_Summary of Data Quality Objectives..xlsx]Table 2

Table 3. Historical Target Compound Detections
Stoughton City Landfill, Stoughton, Wisconsin / SCS Engineers Project #25216022

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)		Historicol 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 perfomed by BT^2, Inc. (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 in Table 3 of the QAPP submitted September 2000.

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

I:\25216022.00\Deliverables\QAPP\[Table 3_Historical Target Compound Detections.xlsx]Toble 3

Table 4. Project Schedule
Stoughton City Landfill, Stoughton, Wisconsin / SCS Engineers Project #25216022

Bid Item	Task	Anticipated Date	Actual Date
3	Bimonthly gas probe monitoring and testing	February 2016	February 2016
3	Bimonthly gas probe monitoring and testing	April 2016	
9	QAPP Submittal	April 2016	
9	Health & Safety Plan Submittal	March 2016	March 8, 2016
3	Bimonthly gas probe monitoring and testing	June 2016	
1	Semiannual Focility Inspection	April 2016	
4	Annual Groundwater Monitoring	April 2016	
6	Monitoring well sampling purge water containerizing/disposal	April 2016	
2	Semiannual Focility Inspection Report	May 2016	
3	Bimonthly gas probe monitoring and testing	August 2016	
5	Annual Preparation and Submission of the Groundwater Monitoring Report	June 2016	
7	Electronic submission of analytical results to GEMS System	June 2016	
8	Annual Mowing of the Landfill Cap vegetation	August/September 2016	
3	Bimonthly gas probe monitoring and testing	October 2016	
1	Semiannual Facility Inspection	October 2016	
3	Bimonthly gas probe monitoring and testing	November 2016	
2	Semiannual Facility Inspection Report	November 2016	
	Year 2		
3	Bimonthly gas probe monitoring and testing	February 2017	
3	Bimonthly gas probe monitoring and testing	April 2017	
1	Semiannual Facility Inspection	April 2017	
4	Annual Groundwater Monitoring	April 2017	
6	Monitoring well sampling purge water containerizing/disposal	April 2017	
2	Semionnual Facility Inspection Report	May 2017	
3	Bimonthly gas probe monitoring and testing	June 2017	
5	Annual Preparation and Submission of the Groundwater Monitoring Report	June 2017	
7	Electronic submission of analytical results to GEMS System	June 2017	
8	Annual Mowing of the Landfill Cap vegetation	June 2017	
3	Bimonthly gas probe monitoring and testing	August 2017	
1	Semiannual Facility Inspection	October 2017	
3	Bimonthly gas probe monitoring and testing	October 2017	
2	Semiannual Facility Inspection Report	November 2017	

By: S. Smith
Date: 3/10/06
Revised: 3/20/16
Checked by: L. Busse

I:\25216022.00\Deliverables\QAPP\[Table 4_Project Schedule.xlsx]Table 4

Table 5. Sample Container, Preservation, and Hold Time Requirements
Stoughton City Landfill, Stoughton, Wisconsin / SCS Engineers Project #25216022

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

I:\25216022.00\Deliverables\QAPP\[Table 5_Sample Container_Preservation_Hold Time_Requirements.xlsx]Table 5

Table 6. Standard Decontamination Protocol for Sampling Equipment
Stoughton City Landfill, Stoughton, Wisconsin / SCS Engineers Project #25216022

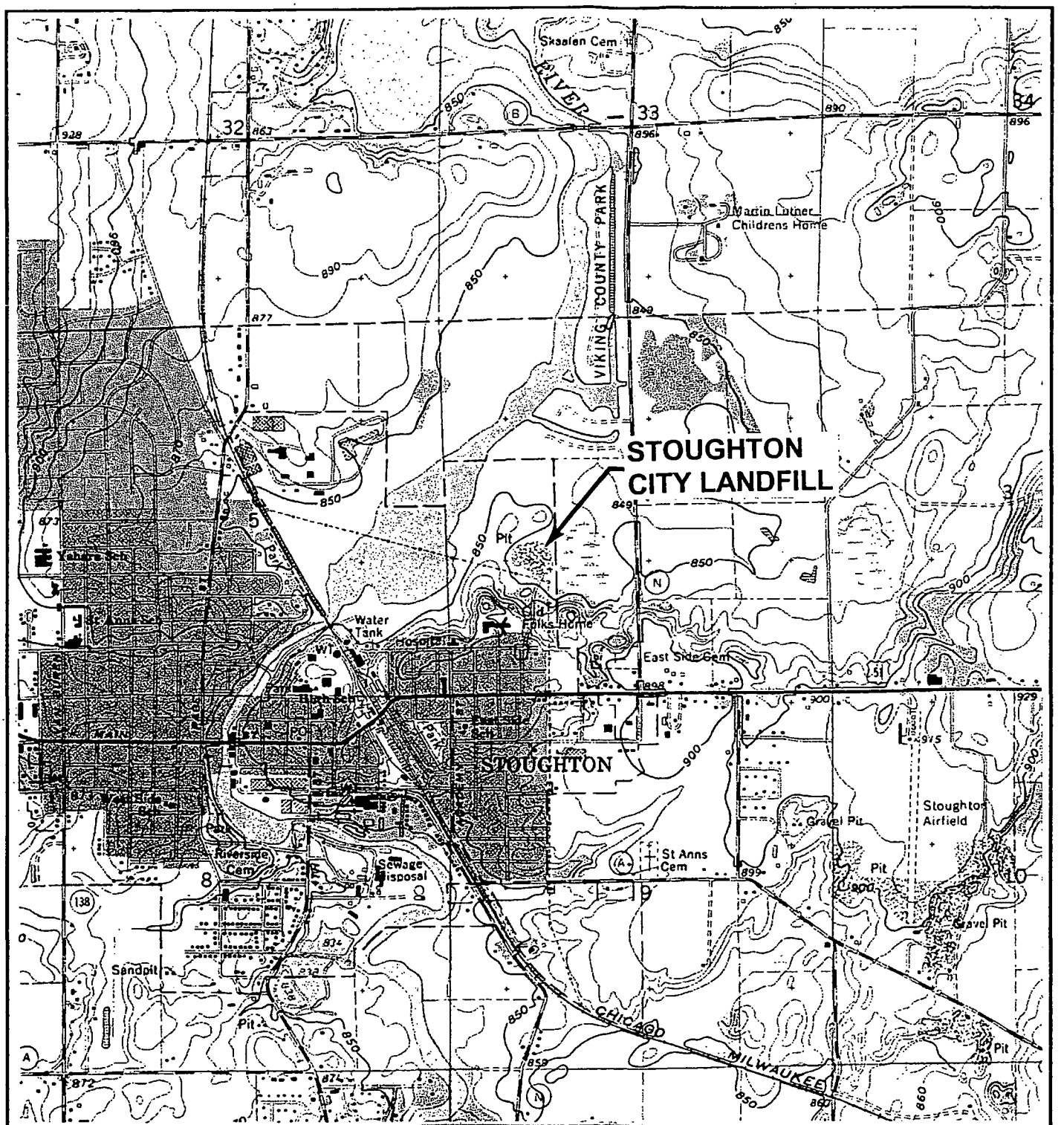
Step	Procedure
1	Scrub equipment thoroughly with soft-bristle brushes in a phosphate-free, low-sudsing detergent solution.
2	Rinse equipment with tap water by submerging and/or spraying. (See note below.)
3	Rinse equipment with distilled/deionized water until dripping and allow to air dry for 1 to 2 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

I:\25216022.00\Deliverables\QAPP\[Table 6_Standard Decontamination Protocol.xlsx]Table 6

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

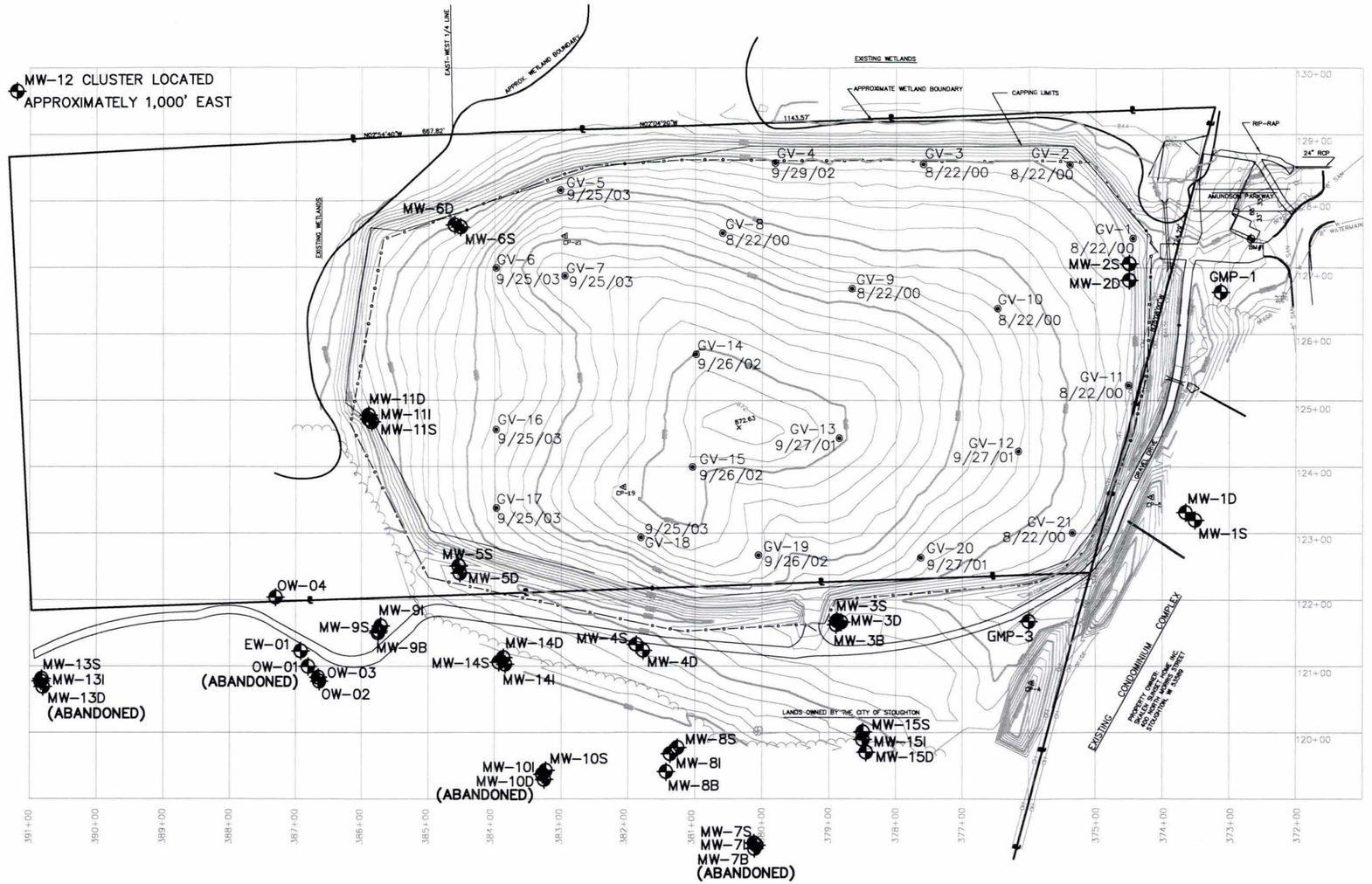
PROJECT NO.	1764
DRAWN BY:	KP
CHECKED BY:	SS
DRAWN:	08/02/00
SCALE:	1" = 2,000'

FIGURE 1
SITE LOCATION MAP
STOUGHTON CITY LANDFILL
STOUGHTON, WISCONSIN

J:\1764\FIG1.DWG

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 REI 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-9,10,12,13,20
YEAR 3 (2002)	GV-4,8,14,15,19
YEAR 4 (2003)	GV-5,6,7,16,17,18

200 0 200

SCALE: 1" = 200'

PROJECT NO.	25216022.00	DRAWN BY:	AHB
DRAWN:	06/06/16	CHECKED BY:	LB
REVISED:	03/28/16	APPROVED BY:	

ENGINEER: SCS ENGINEERS, 2830 DAIRY DRIVE MADISON, WI 53718-6751, PHONE: (608) 224-2830

CLIENT: WISCONSIN DEPARTMENT OF NATURAL RESOURCES

SITE: STOUGHTON CITY LANDFILL, STOUGHTON, WISCONSIN

SITE PLAN

FIGURE 2

I:\25216022.00\Drawings\site1.dwg, 3/31/2016 4:21:38 PM

APPENDIX A

TestAmerica Laboratory Standard Operating Practices

TITLE: Gas Chromatography Mass Spectrometry - Volatiles SW-846 Method 8260B

Approvals (Signature/Date):

JoAnn Petruszak-Kmetty 11-30-15 JoAnn Petruszak-Kmetty Date Supervisor, GC/MS VOA	Chris Hoham 11/30/15 Chris Hoham Date Env. Health & Safety Coor.
Terese A. Preston 11/30/15 Terese A. Preston Date Quality Assurance Manager	Michael J. Healy 11/30/15 Michael J. Healy Date Laboratory Director

Facility Distribution No: _____ **Distributed To:** Uncontrolled

1.0 SCOPE / APPLICATION

To outline the guidelines for the analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) using SW-846 Methods 8260B and 8000B as references. The preparation of all volatile samples is based on Methods 8000B and 5030B. Method 5035 is covered by a separate SOP (UP-SP-5035), but can also be found in this SOP.

On occasion, clients request slight modifications to this SOP. These modifications are addressed on a case-by-case basis with the range of accuracy (i.e., MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into a Quality Assurance Plan (QAP), authorized via laboratory signature approval, and mentioned in the data package's case narrative.

1.1 Method Sensitivity

1.1.1 Method Detection Limits

The method detection limit (MDL), referred to as the detection limit (DL) in NELAC documents, is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to Appendix B of 40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" with additional details are provided in the TestAmerica Corporate SOP, *CA-QS-006, Detection Limits* and the TestAmerica Chicago SOP, *UP-QA-017, Method Detection Limit Studies.* MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; the MDL will be verified on an annual basis.

1.1.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. A demonstration of capability is performed whenever there is a change in instrument type, method or personnel. An Initial Demonstration of Capability (IDOC) must be thoroughly documented and approved by the Department Manager/Supervisor and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in the QA Department and in the Analyst Training files. For additional details on the demonstration of capability procedures followed, refer to the laboratory SOP, *UP-QA-QAM, Quality Assurance Manual, Sections 20.4.2 and 20.4.3.*

1.1.3 Reporting Limits

Reporting Limits [a.k.a., Estimated Quantitation Limits (EQLs) as designated in the method] are defined as the lowest concentration of an analyte determined by a given method in a given matrix that the laboratory feels can be reported with acceptable quantitative error or client requirements, values specified by the EPA methods or other project and client requirements. The laboratory maintains reporting limits that are higher than the MDL. Wherever possible, reporting is limited to values approximately 3-5x the respective MDL to ensure confidence in the value reported.

Table 1 defines the reporting limits and analyte list for SW-846 Method 8260B.

1.1.4 Definitions

Refer to Section 3.0 of the Laboratory's Quality Assurance Manual (UP-QA-QAM).

1.2 Summary of Method

This method is used to determine volatile organic compounds in a variety of matrices. It is applicable to water, soil, sediment, sludge and waste drum samples.

This method can be used to quantify most volatile organic compounds that have a boiling point less than 200°F. It is also limited to those compounds that elute as sharp peaks from a capillary column. A listing of applicable compounds and their characteristic ions appears in Table 2.

A portion of sample, measured into a sample vessel, is purged with an inert gas. The volatile compounds are transferred to a trap, containing retarding materials. The trap is then backflushed with the inert gas and rapidly heated to effectively transfer the compounds to the GC column. The GC oven is then, temperature ramped to separate the compounds and introduce them to the source. The mass filter separates the ions, which are then detected by the analyzer. The data system then provides qualitative and quantitative information concerning the sample.

Instrument calibration occurs about every 12-hours, or prior to analysis. Instrument maintenance is performed as needed or daily basis.

2.0 INTERFERENCES

2.1 External interferences can be caused by contaminants from sample containers, preparative glassware and reagents, syringes and columns and manifest themselves as high background and/or discrete peaks. Some contaminants are also introduced through the sample vial seal and/or instrument sample connections. Proper glassware preparation including rinsing of all volumetric glassware with methanol/water, the rinsing of syringes with water, the baking of both for a minimum of 30 minutes, and proper sample handling and instrument maintenance should eliminate these sources. The rinse water bottles that are filled with Milli-Q water will be rinsed with Methanol on a monthly basis or more frequently as needed to prevent biologic growth within the bottles. A laboratory method blank (MB) is analyzed prior to any analysis to show absence of any contaminants. Reagent (Milli-Q) water sampled in the lab and carried through all field operations is also analyzed to show absence of contaminants from field sampling.

2.2 Carryover is also another source of contamination. Any time a high-level sample is analyzed, the next sample in the batch is checked for carryover. If carryover is suspected, that sample is re-analyzed. If the carryover is excessive and continues into the next samples, the batch is aborted/paused, the column and trap baked, and/or blanks analyzed until all contamination is absent. If further response is required (i.e., trap replacement), it is documented in the maintenance logbook. Refer to Section 7.4 for information on preventive maintenance.

2.3 Internal interferences can be purged from the sample with the target compounds and appear as elevated baselines or distinct peaks. Internal interferences most often manifest themselves as low/high recoveries of surrogate/matrix spike compounds. Matrix interferences vary from sample to sample.

2.4 The volatile lab must be free of solvents. All analytes must be less than their RLs or < 3X the RL for Acetone and Methlyene Chloride. The volatile lab is under positive pressure to reduce lab contamination; however, intermittent low levels of acetone and methylene chloride may occasionally be detected. Refer to Section 8.2 (Corrective Action) for clarification for blank contamination.

3.0 SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document. This procedure may involve hazardous material, operations and equipment. This SOP 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 coat and closed-toe, nonabsorbent shoes are a minimum.

3.1 Specific Safety Concerns or Requirements

- The GC contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- All employees will adhere to the practices and policies in the TestAmerica Corporate Safety Manual (CSM) and will read the SDS's for the materials used in this method before handling or using the material.

3.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Sodium Bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure may cause burn if not flushed with water. May cause mild irritation to skin. Prolonged exposure may cause burn if not flushed with water.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

4.0 EQUIPMENT AND SUPPLIES

4.1 Current Hardware/Software

- 5 Hewlett-Packard 6890 GC interfaced with a 5973 MSD. Equipped with DB-624 column.
- 3 Hewlett-Packard 6890 GC interfaced with a 5975 MSD. Equipped with DB-624 column
- 1 Hewlett-Packard 6890 GC interfaced with a 5973 MSD. Equipped with DB-624 column used as screener.
- 2 Hewlett-Packard 5890 GC interfaced with a 5972 MSD. Equipped with DB-624 column used as Screeners.
- 4 Tekmar 3000 concentrators, 7 OI Eclipse 4660 concentrator in connection to 10 Varian Archon Autosamplers for ten systems.
- 11-Hewlett-Packard Chemstations and peripheral hardware. Using B.02.04, version D02.00 and version D00.00 software.
- 1-Hewlett-Packard Chemserver 9000 series running HP-UX10.2 OS with Chrom/NT version 4.14
- Chrom version 1.2

The GC/MS has a temperature programmable chromatograph interfaced with a mass-selective detector capable of scanning from 35 - 260 amu every second or less using 70 volts of electron energy in the electron ionization mode. The system is capable of producing an acceptable spectrum of bromofluorobenzene when 50 ng is analyzed.

4.2 Data System

The analytical systems are interfaced with stand alone PC's which are Pentium based systems running Agilent Chemstation. This system is capable of continuous acquisition and storage of mass spectral data. The software allows plotting specific masses versus time or scan numbers (Extracted Ion Current Profile-EICP) and integration of that abundance. The system also stores the data. The system contains the latest NBS Library.

4.3 Data File Name/ Batch Directory Assignment

Tune, standard, blank, and laboratory control sample (LCS) data files are designated by specific letters unique to each instrument in conjunction with the appropriate month and day (example : 3b0318 = instrument #3, first 12 hour BFB tune, March 18). When a worklist is made in Chrom, a unique Chrom ID# is assigned to each file which aids in the transfer of data from chemstation to Chrom.

4.4 Miscellaneous

- assorted syringes (10, 25, 50, 100, 500 and 1000 uL)
- 5 mL luer-lock gas-tight syringes
- assorted purge vessels (water, 5/25 mL)
- top-loading balance, capable of weighing to $\pm$ 0.1 g, stainless steel spatula
- assorted amber and clear Teflon-lined screw-capped vials (1.5-2.0 mL, 3.5-5.0 mL)
- cleaned 40 mL vials w/Teflon-lined screw-caps
- assorted volumetrics (10 mL, 20 mL, 25 mL, 50 mL and 100 mL)

5.0 REAGENTS AND STANDARDS

The majority of the calibration standards are EPA certified, A2LA or second-source verified by the standard vendor in situations where suitable SRMs (Standard Reference Material) was available. For those compounds where standards must be made from neat material (due to instability) or some non-routine compounds, **where available**, a second-source is purchased and used in the LCS to verify the standard

Each time a new initial calibration is required, new standards are prepared and the standards are verified against a second-source LCS (ICV-Initial Calibration Verification standard) prior to any sample analysis. This holds for all routine compounds and those available as second-source material in the LCS (see page 11 for list of compounds and venders).

All neat standards received are entered into TALs (LIMS). A label is printed from TALs and placed on the bottle. All neat standards are then stored according to manufacturer's recommended storage conditions. The standard is issued a unique ID# [i.e., Neat Standards Reference Number (NSRN)] which is used to track all standards as they are used as is or in preparation of stock/working solutions. The format of the standards in TALs (LIMS) will prevent working or intermediate level solutions from being used past the expiration date of the neat or stock solutions. Lot # of Methanol reagent used should be recorded in the comment section of the prep batch in TALs. In addition, the preparation of the 20% Sodium Bisulfate should be recorded in TALs.

5.1.1 Reagent Water (Milli-Q)

1-Liter of water is continuously purged with pre-purified nitrogen. The reagent water is routinely demonstrated to be interference-free. All compounds are < EQL or 3x EQL for methylene chloride and acetone.

5.1.2 Methanol (MeOH)

All new lot numbers of P & T J.T.Baker Methanol are analyzed and verified to be free of contaminants. This information is available on the TestAmerica Oasis Web-Site that can be accessed by all analysts. The currently approved lot numbers are listed on this site.

5.2 Surrogate Spiking Solution

Surrogates are purchased as custom mix solutions from Restek in 5.0 mL ampules. The following surrogates are used:

Compound	Concentration
4-Bromofluorobenzene	2500 ppm
1,2-Dichloroethane-d_4	
Toluene-d_8	
Dibromofluoromethane	

Upon opening, all contents are transferred to 5 mL amber, Teflon-lined screw-capped vials. The standard is issued another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: 1-year unopened or manufacturers expiration; once opened, they are used for a period of 3 months or until used.
- Storage Requirements: Stored in a freezer at ~ –10°C in the dark and kept for a period of 1-year unopened. *

Working Surrogate mix are prepared using the custom mix above and diluting to 30 ppm as follows:

Custom Mix	Volume (uL)	MeOH	Concentration
2500 ppm Surrogate Mix	24	Dilute to 2mL	30 ppm each component

5uL injected into 5 mL of water/sample results in 30 ppb surrogate concentrations.

- The mixture is transferred to and stored in 1.5-2.0 mL amber Teflon-lined screw-capped vials at ~ –10 °C in the dark. The transfer is entered into TALs (LIMS). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: Working SS solutions have an expiration date of 2-weeks.

5.3 Internal Standard Spiking Solutions

Internal standards are purchased as custom mix solutions from Restek in 5.0 mL ampules. The following internal standards are used:

Compound	Concentration
Fluorobenzene	250 ppm
Chlorobenzene-d_5	250 ppm
1,4-Dichlorobenzene-d_4	250 ppm
tert-butyl-alcohol-d9	5000 ppm
1,4-Dioxane-d8	5000 ppm

Upon opening, all contents are transferred to 5 mL amber, Teflon-lined screw-capped vials. The standard is issued another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: 1-year unopened or manufacturers expiration; once opened, they are used for a period of 3 months or until used.
- Storage Requirements: Stored in a freezer at ~ –10°C in the dark and kept for a period of 1-year unopened. *

Working Internal standard mix are prepared using the custom mix above and diluting to 50/1000 ppm as follows:

Custom Mix	Volume (uL)	MeOH	Concentration
250/5000 ppm IS Mix	400	Dilute to 2mL	50/1000 ppm each component

5uL injected into 5 mL of water/sample results in 50/1000 ppb internal standard concentrations.

- The mixture is transferred to and stored in 1.5-2.0 mL amber Teflon-lined screw-capped vials at ~ –10 °C in the dark. The transfer is entered into TALs (LIMS). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: Working IS solutions have an expiration date of 2-weeks.

5.3 Internal Standard/Surrogate Spiking Solutions

Internal Standards/Surrogate mix are purchased as a custom mix solution from Restek in 5.0 mL ampules. The following internal/surrogate standards are used:

Compound	Concentration
Fluorobenzene	250 ppm
Chlorobenzene-d_5	250 ppm
1,4-Dichlorobenzene-d_4	250 ppm
tert-butyl-alcohol-d9	5000 ppm
1,4-Dioxane-d8	5000 ppm
4-Bromofluorobenzene	150 ppm
1,2-Dichloroethane-d_4	150 ppm
Toluene-d_8	150 ppm
Dibromofluoromethane	150 ppm

Upon opening, all contents are transferred to 5 mL amber, Teflon-lined screw-capped vials. The standard is issued another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: 1-year unopened or manufacturers expiration; once opened, they are used for a period of 3 months or until used.
- Storage Requirements: Stored in a freezer at ~ –10°C in the dark and kept for a period of 1-year unopened. *

Working Internal standard/Surrogate mix are prepared using the custom mix above and diluting to 30/50/1000 ppm as follows:

Custom Mix	Volume (uL)	MeOH	Concentration
150/250/5000 ppm IS/SS Mix	4000	Dilute to 20mL	30/50/1000 ppm each component

5uL injected into 5 mL of water/sample results in 50/1000 ppb internal standard/surrogate concentrations.

- The mixture is transferred to and stored in 1.5-2.0 mL amber Teflon-lined screw-capped vials at ~ –10 °C in the dark. The transfer is entered into TALs (LIMS). The standard issued is another unique ID# [i.e., SRN (Standard Reference Number)] which can be traced back to the parent ID# (i.e., NSRN with the date of receipt, date of opening, and the supplier).

- Life of Standard: Working IS/SS solutions have an expiration date of 2-weeks.

Purgeables
Calibration Standards

8260 List 1/Std #1 Mega Mix 2,500 ug/mL in MeOH		8260List 1/Std#5 Acrolein 20,000 ug/mL in H2O
1,1,1,2-Tetrachloroethane	n-Propylbenzene	Acrolein
1,1,1-Trichloroethane	Methyl-tert-butyl ether	
1,1,2-Trichlorotrifluoroethane	Naphthalene	**8260 List2/Std#1 Additions**
1,1,2,2-Tetrachloroethane	o-Xylene (1,250)	2,500 ug/mL in MeOH
1,1,2-Trichloroethane	sec-Butylbenzene	Ethanol (100,000)
1,1-Dichloroethane	Styrene	2-Propanol (isopropanol) (25,000)
1,1-Dichloroethylene	tert-Butylbenzene	Acetonitrile (25,000)
1,1-Dichloropropylene	Tetrachloroethylene	Diisopropyl ether (DIPE)
1,2,3-Trichlorobenzene	Tetrahydrafuran	Chloroprene (2-chloro-1,3-butadiene)
1,2,3-Trichloropropane	Toluene	Ethyl-tert-butyl ether (ETBE)
1,2,4-Trichlorobenzene	trans-1,2-Dichloroethylene	Proprionitrile (25,000)
1,2,4-Trimethylbenzene	trans-1,3-Dichloropropylene	Ethyl Acetate (5,000)
1,2-Dibromo-3-chloropropane	Trichloroethylene	Methacrylonitrile (25,000)
1,2-Dibromoethane	Methyl Acetate (12,500)	Tert-Amyl methyl ether (TAME)
1,2-Dichlorobenzene	Acrylonitrile (25,000)	1-Butanol (62,500)
1,2-Dichloroethane	n-Hexane	Ethyl acrylate
1,2-Dichloropropane	Isobutanol (62,500)	Methyl methacrylate (5,000)
1,3,5-Trimethylbenzene	Cyclohexane	2-Nitropropane (5,000)
1,3-Dichlorobenzene	Methylcyclohexane	Butyl Acetate
1,3-Dichloropropane	1,4-Dioxane (50,000)	1,2,3-Trimethylbenzene
1,4-Dichlorobenzene	Ethyl methacrylate	Benyl Chloride
2,2-Dichloropropane	Trans-14-dichlor-2-butene	1,3,5-Trichlorobenzene
2-Chlorotoluene		
4-Chlorotoluene		**8260 List2/Std#2 Extra**
4-Isopropyltoluene		2,500 ug/mL in MeOH
Benzene	**8260 List1/Std#3 Gases**	Pentachloroethane
Bromobenzene	2,500 ug/mL in MeOH	2-Methylnaphthalene
Bromochloromethane	1,3-Butadiene	
Bromodichloromethane	Dichlorofluoromethane (CFC-21)	**8260 List2/Std#3 Cyclohexanone**
Bromoform	Chloroethane (ethyl chloride)	25,000 ug/mL in H2O
Carbon tetrachloride	Methyl Bromide (Bromomethane)	Cyclohexanone
Chlorobenzene	Methyl chloride (Chloromethane)	
Chloroform	Trichlorofluoromethane (CFC-11)	
Chlorohexane	Dichlorodifluoromethane	
cis-1,2-Dichloroethylene	Vinyl chloride	
cis-1,3-Dichloropropylene		
Dibromomethane	**8260 List1/Std#2 Ketones**	
Dibromochloromethane	12,500 ug/mL in MeOH	
Dichloromethane	Acetone	
Ethylbenzene	2-Hexanone	
Hexachlorobutadiene	Methyl ethyl ketone (2-Butanone)	
Isopropylbenzene	4- Methyl-2-pentanone (MIBK)	
m-Xylene & p-Xylene		
n-Butylbenzene	**8260 List1/Std#6 Vinyl Acetate**	
Diethyl ether (Ethyl Ether)	5,000 ug/mL in MeOH	
Tert-Butanol (TBA) (25,000)	Vinyl Acetate	
Iodomethane		
Allyl Chloride (3-Chloroprene)	**8260 list1/Std#4 2-CEVE**	
Tetrahydrofuran (5,000)	2,500 ug/mL in MeOH	
n-Heptane	2-Chloroethyl vinyl ether	

5.4 Stock Purgeable Standards

These are obtained as custom mixes from Restek. The contents of each solution and concentration appear on the previous page. Upon opening, all contents are transferred to 1.5-2.0 mL amber, Teflon-lined screw-capped vials. Listed are compounds in the EPA TCL and includes compounds done on a regular basis. Other standards, if needed, are either purchased as neat solutions or neat standards from Supelco, Chem Service or other certified supplier. See appropriate entries in TALs (LIMS).

* If the stock solution has manufacturers' expiration date, that is assigned. If the date is not evident, 1-year is assigned to un-opened ampules. This is applicable for all "neat" standards.

5.4.1.1 Main 8260 Mix

The 8260/624 Mega Working Standard is prepared as follows:

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
8260List1/Std#1 MegaMix (2000-62,500 ug/mL)	80	Dilute to 2 mL	100-2500 ppm

5.4.1.2 Gases

The 8260/624 Gas Working Standard is a prepared as follows:

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
8260 List1/Std#3 Gases 2500ug/mL	80	Dilute to 2 mL	100 ppm each component

5.4.1.3 Additional Compounds

The 8260/624 Ketone Working Standards is prepared as follows:

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
8260 List1/Std#2 Ketones 12,500ug/mL	16	Dilute to 2 mL	100 ppm each component

The 2-CEVE and Vinyl Acetate Working Standards is prepared as follows:

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
8260List1/Std#4 2-CEVE 2,500 ug/mL	80	Dilute to 2 mL	100 ppm each component
8260List1/Std#6 Vinyl Acetate 5,000 ug/mL	40		

The Acrolein Working Standards is prepared as follows:

Stock Compound/Mix	Volume (uL)	Water	Concentration
8260List 1/Std#5 Acrolein 20,000 ug/mL	400	Dilute to 2 mL	4000 ppm each component

5.4.1.7 8260B List 2 Standards

The Appendix IX and extra compounds are prepared as follows:

Compound / TCL Mix	Volume (uL)	MeOH	Concentration
8260 List2/Std1 Additions	80	Diluted to 2mLs	100/200/1000/2500/5000 ppm

Compound / TCL Mix	Volume (uL)	Water	Concentration
8260 List2/Std 2 Extras (2500ug/mL)	80	Diluted to 2mLs	100ppm

Compound / TCL Mix	Volume (uL)	Water	Concentration
8260 List2/Std3 Cyclohexanone (25,000ug/mL)	800	Diluted to 2mLs	10,000 ppm

- Life of Standard: Unopened ampules are assigned the manufacturer's expiration date or 1 year from receipt. Working solutions have an expiration date of 1-week (Gases, 2-CEVE, Vinyl Acetate, Acrolein and nitriles) and 2-weeks for all others.
- Storage Requirements: These mixtures are stored in 1.5-2.0 mL amber Teflon-lined screw-capped vials at ~ –10 °C in the dark.

NOTE: All standard 'recipes' are listed here in this SOP for guidelines for standard preparation. These 'recipes' are subject to change. The standards listed here are standards that are regularly used. Some clients may request other compounds not listed here. Those compounds will be evaluated for accuracy via this method and analyzed for on a project basis.

5.4.1.8 Low Level Standard

A low level standard is prepared by making a 1/10 dilution of the stock standards of each of the above (nitriles and acrolein included). This standard is used to prepare the low points in the initial calibration. The low level standard may contain the Main 8260 Mix, gases, nitriles and acrolein, and any other required standard. A low-level standard for the Appendix IX compounds is also prepared separately due to duplication of some compounds.

A low level surrogate solutions is also prepared by a 1/10 dilution of the working for low points in the water curve. The calibration levels may vary with the compounds. See recipes in the calibration section for the levels. The low point in the calibrations is based on each compounds reporting limit.

All solutions are stored in a 1.5-2.0 mLs amber Teflon-lined screw-capped vials at –10°C in the dark. All standard preparation is recorded in the TALs (LIMS) system. Solutions are prepared every 2-weeks (1-week for the gases, 2-CEVE, Vinyl Acetate, Acrolein and Nitriles).

Purgeable Spike Standard from a Different Lot Number
Intial Calibration Verification (ICV) Mixes

8260 List 1/Std #1 Mega Mix.sec 2,500 ug/mL in MeOH		**8260List 1/Std#5 Acrolein.sec** 5,000 ug/mL in H2O
1,1,1,2-Tetrachloroethane	n-Propylbenzene	Acrolein
1,1,1-Trichloroethane	Methyl-tert-butyl ether	
1,1,2-Trichlorotrifluoroethane	Naphthalene	**8260 List2/Std#1 Additions.sec**
1,1,2,2-Tetrachloroethane	o-Xylene (1,250)	2,000 ug/mL in MeOH
1,1,2-Trichloroethane	sec-Butylbenzene	Ethanol (100,000)
1,1-Dichloroethane	Styrene	2-Propanol (isopropanol) (20,000)
1,1-Dichloroethylene	tert-Butylbenzene	Acetonitrile (20,000)
1,1-Dichloropropylene	Tetrachloroethylene	Diisopropyl ether (DIPE)
1,2,3-Trichlorobenzene	Tetrahydrafuran	Chloroprene (2-chloro-1,3-butadiene)
1,2,3-Trichloropropane	Toluene	Ethyl-tert-butyl ether (ETBE)
1,2,4-Trichlorobenzene	trans-1,2-Dichloroethylene	Proprionitrile (20,000)
1,2,4-Trimethylbenzene	trans-1,3-Dichloropropylene	Ethyl Acetate (4,000)
1,2-Dibromo-3-chloropropane	Trichloroethylene	Methacrylonitrile
1,2-Dibromoethane	Methyl Acetate (12,500)	Tert-Amyl methyl ether (TAME)
1,2-Dichlorobenzene	Acrylonitrile	1-Butanol
1,2-Dichloroethane	n-Hexane	Ethyl acrylate
1,2-Dichloropropane	Isobutanol (62,500)	Methyl methacrylate
1,3,5-Trimethylbenzene	Cyclohexane	2-Nitropropane
1,3-Dichlorobenzene	Methylcyclohexane	Butyl Acetate
1,3-Dichloropropane	1,4-Dioxane (50,000)	1,2,3-Trimethylbenzene
1,4-Dichlorobenzene	Ethyl methacrylate	Benyl Chloride
2,2-Dichloropropane	Trans-14-dichlor-2-butene	1,3,5-Trichlorobenzene
2-Chlorotoluene		
4-Chlorotoluene		**8260 List2/Std#2 Extra.sec**
4-Isopropyltoluene		2,500 ug/mL in MeOH
Benzene	**8260 List1/Std#3 Gases.sec**	Pentachloroethane
Bromobenzene	2,500 ug/mL in MeOH	2-Methylnaphthalene
Bromochloromethane	1,3-Butadiene	
Bromodichloromethane	Dichlorofluoromethane (CFC-21)	**8260 List2/Std#3 Cyclohexanone.sec**
Bromoform	Chloroethane (ethyl chloride)	25,000 ug/mL in H2O
Carbon tetrachloride	Methyl Bromide (Bromomethane)	Cyclohexanone
Chlorobenzene	Methyl chloride (Chloromethane)	
Chloroform	Trichlorofluoromethane (CFC-11)	
Chlorohexane	Dichlorodifluoromethane	
cis-1,2-Dichloroethylene	Vinyl chloride	
cis-1,3-Dichloropropylene		
Dibromomethane	**8260 List1/Std#2 Ketones.sec**	
Dibromochloromethane	12,500 ug/mL in MeOH	
Dichloromethane	Acetone	
Ethylbenzene	2-Hexanone	
Hexachlorobutadiene	Methyl ethyl ketone (2-Butanone)	
Isopropylbenzene	4- Methyl-2-pentanone (MIBK)	
m-Xylene & p-Xylene		
n-Butylbenzene	**8260 List1/Std#6 Vinyl Acetate .sec**	
Diethyl ether (Ethyl Ether)	5,000 ug/mL in MeOH	
Tert-Butanol (TBA) (25,000)	Vinyl Acetate	
Iodomethane		
Allyl Chloride (3-Chloroprene)	**8260 list1/Std#4 2-CEVE.sec**	
Tetrahydrofuran (5,000)	2,500ug/mL in MeOH	
n-Heptane	2-Chloroethyl vinyl ether	

5.5 Stock Spike Solution

The spike compounds are obtained as solutions from a different source from Restek in 1.5-2.0 mL ampules. These are listed on the previous page. A different analyst than the one who prepared the calibration solutions usually prepares spike solutions. These are stored according to manufacturer's recommended storage conditions. Neat standards are kept for a period of 1-year un-opened or the manufacturer's expiration date. Once opened, the stock may be used for 3-months.

The spike solutions are prepared as follows:

5.5.1 VOC Spike

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
8260List1/Std#1.sec MegaMix (2000-20,000 ug/mL)	40	Dilute to 2 mL	50-500 ppm

5.5.2 Gas Spike

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
8260 List1/Std#3.sec Gases 2000ug/mL	40	Dilute to 2 mL	50 ppm each component

5.5.3 Additional Spike Compound Mix

The 8260/624 Ketone Working Spike is prepared as follows:

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
8260List1/Std#2.sec Ketones 10,000ug/mL	8	Dilute to 2 mL	50 ppm each component

The 2-CEVE and Vinyl Acetate Working Standards is prepared as follows:

Stock Compound/Mix	Volume (uL)	MeOH	Concentration
8260List1/Std#4.sec 2-CEVE 2,000 ug/mL	40	Dilute to 2 mL	50 ppm each component
8260List1/Std#6.sec VinylAcetate 4,000 ug/mL	20		

The Acrolein Working Standards is prepared as follows:

Stock Compound/Mix	Volume (uL)	Water	Concentration
8260 List1/Std#5.sec Acrolein 20,000 ug/mL	100	1 mL	2000 ppm each component

5.5.4 List 2 Spikes

Compound / TCL Mix	Volume (uL)	MeOH	Concentration
8260 List2/Std1 .sec Additions	40	Diluted to 2mLs	50/100/500/1250/2500 ppm

Compound / TCL Mix	Volume (uL)	Water	Concentration
8260 List2/Std 2.sec Extras (2000ug/mL)	40	Diluted to 2mLs	50ppm

Compound / TCL Mix	Volume (uL)	Water	Concentration
8260 List2/Std3.sec Cyclohexanone (20,000ug/mL)	200	Diluted to 2mLs	5,000 ppm

For waters, addition of 5 uL of each solution results in all spike compounds at 50 ppb.
For soils, addition of 5 uL of each solution results in all compounds at 50 ppb.

These solutions are stored at ~ -10°C in several 1.5-2.0 mL amber Teflon-lined screw-capped vials. All standard preparation is recorded in the TALs (LIMS) system. Working matrix spike solutions have a 2-week/1-week (Gases) expiration date. See above for label information.

5.6 Stock BFB Solution

The BFB standard is purchased as a neat solution from Supelco.

Stock	Amount	MeOH	Concentration
2000 ppm BFB	25 uL	Dilute to 2 mLs	25 ppm

- Life of Standard: This stock can be kept for a period of 1-year until opening. Upon opening, the solution is transferred to a 1.5 - 2.0 mL vial and assigned an SRN. Once opened, it is used for a period of 6-months.
- Storage Requirements: The standard is stored at ~ –10°C in the dark

Addition of 2 uL to 5 mLs results in a concentration of 50 ng/5 mLs.
All preparation is recorded in the TALs (LIMS) system. All labels are completed as above.

NOTE: Intermediate and Working Solutions are never assigned an expiration date exceeding the expiration date of the neat/stock standards/solutions.

6.0 CALIBRATION

Before an instrument is used as a measuring device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration is documented.

6.1 PFTBA Autotune or Manual Tune

The instrument is first tuned in one of two ways: autotune or manual tune. The ion abundances in the calibration gas are best monitored near the temperature of analysis of BFB. Monitoring at this temperature produces the most representative cal gas scan and therefore the best estimate of BFB response.

1. If an AUTOTUNE is to be performed, continue below. If not, skip to step 6. An autotune is not run before every initial calibration. If the instrument has been down for any reason previously listed or major difficulties in manual tune are encountered, an autotune is performed. Autotunes are generally NOT performed when an existing initial calibration is being met.

2. The Chemstation software has a menu driven tune program. Begin the autotune program. Key masses are 69, 219 and 502.

3. Follow instructions and retrieve a hardcopy of the autotune results. Check the following:

- passed/fail: in itself, not necessarily an indication of MS performance
- repeller and ion focus settings
- electron multiplier voltage

4. The repeller and EM voltages are good indicators of the sources' cleanliness. Generally, the lower the setting the cleaner the source. Other factors may however, supersede (i.e., the age of the multiplier) and a clean source will not always autotune these low. The EM is set by autotune program to produce a target abundance for mass 69 (varies depending on the tune program and instrument). The operator may plan on having to increase this by 100-200 to achieve normal analysis sensitivity (depends on the tune program and the instrument).

5. Observe peak shape, absence of lead-ons/tailing, the resolution between isotopes, peak width and mass axis. A hardcopy of the profile scan is desirable, and can be filed with the autotune results.

6. If an AUTOTUNE has just been performed, continue here. If not, skip to step 9. Enter MANUAL TUNE and read the autotune (which was automatically stored in a file). For volatiles, edit the scan parameters to monitor ions 69, 131 and 219.

7. Enter one of several methods available and adjust the parameters (usually the ion focus, entrance lens and amu gain) to achieve the following relative abundances:

Mass	Relative Abundance
69	100%
131	40-60%
219	50-70%

These will vary with the MS. Mass 219 is usually 5-9% greater than mass 131. If necessary, adjust the amu gain for peak shape and high-end isotope resolution. An overall peak-width of 0.500 to 0.550 is desirable.

Again, these adjustments and relative abundances may not guarantee that BFB will meet requirements, but is a good place to start.

8. A hardcopy the profile scan is desirable. This can be filed with the autotune results. This file can serve as a diagnostic tool and can also provide a starting point in the event the operator has trouble meeting the initial calibration.

Save the changes to the appropriate Tune File. Exit the program.

9. If an AUTOTUNE has not been performed, enter MANUAL TUNE and adjust any parameters, if need be. Adjustment may not be necessary, and not desirable, if problems in tuning or meeting the initial calibration have not been encountered. A hardcopy of profile scan can be printed and filed. Save the changes and exit the program.

6.2 BFB Analysis

Once the instrument is tuned, 50 ng of 4-Bromofluorobenzene must meet criteria. The BFB can be purged. The mass spectrum must meet the following criteria:

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

The manner of acquiring the mass spectrum of BFB can be performed in only two specific ways with Chrom software:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is performed and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB. This procedure of averaging and subtracting is performed automatically by the Chrom software and evaluated by the criteria in the table below. If this procedure does not meet the acceptance criteria, then the analyst will use the following procedure:
- One scan of the BFB peak is acquired and a background peak is subtracted and evaluated by Chrom software using the criteria above. This procedure is performed manually by the analyst. No other manner of BFB acquisition and evaluation can be utilized.

The BFB is analyzed by one of the methods in Attachment 1. **(Method parameters listed in the appendices are examples only. This statement applies to all references made to these methods).** Typical concentrator conditions also appear in Attachment 1. The EM voltage may be 100-200 volts above autotune. The abundances of the designated masses above MUST meet the criteria before analyses can begin. If necessary, enter MANUAL TUNE and adjust parameters. BFB analysis is completed about every 12-hours of analysis.

6.3 Description of Initial Calibration

An initial calibration may be completed:

- as needed - continuing calibration can not be met
- after a source cleaning and/or column change or any time a major repair or change has occurred with the instrument that affects calibration where a new calibration is indicated.

Confirm that the GC/MSD is stable and equilibrated. If at all possible, allow the instrument to equilibrate overnight at all operating temperatures if the source/column has been cleaned/changed. Prior to beginning initial calibration it is a good idea to:

- check the background of air/water levels and base ion by scanning for appropriate ions and also visually inspecting the spectrum scan for any other possible and undesirable background.
- recheck the multiplier settings, after a source is cleaned the EM can most often be dropped.

6.4 Initial Calibration

Each calibration standard is analyzed according to one of the methods in Attachment 1. These are examples. The actual number of points in the calibration is determined by the calibration and acceptance criteria table (Attachment 4). The EM voltage may be 100-200 volts above autotune.

Allow standards to come to ambient temperature.

Fill ten 5-mL luer-lock gas-tight syringes with reagent water to overflowing. Replace the plunger and invert. Adjust to 5-mL confirming the absence of any air bubbles. Pull back slightly on the plunger to allow addition of standards. Following the guides found in the Attachment 1, add the appropriate amount of standards and methanol.

Immediately add the standards to a clean 40-mL vial. Following the method parameters outlined in Attachment 1, analyze the 50 ppb standard. A normal standard will appear very similar to the ones in Figures 1 and 2. Quantitate the standard against the appropriate method file. Sufficient areas for the first internal standard will vary somewhat between instruments. Acceptable areas should be based on maintaining sufficient sensitivity for poor responders without saturating the detector at the upper end of the calibration range. Too low an area will almost guarantee poor/unsatisfactory responses of low-response compounds and too high an area will result in saturation of some compounds at higher levels, resulting in false low response factors at high concentrations.

It is helpful to analyze a medium level standard first and assess the areas before continuing with the low/high level standards.

Response factors are calculated by the data system as follows:

$$RF = \frac{A_x \times Q_s}{A_s \times Q_x}$$

Where:
A_x = ion abundance for analyte
A_s = ion abundance for its internal standard
Q_s = concentration of its internal standard
Q_x = concentration of analyte
(Response Factors have no units)

The appropriate quant ion must be in the method file. A listing of the target compounds with their appropriate internal standards appears in Attachment 2. Confirm the presence of all targets and the separation of non-co-eluting compounds. Note the response factors for the gasses. If necessary, prepare new standards.

If adjustments to the acquisition parameters are necessary, make them and re-analyze the 50 ppb standard.

The entire initial calibration shall be analyzed. If a point is found to be invalid due to an injection or instrument problem confined to that run, than the point may be repeated once only. If that point fails again a new ICAL must be performed. Removal of points for individual analytes from levels other than the highest or lowest level is not permitted in any event. Unused or replaced standards must have a clear explanation of why they were not used or why they were replaced. This explanation must be documented on the Corrective Action/Qualification Report bound with the run log.

When a standard is analyzed and processed as part of the initial calibration the RF's are automatically updated in the daily method. After all initial calibration standards are processed, checked and confirmed as being accurate and passing method criteria, the initial calibration is locked and set as the most recent. This ensures that the correct initial calibration is used for each ensuing continuing calibration check. A hardcopy of the calibration report is generated. All method criteria are assessed for compliance. Confirm that:

1) all CCC's are below 30% (Vinyl Chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene)

2) the RF's for SPCC compounds are >0.300 (Chlorobenzene, 1,1,2,2-Tetrachloroethane) (Minimum RF for Chloromethane, Bromoform and 1,1-Dichloroethane is 0.100).

Calibration curves are evaluated following the "Evaluation and Acceptance Criteria" table (Attachment 4). For all compounds in the initial calibration with a %RSD > 15.0%, calibration curves of area ratio versus concentration using a first or higher order regression curve of the calibration curve points will be performed. Weighted linear curves may be used. Quadratic curves may only be used after evaluation of the curve plot and Supervisor approval.

Note: The state of South Carolina does not allow the use of quadratic curves, therefore, in those cases, only the weighted linear option is allowed. If the %RSD criteria are not met, only the linear regression model will be used. The state of Wisconsin will only allow the use of quadratic curves if verification is performed at two spiking levels. The CCVL will be used for this purpose.

Method 8000B/8260B specifies a minimum coefficient of determination (R^2) of 0.990. The methods also specify a minimum of 5 calibration points for a linear model and a minimum of 6 calibration points for a higher order regression. The laboratory, in order to meet requirements, will analyze a minimum number of points to satisfy both. All efforts will be made to meet the minimum COD of 0.990. However, there are some compounds that historically present a problem meeting this requirement (See attachment 9 for a list of poor purgers). These compounds are usually those listed in the analyte table of Method 8260B with qualifying remarks. Many of these have various known issues that would effect reproducibility (i.e., Acetone qualifier pp = poor purger). These typically include many of the Appendix IX compounds as well. The laboratory will take minimal action for these compounds.

The preparation instructions noted above will be modified to include the necessary calibration levels. These instructions are for guidance only and may change as needed.

An example of an acceptable initial calibration appears in Attachment 2. The BFB tune, and all standard raw data are filed. Each instrument has its own initial calibration.

Each time a new initial calibration is required, the standards are verified against a second-lot number LCS (ICV-Initial Calibration Verification standard) prior to any sample analysis. This holds for all routine compounds and those available as second-source material in the LCS (see page 11 for list of compounds). The ICV must meet 30%D for all compounds or corrective action must be taken. Some client's require 20%D. For South Carolina, all compounds must meet 30%D unless there are compounds identified as poor purgers in the SOP. The poor purgers should still meet 40%D. South Carolina does not allow the use of marginal exceedances. Multiple ICV's without documented corrective action or valid reason for doing so, is not acceptable.

NOTE: The actual number of points in the calibration and the low point in the calibration may vary with client and project need. Clients may have additional requirements, which would be covered in a client-specific or regulatory/agency QAPP.

6.5 Daily or Continuing Calibration

Continuing calibration occurs prior to analysis.

If time remains after the initial calibration, and the 50 ppb standard meets continuing calibration criteria, samples can be analyzed up to the 12-hour tune limit. The samples are quantitated against the average RF or appropriate as per method. See later sections describing calculations.

After having satisfied BFB tune requirements, a continuing calibration standard must be analyzed. Analyze a 50 ppb standard following the procedure outlined above. Confirm Form 7 that:

1. all CCC's are below 20% (Vinyl Chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene)

2. the RF's for SPCC compounds are >0.300 (Chlorobenzene, 1,1,2,2-Tetrachloroethane) (Minimum RF for Chloromethane, Bromoform and 1,1-Dichloroethane is 0.100).

NOTE: Method 8260B stipulates that if the CCC's are not part of the analyte list or a short list of compounds is being analyzed, then all compounds being reported must be < 20% drift.

If continuing calibration can not be met, either new standards and/or a new calibration are needed. Multiple CCV's without documented corrective action or valid reason for doing so is not acceptable. Since a CCV can fail for many reasons it is important to determine why it failed or where the problem originated (reagents, autosampler, concentrator, GC, Mass spectrometer).
If the initial CCV fails method criteria, try and determine why it failed:

need new standards?
manual tune needs adjusting?
carry-over issues?
low or high internal standard responses?

Take appropriate actions to correct the problem. Document corrective actions on the corrective action/qualification report. Re-analyze the CCV. If the second CCV fails method criteria, compare the two standards:

Did corrective action help?
Are there any trends between the two standards?
Baseline rise or ghost peaks?
Any indication of leak or gas contamination?
Does further maintenance need to be performed?
- Autosampler
  - replace needle?
  - Check flows and temperatures
- Concentrator
  - Change trap?
  - Check flows and temperatures
- GC
  - liner/seal need to be replaced or cleaned?
  - Check flows/temperatures
- Mass spectrometer
  - Instrument need to be cleaned?
  - Mutltiplier and filaments stable?

TNI standard EL-VM4-2009 Mod 4 section 1.7.2 states: " If the continuing calibration verification results obtained are outside the established acceptance criteria and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after corrective action with two consecutive calibration verifications, or a new initial calibration shall be performed". Immediately running an ICAL without correcting the problem would not be productive. It could result in analyzing samples on a poor ICAL or on an instrument that is not functioning correctly. If two CCV's fail and corrective action was performed then, two acceptable consecutive CCV's must be obtained to prove the corrective action fixed the problem. If an acceptable CCV can not be obtained and the instrument appears to be stable then a new initial calibration should be analyzed.

All internal standard areas and retention times are assessed immediately after calibration. Areas and times compared to the mid point of the initial calibration. Internal standard areas should not deviate by a factor of two or the retention times should not deviate by > 30 s. If the situation occurs, appropriate action is taken and the standard re-analyzed. All corrective action and return to control are documented in the Corrective Action section in the analysis logbook for the appropriate instrument.

7.0 PROCEDURE

7.1 Quality Control Checks

Quality Control is accomplished through:
1) daily tuning and calibration checks and
2) preparation QC traceable through individual batches.

7.1.1 Initial Calibration

PFTBA		
BFB TUNE	Prior to Initial Cal	*limits in Section 6.2
200 \		
150 |		
100 |		
50 |	Initial Cal need dependent on	*limits in Section 6.4
20 |	situation.	
5 |		
2 |		
1 |		
0.5 /		

NOTE: As stated, the actual number of points in the calibration and the low point in the calibration may vary with client and project need. Minimum number of points 3rd Edition SW-846 may be 6 or 7 depending on matrix. Other clients may have additional requirements, which would be covered in a client-specific QAPP.

7.1.2 Method Blank (MB)

Prior to any analysis, the reagent water and the instrument must be shown to be free of interference's and target compounds.

A 5 mL portion of reagent water is analyzed using one of the methods in Attachment 1. Internal Standards (ISS), Surrogates (SSS) and methanol will be added prior to analysis. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. All target compounds must be less than the quantitation limit (See Section 2.0). Once the MB analysis is complete and acceptable, analysis can proceed.

7.1.3 Daily Analysis

BFB	Prior to continuing	* See above calibration
Daily Calibration Standard	Prior to samples	* See Section 6.5

Prep QC	Frequency
MB	Prior to analysis
LCS[1]	1 per analysis batch
MS/MSDs[2]	at least 1 set in 20
Surrogates	every blank, sample and QC Sample
Samples *	

***Any given 12-hour period contains a tune, standard, blank and LCS. Preparation QC is at a 5% frequency. Instrumental controls are outlined above and further discussed in the procedure section.**

[1] LCS Duplicate (LCD) is performed when insufficient sample is available for an MS/MSD.
[2] The sample selection for MS/MSD, if not specified by the client on the chain-of-custody, is rotated among client samples so that various matrix problems may be noted and/or addressed.

7.2 Sample Preservation and Storage

Sample containers, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance and/or specific contract or client requests. Listed below are the holding times and the references that include container and preservation requirements for compliance with the Resource Conservation and Recovery Act (RCRA).

Matrix	SW-846
soils	14-days
Preserved waters	14-days
Non-preserved waters	7-days

All samples received for volatile analysis are refrigerated upon receipt at 4±2°C. Refrigeration is the only preservative for 5030 soil samples, while 5035 soils can be preserved with sodium bisulfate or frozen. Water samples are preserved with 3 drops of 36% HCl to a pH <2. Water samples marked as un-preserved are analyzed within 7-days.

7.3 Sample Preparation / Screening /Analysis

Once the samples are logged into the TALs (LIMS) database upon receipt, a paperwork trail is initiated. The Supervisor or Analyst prints and prepares the necessary information (Sample Tracking Sheets) and places it in the appropriate file bin in the GC/MS VOA lab. The analysts take this information and subsequently screen the associated samples. Samples are screened by MSD prior to analysis. The actual screening procedures vary due to sample appearance, sample matrix, client history and analytical method. Once the samples are screened, the paperwork is transferred to a second file appropriately labeled. This file contains information about samples that have been screened but need to be reviewed. Once screened, an 'X' is placed on top of the vial, indicating both that the sample has been screened, and that the particular vial can not be used for subsequent analysis. The screened analysis can be reviewed on screen or hard-copy, as all screening data is collected and stored on the data system as with all GC/MS analyses. Upon review, the analyst makes decisions concerning the screen and indicates if an initial dilution is required. This information is physically recorded on the paperwork. Once reviewed, the paperwork is then placed in appropriate files that are broken down by matrix and method. The samples are now ready to be analyzed.

7.3.1 Waters

Allow samples and standards to come to ambient temperature. Observe all vials of sample and confirm the absence of any air bubbles. If a vial has an air bubble do not use that vial and notify the Supervisor of the situation. Make sure the vial used for screening is properly marked and do not use this vial for analysis.

Remove the plunger from a 5 mL luer-lock gas-tight syringe and fill to near over-flowing. Replace the plunger. The pH of all samples is verified at time of analysis. If the is pH < 2, a check-mark is placed in the appropriate column on the sample tracking sheet. If the pH > 2, the actual estimated pH is written in the same column. pH checks and verification of hold-times are documented on the review form. Samples lacking preservation may be noted in the case narrative. Invert the syringe, and adjust the volume to 5 mLs. Confirm the absence of all air bubbles.

Add IS, SS and methanol to the 5 mL syringe. Initial concentrations of both internal standard and surrogate solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. Immediately add the sample to a clean 40 mL vial using the method described in attachment 2 to analyze the sample.

If a batch is going to be analyzed, which is usually the case, load all samples, including all batch QC prepped in the same sequence and manner as the samples, following the procedure above. After the batch is loaded, replace all samples and standards back in storage. Appropriate documentation is made on the ICOC page.

If a dilution is required as indicated from the screening results, the following guidelines are followed. If the dilution is > 1/100 (250 uL of sample) an initial dilution is made into a volumetric flask. If serial dilutions are required, no less than 1 mL is taken for further dilutions. The final sample aliquot taken for analysis from the volumetric is no less than 250 uL. If the dilution is < 1/100, the appropriate sample amount is added directly to the 5 mL syringe.

Opened sample vials are used only once unless: (a) any necessary dilutions/reruns are done the same day or (b) there are no other vials for that sample.

7.3.2 Soils

As some clients still request method 5030 at the present time, soils are still being analyzed as indicated below. As clients convert to Method 5035 completely, this section will be removed.

Before weighing any samples, check the balance using the appropriate class weights. Record the actual weights in the Balance Logbook. If a problem is noted, contact the QC department.

Allow samples and standards to come to ambient temperature.

Weight out 5 grams of the sample into a clean 40-mL vial. Add 5-mL reagent water into vial. IS/SS will be added though the septum. The autosampler will add an additional 10-mLs of reagent water. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. Using the methods described in Attachment 1, analyze the sample. All soil samples are analyzed with a heated purge (40°C).

If a batch of samples is to be analyzed, prepare each as above. After the batch is loaded, including all batch QC prepped in the same sequence and manner as the samples, replace all samples and standards to their appropriate storage location. Documentation is made on the ICOC page.

Any sample that based on screening results/historical data has shown to contain high concentrations of compounds is analyzed at an initial dilution. Any sample that after the initial run contains targets above the calibration range is diluted to accurately quantitate those compounds. If an initial analysis over-diluted the given sample it is re-analyzed as a low level soil. If the low-level analysis contains compounds above the calibration range, and the same compounds are within range in the dilution, both sets of data may be reported to the client.

If a 1/2 or 1/5 dilution is required, 2.5g/1.0g of sample is weighed into the purge vessel.

7.3.3 Medium-Level Soil Extracts

If a larger dilution is required, a medium-level soil extract is prepared as follows. Five grams of sample is weighed into a tarred vial. Five 5-mLs of MeOH is added to the vial and the vial sealed. After the 24-48 hour contact time, the MeOH portion is decanted and stored in a 1-1.5 mL Teflon-lined screw-capped vial for storage. A portion of the extract (100 uL maximum) is taken for analysis. ISS and SSS will be added prior to analysis. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and surrogate tables provided in this SOP. Serial dilutions, if needed, are made from the extract and appropriate amounts taken for analysis.

If the sample upon which a medium-level prep as been performed also required an MS/MSD, the appropriate amount of MS solution is also added.

All samples prepared in this manner will be analyzed against a medium-level soil curve. The standards, blanks and LCS samples will contain 100 uL MeOH. The curve will be at ambient temperature.

NOTE: Some soils are analyzed initially at low levels due to increasing client requests for lower reporting limits. The same samples may then require large dilutions to bring compounds into the calibration range of the instrument. Some compounds, most notably the ketones, have very different responses when heated versus non-heated, despite the sample matrix. Traditionally, the lab heats soils. Therefore, the match between original analyses and dilutions for compounds such as these may not appear to correlate.

Dilution	Sample Weight	Vol. MeOH (1/2.5) Extract
1/2	2.5 grams	---
1/5	1.0 gram	---
1/50	5 grams / 5 mLs	100 uL
1/250	5 grams / 5 mLs	20 uL
1/500	5 grams / 5 mLs	10 uL

Using those parameters in Attachment 1, analyze all samples in the batch.

Sample vials/jars are only used once unless: (a) any dilutions/reruns are analyzed the same day or (b) there is only one jar for analysis.

7.3.4 Method 5035

NOTE: ICAL Standards are prepared with 5 mL milli-Q water.

Samples for low level VOA soil analysis may be received at the lab in one of three manners: First, as replicate 5 gram core samples in 40 mL vials containing organic free water and frozen within 48 hours of collection. Secondly, as replicate 5 gram core samples in 40 mL vials containing a Sodium Bisulfate preservative solution (refer to USP-5035 for collection/preservation). Thirdly, unpreserved 5 gram core samples may be received in Encore containers. These core samples must be placed in organic free water and frozen within 48-hours of collection. This time requirement is currently under review by appropriate regulatory agencies and may be extended beyond the 48-hours. Until such time, the laboratory will endeavor to "fix" the sample cores in preservative within 48-hours of collection. The laboratory may receive replicate 5 gram soil cores to be used for reanalysis if needed.

In addition to low level samples, an additional soil aliquot should be received for use as a screen and possible use as a mid-level extraction/analysis. This additional core must also be fixed in MeOH within 48-hours of collection. The amount of MeOH added must closely correspond to a soil to solvent ratio of 1:1. TestAmerica may adjust the methanol levels of any sample prepared in the laboratory from encore plugs with a corresponding 1:1 ratio of MeOH to soil. Any sample prepared in the field and fixed in methanol prior to arrival to the laboratory will not be adjusted but an NCM will document the discrepancies. Though not specified in the method, TestAmerica will pursue a goal of removing the MeOH from the soil within 24-48 hours after the initial extraction. A portion of the MeOH be removed and placed in an amber 1.5 - 2.0 mL Teflon-lined screw-capped vial for storage. This time limit should standardize the amount of time the MeOH comes in contact with the sample.

MeOH extracts of soils will be analyzed as stated above at ambient-temperature against a medium-level soil initial calibration. All surrogate and internal standard solutions will be added at time of analysis.

Low level soils will be analyzed using the Closed Purge and Trap Auto Sampler System. Surrogate and internal standard solutions will be added at the time of analysis through the septum by either a small gauge (10uL) syringe or automatically by the Archon autosampler. Initial concentrations of both surrogate and internal standard solutions shall be such that "sample concentrations" of the analytes conform to the method and the spike and surrogate tables provided in this SOP. The concentration of the solution and amounts spiked may vary depending on the precision obtained with a given solution/volume combination. However, the final concentrations of such compounds in the samples will follow the same guidelines as previously stated in this SOP for all other samples.

As with the internal standard and surrogate, all QC spike solutions must also be added to the closed sample container. This is accomplished by the addition of the spike solutions through the septum with a small gauge (10 uL) syringe just prior to the sample being placed on the instrument for analysis.

7.3.5 Drum/Waste Samples

- Non-MeOH Miscible
- MeOH Miscible

These samples are normally treated as medium level soils or waste dilutions. Waste dilutions normally consist of 5 grams of sample to 5 mLs of MeOH. If the sample is non-miscible with methanol, mix the sample and allow sample to separate. Draw off the methanol into a screw top vial. Use a portion of this methanol extract to screen. Continue to analyze the sample as high a high level methanol extract at a dilution based on the screening result.

If the sample is miscible with methanol, notify the PM and provide a detailed description of sample matrix and any matrix issues. The sample will automatically be given a 1:2 dilution factor, as a result of its miscibility with the methanol. Screen the sample and continue to analyze by the high level methanol extract procedure based on the screening results. Both ISS and SSS are added prior to analysis. The laboratory can also pre-spike the surrogates and spike compounds if client-specified to do so. In many cases, due to the high level dilutions required, the surrogates and matrix spike compounds may be diluted out. Therefore, unless specifically instructed, both solutions will be added at the time of analysis.

Drum/waste samples that are biphasic in nature require a discussion with the PM and client in order to determine how the samples will be prepared and analyzed (one phase, both phases?). The preparation and analysis of the sample will be documented.

7.4 Preventive Maintenance

Instrumental maintenance can be categorized as daily and "as required". Required maintenance may be performed for a variety of reasons. Certain trouble-flags will indicate what maintenance procedures may be required. A description of the situation, actions taken and follow-up must be documented in the instrument maintenance logbook on the day of maintenance and initialed / dated. An example Maintenance logbook page can be found in Attachment 3. Maintenance logs are to be peer reviewed by a qualified analyst other than the analyst who recorded the maintenance in the logbook. The maintenance logs should be reviewed on a monthly basis.

7.4.1 Daily Maintenance

The most routinely performed maintenance includes:

- Purge-line or sample transfer line rinses within the concentrator and vial autosampler
- Analysis of blanks after high level samples
- GC oven bake after high level samples

The Corrective Action/Qualification Report for GC/MS VOA is used to document that the instrument Preventive Maintenance as described in this SOP has been performed.

7.4.2 "As Required"

Most maintenance is done on an "as needed" basis, is operator determined and can be categorized as GC, Concentrator, or MS related.

7.4.2.1 GC Related

- change column; condition new column
- clean separator; change separator
- check helium flow rate
- change gas cylinders and moisture trap
- Replace liners

7.4.2.2 MS Related

- clean source/rods and anything associated with that activity
- replace electron multiplier
- change filaments

7.4.2.3 Concentrator Related

- change transfer line; clean transfer line
- replace trap; condition new trap
- run 20 ug/L Bromoform standard to check for formation of Chloromethane and Bromomethane.
- refurbish Concentrator
- check purge pressure and flow rate
- analysis of position blanks after high-level samples
- change bulk head fitting

7.4.2.4 Autosampler Related

- change sparge needle
- change pencil filters
- flush standard pickups
- calibrate standard valve
- run vial position calibration
- clean transfer rods
- oil bearings

7.5 Documentation/Tracking of Sample Analyses

7.5.1 The GC/MS VOA lab employs several forms that serve both a tracking and review function. The Sample Tracking Sheet is filled out for each job. It contains information the analyst needs as for method, QC requirements, special reporting requirements, screening results, methanol lot # etc.., in addition for space to track the analysis of every single sample in the job and the outcome of that analysis. The Sample Tracking Sheet can be found in Attachment 3.

7.5.2 In addition, all samples logged into the department appear on back-logs ordered by both Hold Time and Due Date. The back-logs are utilized by the analysts when making decisions as to methods and analyses that are needed for the day. As samples are analyzed and reviewed, the back-logs are updated to reflect those samples completely analyzed, those requiring dilutions and re-analyses.

7.5.3 In addition, an instrument sequence log is printed, reviewed, signed and bound in a log daily for each instrument.

7.6 Archival of Data

Data will be available on the local server for at least 1 year. No data is removed from the system until it has been archived on at least one full back up and a number of differential back-ups.

Data is transferred to servers after each sample run. The servers are backed up to a local backup server each night. The local backup server is backed-up off-site daily. Data can be restored from the backup server. Back-ups are the responsibility of the IT Manager. Clarification of procedures can be found in the *UP-IS-014,* TestAmerica Chicago IT Procedures and Processes SOP.

7.7 Removal of Data

There is a substantial amount of space available to both BNAs and VOAs on the current data system. Data older than approximately six months is zipped and remains on the system. At the IT Manager's, Technical Manager's or GCMS Supervisor's discretion, previously archived data is purged from the system.

8.0 QUALITY CONTROL

8.1 QC Summary

The department will review the quality controls as follows:

8.1.1 Method Blank (MB) / Laboratory Control Standard (LCS)

At least one MB and LCS will be included in each laboratory batch. Regardless of the matrix being processed, the LCS and MB will be in an aqueous media.

The MB will be examined to determine if contamination is being introduced in the laboratory. The LCS will be examined to determine accuracy and precision.

8.1.2 Accuracy

Accuracy will be measured by the percent recovery (%R) of the LCS. Method 8260B refers to Method 8000 for guidance on initial demonstration performance criteria. Guidelines for LCS/LCSD and MS/MSD accuracy limits can also be found in Method 8000. The laboratory's current in-house statistical limits can be found in Table 1. The number of compounds being used for bench level control and the accuracy limits assigned to those compounds may vary with client, QAP, project, or state certifications etc. This information is transmitted to the bench via the COC, kick-off meetings, tech profiles, job note or NCM etc.., and indicated on one of the forms used at the bench. In-house generated limits are subject to change.

8.1.3 Precision

Precision will be measured by the reproducibility of the LCS and will be calculated as Relative Percent Difference (RPD). Current limits are listed in Table 1. RPD's are not used to assess bench level Corrective Action.

8.1.4 Surrogates

Surrogate Compounds will be added to every sample to measure performance of the analysis. Method guidance limits are listed in Attachment 2. In-house statistical limits are listed in Table 1. Guidelines for the generation of statistical surrogate limits can be found in Method 8000. As with LCS samples, surrogate recovery limits may vary with client, QAP, project, etc.., and the information transmitted to the bench in the same manner.

8.1.5 QC Charting/Generation of Statistical Limits

Precision and accuracy are monitored using LCS data. Review of QC Charts and generation of in-house statistical criteria, including surrogate limits, is completed on an annual basis. Additional data may be added at QA/QC discretion during the year for other purposes. Spike levels are 50 ppb. Only routine compounds are spiked and should be representative of the whole. The more non-routine compounds are not part of the spiking solutions. Other limitations (availability of second source) may also prevent adding these to spiking solutions.

8.2 Corrective Actions

Listed below are the steps to be taken when an out-of-control situation occurs. The analyst must address the following issues as described below in the individual sections.

- demonstrate that all of the problems creating the out-of-control situation were addressed;
- document the problem and the action that was taken to correct the problem;
- document that an in-control situation has been achieved; and
- receive approval (signature) of the supervisor, project manager, QC personnel or other qualified personnel prior to release of data associated with the problem.

Corrective Actions are documented on the Corrective Action/Qualification Report included in the instrument logbook. In addition, a sample tracking form, specific to a unique job, is attached to the sample tracking documentation. The corrective action/qualification report and sample tracking form are used to note all out-of-control events, the actions taken to try and correct the problem, the return to control.

Discussed below are the suggested and required courses of action when an out-of-control situation has occurred.

8.2.1 BFB Criteria

If BFB criteria can not be met, determine if the source of the problem is instrumental or tune related. Inspect overall sensitivity, possible excessive background, the proportionality of the masses, relative abundances of the target masses. If it seems tune-related, adjust the tune parameters in Manual Tune slightly, until acceptance is achieved. If the problem seems instrumental, perform suggested trouble-shooting to locate and correct the problem (Suggestions can be found in most of the manuals). NO analysis can proceed until criteria are met. Corrective action for BFB analysis is documented on the corrective action/qualification report in the logbook.

8.2.2 Initial Calibration

If initial calibration can not be met, determine if the problem is analytical or instrumental. Some suggested questions to ask would be:

- were the standards prepared correctly?
- was the proper amount analyzed?
- check the chromatogram - did something happen on one or two analyses; i.e., a leak
- check the response factors - is one concentration level very high or low? re-analyze
- how old are the standards?

All calibration criteria must be met (Section 6.4). If the ICAL does not meet specified criteria, at minimum, the appropriate levels must be re-analyzed. If necessary, new standards should be prepared and the levels re-analyzed. During analysis of an initial calibration, documentation of the re-analyses of specific levels is required. See previous section outlining CA for minimum COD values as well. Refer to the TestAmerica Corporate Policy, CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points* (Attachment 7), for further guidance.

8.2.3 ICV/Continuing Calibration

If continuing calibration can not be met, determine if the problem is analytical or instrumental. Some suggestions:

- check the chromatography
- is overall sensitivity low?
- excessive background?
- how old is the standard?
- need a new 5-point?
- has the tune shifted?

Compare the relative abundances of 69, 131 and 219 from that day's manual tune to those on the day the initial calibration was analyzed. Slight adjustments to the tune may bring the standard in. Certain compounds will help indicate what the problem is.

All calibration criteria must be met (Section 6.5). If the ICV/CCAL does not meet specified criteria, at minimum the standard should be re-analyzed. A new standard may be prepared and then re-analyzed. If necessary, a new ICAL must be run. All corrective action taken for CCAL's must be recorded on the corrective action/qualification report and included in the logbook. Multiple ICV/CCVs without documented corrective action or valid reason for doing so, is not acceptable.

8.2.4 Method Blank (MB)

If the MB is/appears to be contaminated, re-analyze. If contamination is still present, the problem may be in one of the common elements, such as the trap, transfer line, port valve or column. Baking the trap/column and running position blanks may be necessary. If contamination has occurred beyond that, and maintenance is required (i.e., replace trap) it is documented in the Maintenance logbook. All corrective action taken for Method Blanks must be recorded on the corrective action/qualification report and included in the logbook. Under extenuating circumstances, if analysis continues, qualification must be made as to the positive hits above the RL for the compounds in question. Any associated samples analyzed in the tune must be noted. Any samples containing positive hits must be noted. IF, the samples containing positive hits can not be re-analyzed (i.e., past hold-time), the positive hits are flagged with "B" and the situation and data noted and qualified in a case narrative and/or Non-Conformance Memo (NCM). Methylene Chloride and Acetone are known lab contaminants; therefore intermittent low levels of these compounds may occasionally occur. Acetone and Methylene Chloride must be less than 3X the reporting limit. Any positive detects in the samples should be re-analyzed. If the samples can not be re-analyzed then the positive hits must be flagged with a "B" flag and the situation must be documented in a NCM.

8.2.5 Surrogates

All surrogate recoveries are calculated. If ANY surrogates are outside limits in the MB, it must be re-analyzed. Analyses CAN NOT proceed until an in-control situation is demonstrated. Re-analyze the blank. If surrogates are still out, the instrument may need to be re-tuned (BFB) and/or another calibration standard analyzed. If the problem persists, further maintenance action may be required (i.e., trap replacement, clean instrument).

Before pursuing other measures, check to be sure that:

- calculations are correct
- concentrations of the surrogates in the spiking solution are correct
- the correct amount of ISS/SSS solution was added
- ISS/SSS areas are reasonable

If any surrogates in a sample are outside limits, check the above first. Any sample that has a surrogate out must be re-analyzed. The re-analysis can take the form of a dilution, if there is reasonable expectation that a high concentration of a target compound is causing a matrix effect. If the surrogate(s) is/are still outside limits, a matrix effect is demonstrated and both reports are submitted. Depending on the client, the best result may be reported and the other result narrated. If all surrogates are in-control on the re-analysis, only the second analysis is reported.

Every effort is made to complete the re-analysis within hold-time. If this is impossible (i.e., capacity hold-times preclude re-analyses within hold-time), both reports may be submitted. This is documented in the narrative.

If the sample with the out-of-control surrogates is the same sample on which the MS and MSD were performed, and the pattern is duplicated, then re-analysis is NOT required. Documentation of the similarities is required.

Surrogate corrective action is documented on the sample tracking form for samples.

8.2.6 Laboratory Control Sample (LCS)

As specified in Section 8.1.2, the number of compounds and the limits used to assess accuracy vary with client, QAP, project etc. The in-house generated limits are listed in Table 1. In-house limits are subject to change. The need and course of corrective action varies with the number of compounds being used for bench control and positive detected of compounds outside limits. The LCS limits are based on the mean recovery +/- 3 standard deviations; therefore, it is statistically quite likely that there will be exceedances of the limits for a few analytes when a large number of compounds are included in the LCS. Therefore a number of individual analytes are allowed to exceed the LCS control limit before the LCS as a whole is considered to have failed. The control limit for a marginal exceedance is mean +/- 4 standard deviations which results in a larger range of +/-10% outside the in-house generated limits.

Number of analytes in LCS	Number of marginal exceedances allowed
> 90	5
71-90	4
51-70	3
31-50	2
11-30	1
< 11	0

- For QAP's specifying five compounds-- All five compounds must be within limits for analysis to proceed. The LCS samples may be re-analyzed. New spike solutions may be prepared. Or new standards or CCAL's may be analyzed. All corrective action and return to control must be documented at the time on the corrective action/qualification report and included in the logbook. The actual limits used for the five compounds may be QAP-specific (usually those listed in the table in the appendix) or in-house generated by matrix and method. In either case, the above corrective action and required documentation apply.
- For full list spikes, all recoveries are assessed, although no immediate corrective action may be required if within the marginal excedance. If the recoveries are low, in general another LCS may be re-analyzed. The spike solution and standard may be verified for correct concentrations. However, no corrective action is absolutely required by the bench unless an error is discovered. The recoveries may or may not be documented in a NCM, however, they are noted on the review form.

- Although not strictly required to take immediate corrective action, the purpose of the full-spike is two-fold in that the bench should use it as an indicator of the status of the calibration standards, instrument conditions etc., as well as a tool for data interpretation. Therefore, in keeping with good lab practice, the situation should be noted and assessed and any corrective action deemed necessary should be taken within a reasonable amount of time (Example: High recoveries on gases => new calibration standard may be needed).

- For samples that originate in the state of South Carolina, all compounds in the LCS must recover between 70-130% unless the compounds are identified as poor purgers in the SOP. The recoveries for the poor purgers should be within 60-140%. Note: South Carolina does not allow for the use of marginal exceedances. If the sample results are non-detect and the LCS recovery is above the 130% upper control limit for an analyte (140% for poor purgers), the analyst will contact the PM to obtain client approval to report the data with narration. If approval is not obtained, re-analysis of all associated samples will be performed.

8.2.7 Matrix Spikes (MS)

As specified in Section 8.1.2, the number of compounds and the limits used to assess accuracy vary with client, QAP, project, state certifications etc. In-house generated limits are listed in Table 1. In-house limits are subject to change. The need and course of corrective action varies with the number of compounds being used for bench control and recoveries of same compounds in the associated LCS samples. The following guidelines are used:

(1) QAP's etc., specifying 5 compounds.

- ALL 5 compounds are assessed. If recoveries are outside limits, the LCS is reviewed for those compounds. If the recoveries are within limits in the associated LCS samples, no further action is required. See above section concerning LCS corrective action for further information and action required for recoveries outside limits in LCS samples.

- The actual limits used for the five compounds may be QAP-specific (usually those listed in the table in the appendix) or in-house generated by matrix and method. In either case, corrective action and required documentation apply.

- For all other compounds in the full-list spike, all recoveries are assessed, although no immediate corrective action may be required. The affected compounds may be compared to the same compounds in the associated LCS samples. See the above section for further information and action required for these compounds in the LCS samples. The recoveries may or may not be documented in the Job narrative, however, they are noted on the review form. The recoveries of the "un-controlled" compounds may be used for data interpretation.

8.2.8 Internal Standard Policy

Method 8260 does not require re-analyses of samples for low internal standard areas. However, it is TestAmerica's policy to monitor areas and retention times, therefore, the following guidelines apply.

Situations requiring re-analyses :

- If ALL areas are outside limits the sample will be re-analyzed.
- Any sample that has a positive hit associated with any internal standard outside limits will be re-analyzed.
- If ANY surrogates are outside limits the sample will be re-analyzed.

Situations NOT requiring re-analyses :

- If all surrogates are within limits and there are no positive hits associated with those internal that are outside limits, the sample does not have to be re-analyzed. Situation should be addressed in a NCM.
- If all surrogates are within limits, but there is an obvious matrix effect occurring, even if positive hits are noted, the sample does not need to be re-analyzed. This decision will be approved by the supervisor. The situation should be addressed in a NCM.
- If there is historical evidence that shows a repeated pattern for a certain client and site, and this can be documented by reviewing past projects, the samples do not have to be re-analyzed. This decision will be approved by the supervisor and documented in a NCM.
- Corrective action for internal standard areas for samples is documented on the sample tracking form.

Any sample showing retention times outside windows will be re-analyzed. This is documented in the appropriate manner as in the preceding paragraph.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Computer Data Production/Reduction

The Chrom software produces a Total Ion Chromatogram (TIC), header, quant report and background subtracted spectra. For those clients requiring it, a 5 tentatively identified compound (TIC) search is also performed. The data system will produce an integration listing and tentative identification of each hit found at the selected percentage of the largest peak present.

9.1.1 Quantitation of Target Compounds

Quantitation of the target compounds is performed by the data system can be accomplished as follows:

WATERs: $$\text{concentration (mg/L)} = \frac{[A_x \times I_s]}{[A_{is} \times RF]} \times DF$$

Where:
A_x = area of characteristic ion for target
I_s = concentration of internal standard (ng)
A_{is} = area of characteristic ion for int. std.
RF = response factor for target
DF = dilution factor (if any)

SOILs: $$\text{concentration (mg/kg)} = \frac{[A_x \times I_s]}{[A_{is} \times RF \times D]} \times DF$$

Where:
All variables are equal and
D = (100 - % moisture in sample/100) or 1 for wet weight. (As in the case of drum samples)

The target methods all contain calculations for waters and soils that allow automatic processing and calculations of concentrations to be completed. The user may enter some variables (Dilution Factor) and others are imported from LIMS. Sample prep info for VOA's is entered directly into LIMS. The sample volume is considered to be "constant" for calculation purposes. Less sample volume (in the case of waters) and soil weight (in the case of soils) are taken into account in the dilution factor entered by the user. For medium-level soils and waste/drum type samples medium level calculations are needed and actual weights are brought into LIMS.

NOTE: As noted previously, weights are recorded to 0.1 gram. It is SOP to weigh out 5.0 grams (or as appropriate for the dilution), however, to keep data entry and calculations simple. The same holds true for all water volumes.

SOILs_High Level Methanol:

SW-846 8000C, Section 11.10.5 states the following:

Solid samples with a significant moisture content (>10%), designated for volatile organic analysis, that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. This total volume is then expressed as V_t in the sample concentration calculations provided. Therefore, in order to report results for volatiles analysis of samples containing significant moisture content on an "as received" basis, the calculated concentration needs to be corrected using the total solvent/water mixture volume represented as V_t. The total solvent/water volume is calculated as follows:

uL solvent/water V_t = [mL of solvent + ((%moisture/100) x g of sample)] x 1000 uL/mL

Dry Weight Concentration ug/kg = $\frac{(X_s)(V_t)(D)}{(W_s)}$ / (%Solids / 100)

X_s = Calculated concentration of the analyte (ng/uL) in the sample.
V_t = Total volume of the concentrated extract (uL)
D = Dilution Factor
W_s = Weight of the sample extracted or purged (g)

Generally, it is recommended that the calculated concentrations of volatile organics samples that are solvent extracted in a water-miscible solvent/water dilution effect for situations when the sample moisture content is greater than 10%. The potential under reporting of volatile concentrations is more pronounced as the percent moisture content increases.

9.1.2 Accuracy: $\%R = \frac{(A_T - A_0)}{A_F} \times 100$

Where:
A_T = Total amount recovered in fortified sample
A_0 = Amount recovered in unfortified sample
A_F = Amount added to sample

9.1.3 Precision: $\%D = \frac{|B_1 - B_2|}{B_1} \times 100$

$RPD = \frac{|B_1 - B_2|}{(B_1 + B_2)/2} \times 100$

Where:
B_1 = %Recovery MS (or LCS)
B_2 = %Recovered MSD (or LCS)

9.1.4 Modifications for 8260B quantitation

9.1.4.1 Initial Calibration Criteria

Methods 8000B/8260B require the use of linear or higher order calibration curves for those compounds exceeding 15%.

The following equations apply :

Linear Regression: $y = a_0 + a_1 * x$ Quadratic Curve: $y = a_o + (a1 * x) + (a2 * x^2)$
Weighted Linear Regression: $y = a_1 * x + a_0$

Where: $x = Area_{UNK}/Area_{ISTD}$
$y = Amount_{UNK}/Amount_{ISTD}$
a_1 = slope
a_0 = y-intercept

The equation for weighted linear regression follows the linear regression but introduces a weighting factor for the slope and y-intercept. The User manually enters the weighting factor into the method as: 1/Amt or $1/Amt^2$

Once the $Amount_{UNK}$ is solved, the value is adjusted for total solids, dilution factors etc.., to calculate a final concentration.

The quantitation of compounds using linear regressions and quadratic curves as performed automatically by the Target software has been confirmed to be accurate.

Method 8000B/8260B specifies a minimum COD(R^2). The corrective action regarding an initial calibration for method 8260B as it relates to the 0.990 correlation coefficient acceptance criteria is outlined. When a compound has a correlation coefficient less than 0.990, the occurrence is documented by the analyst in the Corrective Action section of the instrument's logbook. Any corrective action or data qualification is also documented on the corrective action/qualification report and included with the logbook. All corrective actions taken may include those listed below.

Samples may be analyzed against an initial calibration that have compounds with a correlation coefficient less than 0.990 and the corrective actions taken may also include some but not all of the following:

- The data for these samples may be reported without qualification if the compounds with a correlation coefficient less than 0.990 are not detected in the sample; therefore no further corrective action is required.
- If a compound is detected in the sample that has a correlation coefficient less than 0.990, the samples may be reanalyzed against an initial calibration with an acceptable correlation coefficient and only the reanalysis will be reported on the sample. If this reanalysis occurs beyond analysis hold times then both analyses on the sample will be reported.
- If a compound is detected in the sample that has a correlation coefficient less than 0.990, the decision to reanalyze or to reported the data without further corrective action is made on a case by case basis with the approval of the supervisor, the project manager and the client. The sample results may require qualification for this compound on the report and will be addressed in the case narrative.

9.1.4.2 Continuing Calibration Check

Prior to sample analysis a 50 ppb calibration check is completed. All minimum RF's must meet same limits. All CCC's must be less than 20% Drift as calculated below; the analysts may verify %DIFF and only calculate those that are close. (Error may only be made in favor of tighter control).

$$\%Drift = \frac{(Ci - Cc)}{Ci} \times 100$$

Where:
Ci = standard conc. (10/50)
Cc = measured conc. in cal check

9.1.5 Quantitation of TIC's (Tentatively Identified Compounds)

Quantitation of TIC's is performed by the Chrom processing software. The formulas above for waters and soils can be used with the following modifications. A_x and A_{is} should be taken from the total ion integration listing accompanying the TIC report produced by the data system. The nearest non-interfered with internal standard should be used. The RF is assumed to be one (1). The concentration is therefore an estimate and is flagged as such with a "J". Any TIC also found in the MB is flagged with a "JB". Any TIC identified with a CAS number is also flagged with an "N", indicating that the ID was based on the mass spectra. The operator should visually confirm that the integration is correct. If not, the peak in question must be manually integrated. The Chrom data system automatically calculates the actual concentration of the TIC's, including dilutions and total solids, once that information is retrieved from TALs (LIMS).

9.2 Operator Data Reduction/Review

The operator does on-screen review of all data and

- makes judgments concerning the "realness" of those target compounds found and
- makes judgments concerning the identification of the tentatively identified compounds
- modifies the output to produce a data package reflective of those decisions

9.2.1 Initial Review

The GC/MS VOA area uses two kinds of corrective action documentation. The first consists of the Batch Information section in Analyst Desktop in TALs (LIMs). This area in TALs contains a comment section to report out-of-control situations, corrective action and return-to-control for documenting problems related to general QC: tune, ICAL, CCAL, internal standard areas from CCAL to ICAL, and LCS samples. The second are the sample tracking forms that refer to a single job. These are used to record events, corrective actions and final actions for surrogates, internal standard areas, carry-over situations, analyses past tune time, MS/MSD data etc.., for each sample in the batch. These forms are attached to the other sample documentation that accompanies the job through analysis. Both may be used during initial review of the data. See Section 8.2 of this SOP for details on Corrective Action.

All data is initially reviewed on-screen. The review is both a QC review and a general review as described below.

- The MB contains no interferences or target compounds at the RL.
- ALL surrogates are in control in the blank. Surrogate limits are listed in Table 1;
- ALL surrogates in samples are in control;

- LCS recoveries meet the limits listed in Table 1. See Section 8.2 concerning compounds and limits for LCS samples. In-house limits have been generated and are in use.
- Internal standard areas and retention times are checked and meet guidelines. Limits are listed in Attachment 2. Additional guidelines can be found in Section 8.2.
- The sample does not require any further dilutions or analysis at a more concentrated level. Dilutions are made to keep the target in the upper half of the calibration range. The MS and MSD are never diluted to get spiked or non-spiked compounds within range, as this would reduce the matrix affect assessment.
- Visually confirm complete integration for any large and/or saturated target compounds.
- The sample does not require re-analysis for any other reason (i.e., leak, analysis past tune time, ISTD areas low, etc.).

9.2.2 Identification of Targets

The following guidelines are used in the positive identification of target compounds.

1. "elution of component at the same relative retention time as the standard component."

The elution times should compare within +- 30 s. The standard must be run on the same 12 hour period as the sample. If co-eluting analytes interfere with the comparisons of retention times, other ions characteristic to that compound can be used to confirm relative retention times.

2. "correspondence of the sample component and standard component mass spectrum." Comparisons of sample spectra to standard spectra must be made using standard spectra obtained from the GC/MS system.

All ions present in the standard spectrum at a 10% relative intensity (most abundant ion being 100%) should be present in the sample.

The relative intensities of the above ions should agree within ±20%, between the standard and sample. If an ion is 50% intensity in the standard the corresponding ion must be between 30 and 70% in the sample.

Ions >10% in the sample but not present in the standard should be considered and accounted for.

3. Operator judgment. If a compound can not be verified by the above, but in the operators technical judgment the ID is correct, it is reported as such.

- Once all positive identification is made, the file is modified to reflect these decisions. At this time TIC's may also be reviewed and name. In each case where the file has been edited or manual integrations have taken place the operator must identify, initial and date the changes on the hardcopy (if such is generated).

9.2.3 Manual Integration Policy

In each case where the file has been edited or manual integrations have been performed the operator must identify, initial, and date the changes on the hardcopy report. The following guidelines apply:

- Manual integrations should be consistent between all files integrated.
- Manual integrations should not be performed to meet QC criteria.
- Manual integrations are automatically flagged with an 'M' on the raw data.
- Excessive manual integrations may reflect an instrumental or methodological problem that should be addressed.

- Manual integrations shall follow the TestAmerica Corporate SOP for Manual Integrations (CA-Q-S-002 – Attachment 8). Example integrations and documentation are provided within this attachment.

Manual integrations are most often performed for the following reasons:

- Assignment of correct peak that was mis-identified by the data system.
- Incomplete auto-integration due to high level of target compound detected.
- Incomplete auto-integration due to background interference.
- Incorrect auto-integration due to co-elution or near co-elution of compounds.
- Missed peaks.

All manual integrations are reviewed, initialed and dated. For those clients requiring full data packages, spectra and Extracted Ion Chromatography Profiles (EICP) are printed for all manually integrated compounds. Manual integrations are documented in the case narrative and a Manual Integration Summary is included in the data package.

9.2.4 Identification of TIC's

In general, up to as many as 5 non-target compounds are tentatively identified by the data system and operator. Compounds with responses >10% of the nearest ISTD are identified. The data system provides the operator with a SUB ADC C sample spectrum, spectra of the first three matches and a listing of two other possibilities. Molecular formulas, molecular weights and CAS #'s are included. The following guidelines are used:

Relative intensities of major ions in the reference spectrum should be present in the sample (ions >10%).

- Relative ions should agree within ±20%;
- Molecular ions in the reference should be in the samples;
- Review the possibility of background and/or co-eluting compounds for those ions present in the sample but not in the standard;
- If ions are present in the sample but not in the standard, review the possibility of the presence of background or co-eluting compounds;
- If ions are present in the standard but not in the sample, review the possibility that the ions were subtracted out because they are also common to the background or co-eluting compounds;
- In the event no valid interpretation can be made, the compound is called "unknown".
- Interpretation can be often narrowed down to a class of compounds, molecular formula or weight.

9.3 Final Review

9.3.1 Once (a) the analysis is determined to be acceptable and (b) the initial review and data reduction has occurred and (c) the analyst has entered sample prep info into TALs (LIMS), the following steps occur. The sample prep information, client ID information and some data applicable to fields in the forms is retrieved from TALs (LIMS).

9.3.2 All necessary forms are then generated using the TALs. The package is then assembled and ready for the first review. For level 2 data reports and similar deliverables other data may be generated for review purposes. In these cases, final packages with raw data and forms are not generated. Review of all reports and associated data is required regardless of data deliverable level.

9.3.3 Analytical data goes through a 200% review cycle. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The analyst transfers the data into TALs in the Analyst Desktop module. Where non-compliance is observed, the analyst creates Non-Conformance Memos (NCMs) in TALs. Flags and data qualifiers can be method, project, program or QAPP specific. The analyst documents the initial review on a data review checklist (Attachment 6) and sets the batch status in LIMs to 2nd level. The peer/supervisor review of the data is conducted by another individual who has been trained on the review process or by the department supervisor. This secondary review is documented on the same checklist, making any necessary corrections to the data or additions to the NCMs as necessary. The batch is then set to lab complete. Any Spectra and all manual integrations are reviewed. For the organic instruments, manual integrations may also be electronically reviewed utilizing auditing software to help ensure compliance to the ethics and manual integration policies. The raw data, including the checklist, instrument print-outs, and manual entries, and electronic files are retained for easy retrieval in accordance with the laboratory's record and retention policy outlined in the SOP, *UP-QA-QAM, Section 15.*

Examples of items included in the above reviews are as follows:

- QC data are outside the specified control limits for accuracy and precision
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration (if applicable)
- Transcription errors
- Results outside of calibration range

Note: The complete analysis scheme can be summarized below (Section 7.1.1 & 7.1.2) and in Attachment 5. The entire sample tracking system can be summarized in Attachment 5.

10.0 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). Employees must abide by the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

10.1 Waste Management

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 UP-WM-001.

The following waste streams are produced when this method is carried out.

- Methanol Waste from this procedure will enter the 'Flammable Vials' wastestream.
- Laboratory generated solid and aqueous waste water remaining from this method is to be disposed of in the carboys labeled Waste Water or Solid Waste.
- All expired standards are to be turned over to the waste technicians or the EHSC for disposal.

11.0 METHOD PERFORMANCE CRITERIA

Refer to Sections 1, 6, 7 and 8.

12.0 REFERENCES

Refer to Section 1.0

13.0 ATTACHMENTS

Table 1. Practical Quantitation Limits for Volatile Analytes; Laboratory Statistical Control Limits/ Surrogate Recovery Guidelines
Table 2. Characteristic Mass for Purgeable Organics Compounds

Figure 1. Example: Total Ion Chromatogram for 5 mL Purge Water
Figure 2. Example: Total Ion Chromatogram for 5 mL Purge Soil

Attachment 1. Example: Initial Calibration Guides; Method Listings; Concentrator Conditions; Flow Settings
Attachment 2. Example: Target and Internal Standards; Internal Standard Guidelines; Initial Calibration (Form 6)
Attachment 3. Example: Sample Run Log; Corrective Action/Qualification Report; GC/MS VOA Maintenance Logbook; Sample Tracking Sheet; GC/MS VOA-ICOC Form
Attachment 4. Example: Continuing Calibration Evaluation and Acceptance Criteria (Form 7)
Attachment 5. Example: Analysis and Sample Tracking Flowcharts
Attachment 6. Example: Data Review Checklist
Attachment 7. CA-Q-P-003: TestAmerica Corporate Policy: Calibraton Curves and the Selection of Calibration Points
Attachment 8. CA-Q-S-002: TestAmerica Corporate SOP: Acceptable Manual Integration Practices
Attachment 9. List of Poor Purging or Poorly Performing Compounds

14.0 REVISION HISTORY

- Revision 26, was updated on 11/30/15
- Annual Review
- Section 4.1 was updated to reflect current equipment listing
- Sections 5.3, 5.4 and 5.5 were update with current standard concentrations and preparation volumes
- Section 10.1 was updated to include additional wastesteams.

Table 1.

Example: Practical Quantitation Limits for Volatile Analytes[a] Laboratory Statistical Control Limits and Surrogate Recovery Guidelines (041-001 to 041-006)

[a]Practical Quantitation Limit (PQL) – The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The PQL is generally 3 to 5 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the PQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable. See the following example information for further guidance on matrix-dependent PQLs.

[b]PQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, PQLs will be higher, based on the percent dry weight in each sample.

Note: the 8260B high level Methanol (5035A_M_Calc) RL, LOD and MDL limits listed on the attached summary have a 50x dilution factor applied to them in the analytical batch.

Analyte Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOQ	Units	LCS Low	LCS High	LCS - RPD %	MS Low	MS High	MS - RPD %	Surrogate Low	Surrogate High
MDL W GCMS	Volatile Organic Compo	8260B	5030B	1,1,1,2-Tetrachloroethane	630-20-6	1.00	0.462	0.500	ug/L	70	124	20	70	124	20		
				1,1,1-Trichloroethane	71-55-6	1.00	0.379	0.500	ug/L	70	125	20	70	125	20		
				1,1,2,2-Tetrachloroethane	79-34-5	1.00	0.398	0.500	ug/L	68	133	20	68	133	20		
				1,1,2-Trichloro-1,2,2-trifluoroet	76-13-1	1.00	0.460	0.500	ug/L	64	120	20	64	120	20		
				1,1,2-Trichloroethane	79-00-5	1.00	0.351	0.500	ug/L	70	125	20	70	125	20		
				1,1-Dichloroethane	75-34-3	1.00	0.410	0.500	ug/L	70	127	20	70	127	20		
				1,1-Dichloroethene	75-35-4	1.00	0.391	0.500	ug/L	68	121	20	68	121	20		
				1,1-Dichloropropene	563-58-6	1.00	0.297	0.500	ug/L	70	126	20	70	126	20		
				1,2,3-Trichlorobenzene	87-61-6	1.00	0.458	0.500	ug/L	70	133	20	70	133	20		
				1,2,3-Trichloropropane	96-18-4	1.00	0.414	0.500	ug/L	53	139	20	53	139	20		
				1,2,4-Trichlorobenzene	120-82-1	1.00	0.342	0.500	ug/L	70	125	20	70	125	20		
				1,2,4-Trimethylbenzene	95-63-6	1.00	0.358	0.500	ug/L	70	127	20	70	127	20		
				1,2-Dibromo-3-Chloropropane	96-12-8	5.00	1.99	2.50	ug/L	59	139	20	59	139	20		
				1,2-Dibromoethane	106-93-4	1.00	0.386	0.500	ug/L	70	124	20	70	124	20		
				1,2-Dichlorobenzene	95-50-1	1.00	0.334	0.500	ug/L	70	123	20	70	123	20		
				1,2-Dichloroethane	107-06-2	1.00	0.392	0.500	ug/L	66	132	20	66	132	20		
				1,2-Dichloropropane	78-87-5	1.00	0.428	0.500	ug/L	70	127	20	70	127	20		
				1,3,5-Trimethylbenzene	108-67-8	1.00	0.254	0.500	ug/L	70	129	20	70	129	20		
				1,3-Dichlorobenzene	541-73-1	1.00	0.400	0.500	ug/L	70	122	20	70	122	20		
				1,3-Dichloropropane	142-28-9	1.00	0.361	0.500	ug/L	70	127	20	70	127	20		
				1,4-Dichlorobenzene	106-46-7	1.00	0.364	0.500	ug/L	70	120	20	70	120	20		
				1,4-Dioxane	123-91-1	100	41.1	50.0	ug/L	50	150	20	50	150	20		
				2,2-Dichloropropane	594-20-7	1.00	0.444	0.500	ug/L	68	120	20	68	120	20		
				2-Chloroethyl vinyl ether	110-75-8	2.00	0.766	1.00	ug/L	57	145	20	57	145	20		
				2-Chlorotoluene	95-49-8	1.00	0.314	0.500	ug/L	70	128	20	70	128	20		
				2-Hexanone	591-78-6	5.00	1.56	2.50	ug/L	53	140	20	53	140	20		
				3-Chloropropene	107-05-1	2.50	0.863	1.20	ug/L	50	150	20	50	150	20		
				4-Chlorotoluene	106-43-4	1.00	0.349	0.500	ug/L	70	127	20	70	127	20		
				Acetone	67-64-1	5.00	1.73	2.50	ug/L	47	131	20	47	131	20		
				Acrolein	107-02-8	100	22.6	50.0	ug/L	24	120	20	24	120	20		
				Acrylonitrile	107-13-1	20.0	4.45	10.0	ug/L	68	127	20	68	127	20		
				Benzene	71-43-2	0.500	0.146	0.250	ug/L	70	120	20	70	120	20		
				Bromobenzene	108-86-1	1.00	0.356	0.500	ug/L	70	129	20	70	129	20		
				Bromochloromethane	74-97-5	1.00	0.429	0.500	ug/L	70	121	20	70	121	20		
				Bromodichloromethane	75-27-4	1.00	0.372	0.500	ug/L	70	127	20	70	127	20		
				Bromoform	75-25-2	1.00	0.484	0.500	ug/L	70	135	20	70	135	20		
				Bromomethane	74-83-9	2.00	0.797	1.00	ug/L	30	170	20	30	170	20		
				Butadiene	106-99-0	1.00	0.474	0.500	ug/L	50	150	20	50	150	20		
				Carbon disulfide	75-15-0	2.00	0.448	1.00	ug/L	61	120	20	61	120	20		
				Carbon tetrachloride	56-23-5	1.00	0.384	0.500	ug/L	70	136	20	70	136	20		
				Chlorobenzene	108-90-7	1.00	0.385	0.500	ug/L	70	120	20	70	120	20		
				Chloroethane	75-00-3	1.00	0.505	0.505	ug/L	40	150	20	40	150	20		
				Chloroform	67-66-3	1.00	0.370	0.500	ug/L	70	120	20	70	120	20		
				Chloromethane	74-87-3	1.00	0.320	0.500	ug/L	45	140	20	45	140	20		
				cis-1,2-Dichloroethene	156-59-2	1.00	0.409	0.500	ug/L	70	120	20	70	120	20		
				cis-1,3-Dichloropropene	10061-01-5	1.00	0.417	0.500	ug/L	70	122	20	70	122	20		
				Cyclohexane	110-82-7	1.00	0.485	0.500	ug/L	60	133	20	60	133	20		
				Dibromochloromethane	124-48-1	1.00	0.488	0.500	ug/L	70	120	20	70	120	20		
				Dibromomethane	74-95-3	1.00	0.271	0.500	ug/L	70	120	20	70	120	20		
				Dichlorodifluoromethane	75-71-8	2.00	0.674	1.00	ug/L	30	150	20	30	150	20		
				Dichlorofluoromethane	75-43-4	1.00	0.377	0.500	ug/L	50	150	20	50	150	20		
				Ethyl ether	60-29-7	1.00	0.453	0.500	ug/L	69	125	20	69	125	20		
				Ethyl methacrylate	97-63-2	2.50	0.525	1.20	ug/L	50	150	20	50	150	20		
				Ethylbenzene	100-41-4	0.500	0.183	0.250	ug/L	70	125	20	70	125	20		
				Hexachlorobutadiene	87-68-3	1.00	0.446	0.500	ug/L	70	138	20	70	138	20		
				Hexane	110-54-3	1.00	0.491	0.500	ug/L	55	131	20	55	131	20		
				Iodomethane	74-88-4	2.50	0.662	1.20	ug/L	66	124	20	66	124	20		
				Isobutyl alcohol	78-83-1	100	35.7	50.0	ug/L	50	150	20	50	150	20		
				Isopropylbenzene	98-82-8	1.00	0.385	0.500	ug/L	70	132	20	70	132	20		
				m&p-Xylene	179601-23-1	1.00	0.182	0.500	ug/L	70	120	20	70	120	20		
				Methyl acetate	79-20-9	5.00	2.02	2.50	ug/L	56	131	20	56	131	20		
				Methyl Ethyl Ketone	78-93-3	5.00	2.12	2.50	ug/L	51	134	20	51	134	20		
				methyl isobutyl ketone	108-10-1	5.00	2.15	2.50	ug/L	53	135	20	53	135	20		

(Q4-201)

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS Low	LCS High	LCS RPD %	MS Low	MS High	MS RPD %	Surrogate Low	Surrogate High
				Methyl tert-butyl ether	1634-04-4	1.00	0.394	0.500	ug/L	65	120	20	65	120	20		
				Methylcyclohexane	108-87-2	1.00	0.317	0.500	ug/L	60	140	20	60	140	20		
				Methylene Chloride	75-09-2	5.00	1.63	2.50	ug/L	70	120	20	70	120	20		
				Naphthalene	91-20-3	1.00	0.335	0.500	ug/L	59	143	20	59	143	20		
				n-Butylbenzene	104-51-8	1.00	0.389	0.500	ug/L	70	129	20	70	129	20		
				n-Heptane	142-82-5	1.00	0.418	0.500	ug/L	64	132	20	64	132	20		
				N-Propylbenzene	103-65-1	1.00	0.414	0.500	ug/L	70	132	20	70	132	20		
				o-Xylene	95-47-6	0.500	0.219	0.250	ug/L	70	120	20	70	120	20		
				p-Isopropyltoluene	99-87-6	1.00	0.362	0.500	ug/L	70	133	20	70	133	20		
				sec-Butylbenzene	135-98-8	1.00	0.399	0.500	ug/L	70	134	20	70	134	20		
				Styrene	100-42-5	1.00	0.386	0.500	ug/L	70	120	20	70	120	20		
				tert-Butyl alcohol	75-65-0	50.0	12.1	25.0	ug/L	50	150	20	50	150	20		
				tert-Butylbenzene	98-06-6	1.00	0.398	0.500	ug/L	70	137	20	70	137	20		
				Tetrachloroethene	127-18-4	1.00	0.370	0.500	ug/L	70	129	20	70	129	20		
				Tetrahydrofuran	109-99-9	5.00	1.88	2.50	ug/L	50	138	20	50	138	20		
				Toluene	108-88-3	0.500	0.152	0.250	ug/L	70	120	20	70	120	20		
				trans-1,2-Dichloroethene	156-60-5	1.00	0.349	0.500	ug/L	70	120	20	70	120	20		
				trans-1,3-Dichloropropene	10061-02-6	1.00	0.362	0.500	ug/L	70	123	20	70	123	20		
				trans-1,4-Dichloro-2-butene	110-57-6	5.00	1.19	5.00	ug/L	50	150	20	50	150	20		
				Trichloroethene	79-01-6	0.500	0.164	0.250	ug/L	70	122	20	70	122	20		
				Trichlorofluoromethane	75-69-4	1.00	0.427	0.500	ug/L	65	134	20	65	134	20		
				Vinyl acetate	108-05-4	2.00	0.905	1.00	ug/L	30	150	20	30	150	20		
				Vinyl chloride	75-01-4	0.500	0.204	0.250	ug/L	63	127	20	63	127	20		
				1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/L							75	125
				4-Bromofluorobenzene (Surr)	460-00-4				ug/L							75	120
				Dibromofluoromethane	1868-53-7				ug/L							75	120
				Toluene-d8 (Surr)	2037-26-5				ug/L							75	120

(E90-140)

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD	MS - Low	MS - High	MS - RPD	Surrogate - Low	Surrogate - High
MDL S MSVOA	Volatile Organic Compd	8260B	5035A_M_Calc	1,1,1,2-Tetrachloroethane	630-20-6	1.00	0.462	1.00	ug/Kg	70	124	30	70	124	30		
				1,1,1-Trichloroethane	71-55-6	1.00	0.380	0.500	ug/Kg	70	125	30	70	125	30		
				1,1,2,2-Tetrachloroethane	79-34-5	1.00	0.398	0.500	ug/Kg	68	133	30	68	133	30		
				1,1,2-Trichloro-1,2,2-trifluoroet	76-13-1	1.00	0.460	0.500	ug/Kg	64	120	30	64	120	30		
				1,1,2-Trichloroethane	79-00-5	1.00	0.352	0.500	ug/Kg	70	125	30	70	125	30		
				1,1-Dichloroethane	75-34-3	1.00	0.410	0.500	ug/Kg	70	127	30	70	127	30		
				1,1-Dichloroethene	75-35-4	1.00	0.390	0.500	ug/Kg	68	121	30	68	121	30		
				1,1-Dichloropropene	563-58-6	1.00	0.298	0.500	ug/Kg	70	126	30	70	126	30		
				1,2,3-Trichlorobenzene	87-61-6	1.00	0.458	1.00	ug/Kg	70	133	30	70	133	30		
				1,2,3-Trichloropropane	96-18-4	1.00	0.414	0.500	ug/Kg	53	139	30	53	139	30		
				1,2,4-Trichlorobenzene	120-82-1	1.00	0.342	1.00	ug/Kg	70	125	30	70	125	30		
				1,2,4-Trimethylbenzene	95-63-6	1.00	0.358	1.00	ug/Kg	70	127	30	70	127	30		
				1,2-Dibromo-3-Chloropropane	96-12-8	5.00	1.99	2.50	ug/Kg	59	139	30	59	139	30		
				1,2-Dibromoethane	106-93-4	1.00	0.386	1.00	ug/Kg	70	124	30	70	124	30		
				1,2-Dichlorobenzene	95-50-1	1.00	0.334	1.00	ug/Kg	70	123	30	70	123	30		
				1,2-Dichloroethane	107-06-2	1.00	0.392	0.500	ug/Kg	66	132	30	66	132	30		
				1,2-Dichloropropane	78-87-5	1.00	0.428	0.500	ug/Kg	70	127	30	70	127	30		
				1,3,5-Trimethylbenzene	108-67-8	1.00	0.380	1.00	ug/Kg	70	129	30	70	129	30		
				1,3-Dichlorobenzene	541-73-1	1.00	0.400	1.00	ug/Kg	70	122	30	70	122	30		
				1,3-Dichloropropane	142-28-9	1.00	0.362	0.500	ug/Kg	70	127	30	70	127	30		
				1,4-Dichlorobenzene	106-46-7	1.00	0.364	1.00	ug/Kg	70	120	30	70	120	30		
				1,4-Dioxane	123-91-1	100	41.1	50.0	ug/Kg	50	150	30	50	150	30		
				2,2-Dichloropropane	594-20-7	1.00	0.444	0.500	ug/Kg	68	120	30	68	120	30		
				2-Chloroethyl vinyl ether	110-75-8	2.00	0.766	1.00	ug/Kg	57	145	30	57	145	30		
				2-Chlorotoluene	95-49-8	1.00	0.314	0.500	ug/Kg	70	128	30	70	128	30		
				2-Hexanone	591-78-6	5.00	1.56	2.50	ug/Kg	53	140	30	53	140	30		
				3-Chloropropene	107-05-1	2.50	0.862	2.00	ug/Kg	50	150	30	50	150	30		
				4-Chlorotoluene	106-43-4	1.00	0.350	0.500	ug/Kg	70	127	30	70	127	30		
				Acetone	67-64-1	5.00	1.73	2.50	ug/Kg	47	131	30	47	131	30		
				Acrolein	107-02-8	100	22.6	50.0	ug/Kg	24	120	30	24	120	30		
				Acrylonitrile	107-13-1	20.0	4.44	10.0	ug/Kg	68	127	30	68	127	30		
				Benzene	71-43-2	0.250	0.146	0.250	ug/Kg	70	120	30	70	120	30		
				Bromobenzene	108-86-1	1.00	0.356	1.00	ug/Kg	70	129	30	70	129	30		
				Bromochloromethane	74-97-5	1.00	0.428	1.00	ug/Kg	70	121	30	70	121	30		
				Bromodichloromethane	75-27-4	1.00	0.372	1.00	ug/Kg	70	127	30	70	127	30		
				Bromoform	75-25-2	1.00	0.484	1.00	ug/Kg	70	135	30	70	135	30		
				Bromomethane	74-83-9	2.00	0.796	1.00	ug/Kg	30	170	30	30	170	30		
				Butadiene	106-99-0	1.00	0.474	1.00	ug/Kg	50	150	30	50	150	30		
				Carbon disulfide	75-15-0	2.00	0.802	1.00	ug/Kg	61	120	30	61	120	30		
				Carbon tetrachloride	56-23-5	1.00	0.384	0.500	ug/Kg	70	136	30	70	136	30		
				Chlorobenzene	108-90-7	1.00	0.386	0.500	ug/Kg	70	120	30	70	120	30		
				Chloroethane	75-00-3	1.00	0.504	0.504	ug/Kg	40	150	30	40	150	30		
				Chloroform	67-66-3	1.00	0.370	0.500	ug/Kg	70	120	30	70	120	30		
				Chloromethane	74-87-3	1.00	0.320	0.500	ug/Kg	45	140	30	45	140	30		
				cis-1,2-Dichloroethene	156-59-2	1.00	0.408	0.500	ug/Kg	70	120	30	70	120	30		
				cis-1,3-Dichloropropene	10061-01-5	1.00	0.416	0.500	ug/Kg	70	122	30	70	122	30		
				Cyclohexane	110-82-7	1.00	0.484	0.500	ug/Kg	60	133	30	60	133	30		
				Dibromochloromethane	124-48-1	1.00	0.488	1.00	ug/Kg	70	120	30	70	120	30		
				Dibromomethane	74-95-3	1.00	0.270	0.500	ug/Kg	70	120	30	70	120	30		
				Dichlorodifluoromethane	75-71-8	2.00	0.674	1.00	ug/Kg	30	150	30	30	150	30		
				Dichlorofluoromethane	75-43-4	1.00	0.376	1.00	ug/Kg	50	150	30	50	150	30		
				Ethyl ether	60-29-7	1.00	0.452	1.00	ug/Kg	69	125	30	69	125	30		
				Ethyl methacrylate	97-63-2	2.50	0.524	2.00	ug/Kg	50	150	30	50	150	30		
				Ethylbenzene	100-41-4	0.250	0.183	0.250	ug/Kg	70	125	30	70	125	30		
				Hexachlorobutadiene	87-68-3	1.00	0.446	0.500	ug/Kg	70	138	30	70	138	30		
				Hexane	110-54-3	1.00	0.492	0.500	ug/Kg	55	131	30	55	131	30		
				Iodomethane	74-88-4	2.50	0.662	2.00	ug/Kg	66	124	30	66	124	30		
				Isobutyl alcohol	78-83-1	100	35.7	100	ug/Kg	50	150	30	50	150	30		
				Isopropylbenzene	98-82-8	1.00	0.384	1.00	ug/Kg	70	132	30	70	132	30		
				m&p-Xylene	179601-23-1	0.500	0.182	0.500	ug/Kg	70	120	30	70	120	30		
				Methyl acetate	79-20-9	5.00	2.02	2.50	ug/Kg	56	131	30	56	131	30		
				Methyl Ethyl Ketone	78-93-3	5.00	2.12	2.50	ug/Kg	51	134	30	51	134	30		
				methyl isobutyl ketone	108-10-1	5.00	2.15	2.50	ug/Kg	53	135	30	53	135	30		

(continued)

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS Low	LCS High	LCS RPD %	MS Low	MS High	MS RPD %	Surrogate Low	Surrogate High
				Methyl tert-butyl ether	1634-04-4	1.00	0.394	1.00	ug/Kg	65	120	30	65	120	30		
				Methylcyclohexane	108-87-2	1.00	0.318	1.00	ug/Kg	60	140	30	60	140	30		
				Methylene Chloride	75-09-2	5.00	1.63	2.50	ug/Kg	70	120	30	70	120	30		
				Naphthalene	91-20-3	1.00	0.334	0.500	ug/Kg	59	143	30	59	143	30		
				n-Butylbenzene	104-51-8	1.00	0.388	0.500	ug/Kg	70	129	30	70	129	30		
				n-Heptane	142-82-5	1.00	0.418	0.500	ug/Kg	64	132	30	64	132	30		
				N-Propylbenzene	103-65-1	1.00	0.414	1.00	ug/Kg	70	132	30	70	132	30		
				o-Xylene	95-47-6	0.250	0.220	0.250	ug/Kg	70	120	30	70	120	30		
				p-Isopropyltoluene	99-87-6	1.00	0.362	1.00	ug/Kg	70	133	30	70	133	30		
				sec-Butylbenzene	135-98-8	1.00	0.398	0.500	ug/Kg	70	134	30	70	134	30		
				Styrene	100-42-5	1.00	0.386	0.500	ug/Kg	70	120	30	70	120	30		
				tert-Butyl alcohol	75-65-0	50.0	12.1	25.0	ug/Kg	50	150	30	50	150	30		
				tert-Butylbenzene	98-06-6	1.00	0.398	0.500	ug/Kg	70	137	30	70	137	30		
				Tetrachloroethene	127-18-4	1.00	0.370	0.500	ug/Kg	70	129	30	70	129	30		
				Tetrahydrofuran	109-99-9	5.00	1.88	2.00	ug/Kg	50	138	30	50	138	30		
				Toluene	108-88-3	0.250	0.147	0.250	ug/Kg	70	120	30	70	120	30		
				trans-1,2-Dichloroethene	156-60-5	1.00	0.350	0.500	ug/Kg	70	120	30	70	120	30		
				trans-1,3-Dichloropropene	10061-02-6	1.00	0.362	0.500	ug/Kg	70	123	30	70	123	30		
				trans-1,4-Dichloro-2-butene	110-57-6	5.00	1.19	5.00	ug/Kg	50	150	30	50	150	30		
				Trichloroethene	79-01-6	0.500	0.164	0.250	ug/Kg	70	122	30	70	122	30		
				Trichlorofluoromethane	75-69-4	1.00	0.428	1.00	ug/Kg	65	134	30	65	134	30		
				Vinyl acetate	108-05-4	2.00	0.904	1.00	ug/Kg	30	150	30	30	150	30		
				Vinyl chloride	75-01-4	0.500	0.262	0.500	ug/Kg	63	127	30	63	127	30		
				1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/Kg							75	125
				4-Bromofluorobenzene (Surr)	460-00-4				ug/Kg							75	120
				Dibromofluoromethane	1868-53-7				ug/Kg							75	120
				Toluene-d8 (Surr)	2037-26-5				ug/Kg							75	120

(400-140)

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS Low	LCS High	LCS/LCSD RPD %	MS Low	MS High	MS/MSD RPD %	Surrogate Low	Surrogate High
MDL S MSVOA	Volatile Organic Compo	8260B	5035A_LP	1,1,1,2-Tetrachloroethane	630-20-6	5.00	1.16	2.50	ug/Kg	70	120	30	70	120	30		
				1,1,1-Trichloroethane	71-55-6	5.00	1.16	2.50	ug/Kg	70	120	30	70	120	30		
				1,1,2,2-Tetrachloroethane	79-34-5	5.00	0.794	2.50	ug/Kg	70	133	30	70	133	30		
				1,1,2-Trichloro-1,2,2-trifluoroet	76-13-1	5.00	1.84	2.50	ug/Kg	70	120	30	70	120	30		
				1,1,2-Trichloroethane	79-00-5	5.00	0.968	2.50	ug/Kg	70	120	30	70	120	30		
				1,1-Dichloroethane	75-34-3	5.00	1.03	2.50	ug/Kg	70	120	30	70	120	30		
				1,1-Dichloroethene	75-35-4	5.00	1.82	2.50	ug/Kg	67	120	30	67	120	30		
				1,1-Dichloropropene	563-58-6	5.00	1.56	2.50	ug/Kg	70	120	30	70	120	30		
				1,2,3-Trichlorobenzene	87-61-6	5.00	2.16	2.50	ug/Kg	70	131	30	70	131	30		
				1,2,3-Trichloropropane	96-18-4	5.00	1.69	2.50	ug/Kg	70	129	30	70	129	30		
				1,2,4-Trichlorobenzene	120-82-1	5.00	2.01	2.50	ug/Kg	70	128	30	70	128	30		
				1,2,4-Trimethylbenzene	95-63-6	5.00	1.29	2.50	ug/Kg	70	123	30	70	123	30		
				1,2-Dibromo-3-Chloropropane	96-12-8	5.00	2.13	2.50	ug/Kg	59	150	30	59	150	30		
				1,2-Dibromoethane	106 93-4	5.00	1.27	2.50	ug/Kg	70	120	30	70	120	30		
				1,2-Dichlorobenzene	95-50-1	5.00	1.15	2.50	ug/Kg	70	125	30	70	125	30		
				1,2-Dichloroethane	107-06-2	5.00	0.741	2.50	ug/Kg	70	128	30	70	128	30		
				1,2-Dichloropropane	78-87-5	5.00	1.31	2.50	ug/Kg	70	120	30	70	120	30		
				1,3,5-Trimethylbenzene	108-67-8	5.00	1.43	2.50	ug/Kg	70	122	30	70	122	30		
				1,3-Dichlorobenzene	541-73-1	5.00	1.51	2.50	ug/Kg	70	123	30	70	123	30		
				1,3-Dichloropropane	142-28-9	5.00	0.961	2.50	ug/Kg	70	120	30	70	120	30		
				1,4-Dichlorobenzene	106-46-7	5.00	1.08	2.50	ug/Kg	70	123	30	70	123	30		
				1,4-Dioxane	123-91-1	100	48.9	50.0	ug/Kg	50	150	30	50	150	30		
				2,2-Dichloropropane	594-20-7	20.0	10.0	10.0	ug/Kg	41	120	30	41	120	30		
				2-Chloroethyl vinyl ether	110-75-8	5.00	0.760	2.50	ug/Kg	67	137	30	67	137	30		
				2-Chlorotoluene	95-49-8	5.00	1.15	2.50	ug/Kg	70	122	30	70	122	30		
				2-Hexanone	591-78-6	5.00	1.55	2.50	ug/Kg	50	150	30	50	150	30		
				3-Chloropropene	107-05-1	5.00	1.58	2.50	ug/Kg	50	150	30	50	150	30		
				4-Chlorotoluene	106-43-4	5.00	1 32	2.50	ug/Kg	70	121	30	70	121	30		
				Acetone	67-64-1	20.0	3.87	10.0	ug/Kg	50	133	30	50	133	30		
				Acrolein	107-02-8	400	214	220	ug/Kg	47	150	30	47	150	30		
				Acrylonitrile	107-13-1	80.0	9.57	40.0	ug/Kg	70	132	30	70	132	30		
				Benzene	71-43-2	5.00	1.11	2.50	ug/Kg	70	120	30	70	120	30		
				Bromobenzene	108-86-1	5.00	1.13	2.50	ug/Kg	70	121	30	70	121	30		
				Bromochloromethane	74-97-5	5.00	0.966	2.50	ug/Kg	70	120	30	70	120	30		
				Bromodichloromethane	75 27-4	5.00	0.844	2.50	ug/Kg	69	120	30	69	120	30		
				Bromoform	75 25-2	5.00	1.02	2.50	ug/Kg	51	127	30	51	127	30		
				Bromomethane	74-83-9	5.00	1.84	2.50	ug/Kg	69	120	30	69	120	30		
				Butadiene	106-99-0	5.00	1.39	2.50	ug/Kg	50	150	30	50	150	30		
				Carbon disulfide	75-15-0	5.00	1.84	2.50	ug/Kg	58	120	30	58	120	30		
				Carbon tetrachloride	56-23-5	5.00	1.07	2.50	ug/Kg	51	124	30	51	124	30		
				Chlorobenzene	108 90 7	5.00	1.18	2.50	ug/Kg	70	120	30	70	120	30		
				Chloroethane	75 00-3	5.00	2.10	2.50	ug/Kg	49	125	30	49	125	30		
				Chloroform	67-66-3	5.00	0.975	2.50	ug/Kg	70	120	30	70	120	30		
				Chloromethane	74-87-3	5.00	1.20	2.50	ug/Kg	63	126	30	63	126	30		
				cis-1,2-Dichloroethene	156-59-2	5.00	1.02	2.50	ug/Kg	70	120	30	70	120	30		
				cis-1,3-Dichloropropene	10061-01-5	5.00	1.14	2.50	ug/Kg	70	120	30	70	120	30		
				Cyclohexane	110-82-7	5.00	1.43	2.50	ug/Kg	66	123	30	66	123	30		
				Dibromochloromethane	124-48-1	5.00	0.575	2.50	ug/Kg	69	126	30	69	126	30		
				Dibromomethane	74-95-3	5.00	1.35	2.50	ug/Kg	70	120	30	70	120	30		
				Dichlorodifluoromethane	75-71-8	5.00	1.63	2.50	ug/Kg	42	150	30	42	150	30		
				Dichlorofluoromethane	75-43-4	5.00	1.15	2.50	ug/Kg	50	150	30	50	150	30		
				Ethyl ether	60-29-7	5.00	0.996	2.50	ug/Kg	65	120	30	65	120	30		
				Ethyl methacrylate	97-63-2	5.00	0.809	2.50	ug/Kg	50	150	30	50	150	30		
				Ethylbenzene	100-41-4	5.00	1.24	2.50	ug/Kg	70	120	30	70	120	30		
				Hexachlorobutadiene	87-68-3	5.00	1.55	2.50	ug/Kg	70	133	30	70	133	30		
				Hexane	110-54-3	5.00	1.73	2.50	ug/Kg	57	120	30	57	120	30		
				Iodomethane	74-88-4	5.00	1.44	2.50	ug/Kg	70	120	30	70	120	30		
				Isobutyl alcohol	78-83-1	400	125	200	ug/Kg	50	150	30	50	150	30		
				Isopropylbenzene	98-82-8	5.00	1.36	2.50	ug/Kg	70	125	30	70	125	30		
				m&p-Xylene	179601-23-1	10.0	1.85	5.00	ug/Kg	70	120	30	70	120	30		
				Methyl acetate	79-20-9	25 0	4.46	12 5	ug/Kg	50	150	30	50	150	30		
				Methyl Ethyl Ketone	78-93-3	5.00	1.78	2.50	ug/Kg	50	148	30	50	148	30		
				methyl isobutyl ketone	108-10-1	5.00	1.03	2.50	ug/Kg	50	150	30	50	150	30		

(641-005)

Analysis Group	Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS Low	LCS High	LCS/LCSD RPD %	MS Low	MS High	MS/MSD RPD %	Surrogate Low	Surrogate High
				Methyl tert-butyl ether	1634-04-4	5.00	1.18	2.50	ug/Kg	70	120	30	70	120	30		
				Methylcyclohexane	108-87-2	5.00	1.48	2.50	ug/Kg	50	150	30	50	150	30		
				Methylene Chloride	75-09-2	5.00	3.78	4.00	ug/Kg	70	120	30	70	120	30		
				Naphthalene	91-20-3	5.00	2.21	2.50	ug/Kg	70	126	30	70	126	30		
				n-Butylbenzene	104-51-8	5.00	1.65	2.50	ug/Kg	70	120	30	70	120	30		
				n-Heptane	142-82-5	5.00	2.06	2.50	ug/Kg	50	150	30	50	150	30		
				N-Propylbenzene	103-65-1	5.00	1.45	2.50	ug/Kg	70	123	30	70	123	30		
				o-Xylene	95-47-6	5.00	1.25	2.50	ug/Kg	70	120	30	70	120	30		
				p-Isopropyltoluene	99-87-6	5.00	1.52	2.50	ug/Kg	70	121	30	70	121	30		
				sec-Butylbenzene	135-98-8	5.00	1.61	2.50	ug/Kg	70	123	30	70	123	30		
				Styrene	100-42-5	5.00	1.17	2.50	ug/Kg	70	120	30	70	120	30		
				tert-Butyl alcohol	75-65-0	50.0	10.9	25.0	ug/Kg	50	150	30	50	150	30		
				tert-Butylbenzene	98-06-6	5.00	1.28	2.50	ug/Kg	70	121	30	70	121	30		
				Tetrachloroethene	127-18-4	5.00	1.04	2.50	ug/Kg	70	120	30	70	120	30		
				Tetrahydrofuran	109-99-9	5.00	1.79	2.50	ug/Kg	48	150	30	48	150	30		
				Toluene	108-88-3	5.00	1.74	2.50	ug/Kg	70	120	30	70	120	30		
				trans-1,2-Dichloroethene	156-60-5	5.00	1.25	2.50	ug/Kg	70	120	30	70	120	30		
				trans-1,3-Dichloropropene	10061-02-6	5.00	1.41	2.50	ug/Kg	68	121	30	68	121	30		
				trans-1,4-Dichloro-2-butene	110-57-6	5.00	1.66	2.50	ug/Kg	50	150	30	50	150	30		
				Trichloroethene	79-01-6	5.00	1.35	2.50	ug/Kg	70	120	30	70	120	30		
				Trichlorofluoromethane	75-69-4	5.00	1.16	2.50	ug/Kg	70	122	30	70	122	30		
				Vinyl acetate	108-05-4	5.00	1.34	2.50	ug/Kg	60	150	30	60	150	30		
				Vinyl chloride	75-01-4	5.00	1.19	2.50	ug/Kg	69	120	30	69	120	30		
				1,2-Dichloroethane-d4 (Surr)	17060-07-0				ug/Kg							70	134
				4-Bromofluorobenzene (Surr)	460-00-4				ug/Kg							70	122
				Dibromofluoromethane	1868-53-7				ug/Kg							75	120
				Toluene-d8 (Surr)	2037-26-5				ug/Kg							75	122

(900-180)

Table 2.
Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion
Dichlorodifluoromethane	85	87
Chloromethane	50	52
Vinyl Chloride	62	64
Butadiene	39	54
Bromomethane	94	96
Chloroethane	64	66
Dichlorofluoromethane	67	69
Tichlorofluoromethane	101	103
Ethanol	45	46
Ethyl Ether	59	74, 45
Acrolein	56	55, 58
1,1-Dichloroethene	96	61, 98
Trichlorotrifluoromethane	101	103, 151
Acetone	43	58
Iodomethane	142	127, 141
Carbon Disulfide	76	78
Isopropyl alcohol	45	59
Acetonitrile	41	40, 39
3-Chloro-1-propene	76	41, 39
Methyl Acetate	43	74
Methylene Chloride	84	49, 51
TBA-d9 (IS)	65	46
2-Methyl-2-propanol (TBA)	59	57, 41
Acrylonitrile	53	52, 51
trans-1,2-Dichloroethene	96	61, 98
Methyl-tert-butyl ether	73	57, 45
Hexane	57	56, 86
1,1-Dichloroethane	63	65, 83
Vinyl Acetate	43	86
Isopropyl Ether	45	43, 87
2-Chloro-1,3-butadiene	53	88
Tert-butyl-ethyl-ether	59	87, 57
2,2-Dichloropropane	77	97
cis-1,2-Dichloroethene	96	61, 98
2-Butanone	43	57, 72
Ethyl acetate	43	45, 61
Propionitrile	54	55, 40
Methacrylonitrile	41	39, 67
Chlorobromomethane	128	49, 130

Tetrahydrofuran	42	71, 72
Chloroform	83	85
Dibromofluoromethane(SS)	113	111, 192
1,1,1-Trichloroethane	97	99, 61
Cyclohexane	56	69, 84
1,1-Dichloropropene	75	110, 77
Carbon Tetrachloride	117	119, 121
Isobutyl alcohol	43	41, 74
1,2-Dichloroethane-d4 (SS)	65	102
Benzene	78	77
1,2-Dichloroethane	62	64, 100
Tert-amyl methyl ether	73	55, 87
n-Heptane	43	57, 71
Fluorobenzene (IS)	96	77
n-Butanol	56	43, 41
Trichloroethene	130	95, 97
Ethyl acrylate	55	56
Methylcyclohexane	83	98
1,2-Dichloropropane	63	65
1,4-Dioxane-d8 (IS)	96	64
Methyl methacrylate	41	69, 39
Dibromomethane	93	95, 174
1,4-Dioxane	88	58
Dichlorobromomethane	83	85
2-Nitropropane	43	41, 39
2-Chloroethyl vinyl ether	63	65, 106
cis-1,3-Dichloropropene	75	77, 49
4-Methyl-2-pentanone	43	58, 100
Toluene-d8 (SS)	98	100
Toluene	92	91
trans-1,3-Dichloropropene	75	77, 49
Ethyl methacrylate	69	41, 99
1,1,2-Trichloroethane	97	83, 85
Tetrachloroethene	166	164, 129
1,3-Dichloropropane	76	78
2-Hexanone	43	58, 57
n-Butyl acetate	43	56, 73
Chlorodibromomethane	129	127
Ethylene Dibromide	107	109, 188
Chlorobenzene-d5 (IS)	117	119
Chlorobenzene	112	114
1,1,1,2-Tetrachloroethane	131	133, 119

Ethylbenzene	106	91
m & p-Xylene	91	106, 77
o-Xylene	91	106
Styrene	104	78
Bromoform	173	171, 175
Isopropylbenzene	105	120
Cyclohexanone	55	69, 42
4-Bromofluorobenzene (SS)	95	174, 176
Bromobenzene	156	77, 158
1,1,2,2-Tetrachloroethane	83	85, 131
trans-1,4-Dichloro-2-butadiene	53	88, 124
n-Propylbenzene	91	120
2-Chlorotoluene	91	126
1,3,5-Trimethylbenzene	105	120
4-Chlorotoluene	91	126
tert-Butylbenzene	119	91, 134
Pentachloroethane	167	117, 165
1,2,4-Trimethylbenzene	105	134
sec-Butylbenzene	105	134
1,3-Dichlorobenzene	146	111, 148
4-Isopropyltoluene	119	134, 91
1,4-Dichlorobenzene-d4 (IS)	152	150, 115
1,4-Dichlorobenzene	146	111, 148
1,2,3-Trimethylbenzene	105	120, 77
Benyl Chloride	126	91, 65
1,2-Dichlorobenzene	146	111, 148
n-Butylbenzene	91	92, 134
1,2-Dichlorobenzene	146	111, 148
n-Butylbenzene	91	92, 134
1,2-Dibromo-3-chloropropane	75	155, 157
1,3,5-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
Hexachlorobutadiene	225	223, 227
Naphthalene	128	129
1,2,3-Trichlorobenzene	180	182, 145
2-Methylnaphthalene	142	141, 143

*NOTE: The primary and secondary ions listed here are taken directly from SW-846 Method 8260. The laboratory uses secondary ions in the cases of Ethanonl, Ethylbenzene, Toluene, 1,1,2-Trichloroethane, Trichloroethene, 1,2,3-Trichloropropane and Xylenes due to interferences and/or to maintain consistency between methods.

Figure 1.

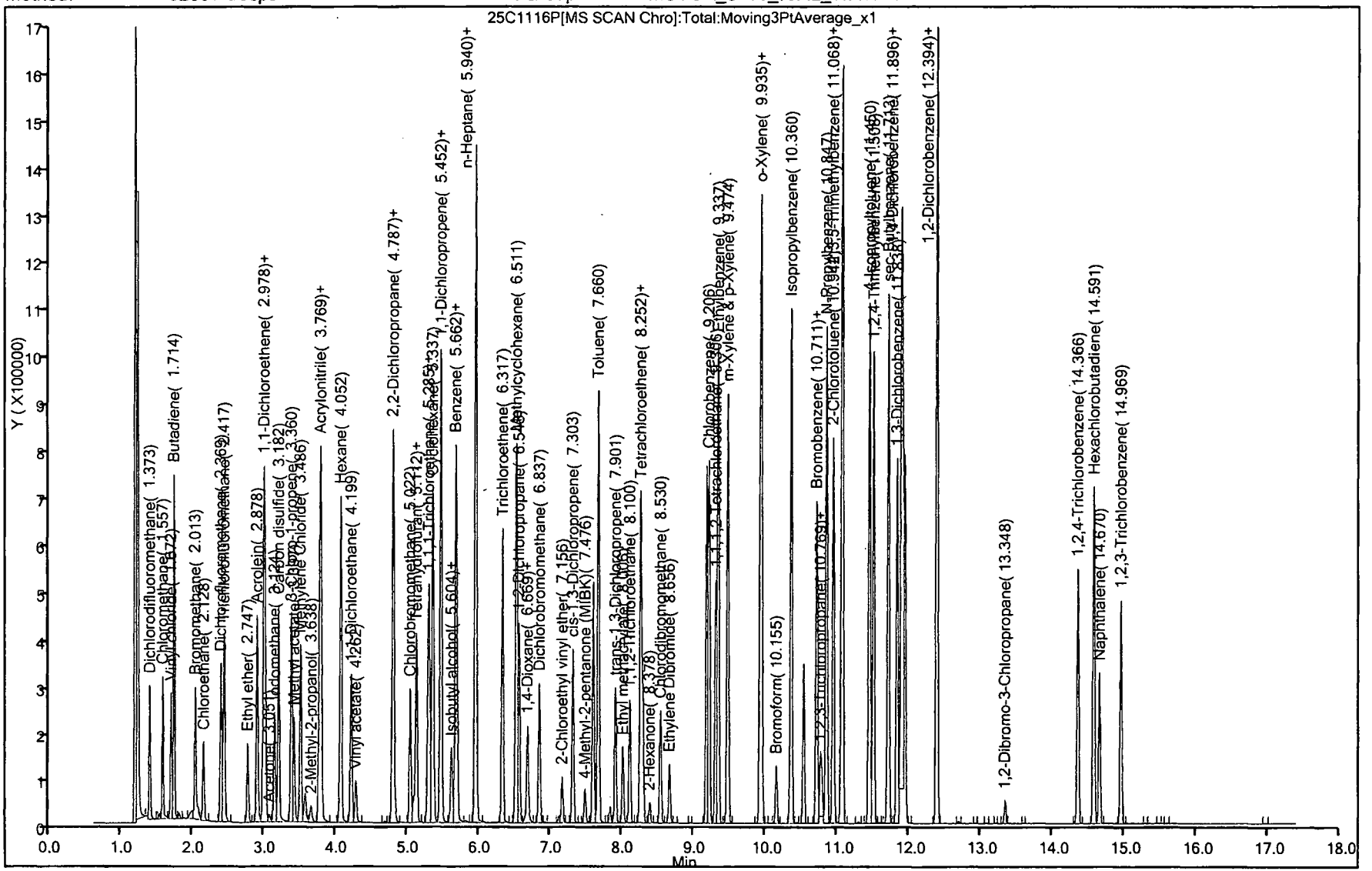
Example: Total Ion Chromatogram for 5 mL Purge Water (045-001)

Report Date: 01-Dec-2015 13:12:52 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

TestAmerica Chicago

Data File:	\\ChromNA\Chicago\ChromData\CMS25\20151116-34579.b\25C1116P.D				
Injection Date:	16-Nov-2015 21:37:30	Instrument ID:	CMS25	Operator ID:	TT
Lims ID:	CCVIS			Worklist Smp#:	2
Client ID:					
Purge Vol:	5.000 mL	Dil. Factor:	1.0000	ALS Bottle#:	29
Method:	8260W25cps	Limit Group:	MSVOA_8260_ICAL_WATER		



(100-540)

Figure 2.

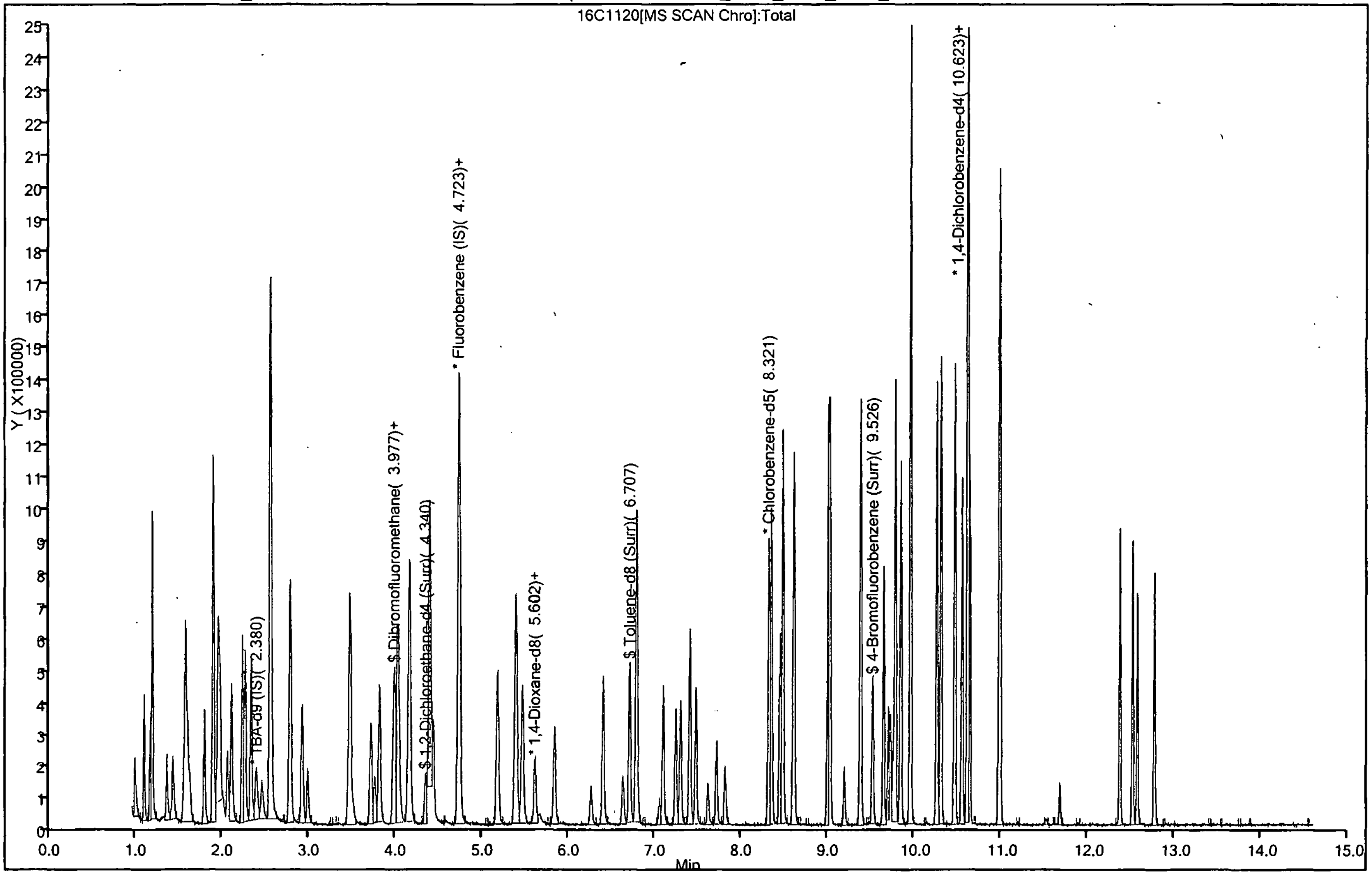
Example: Total Ion Chromatogram for 5 mL Purge Soil (046-001)

Report Date: 01-Dec-2015 13:24:10 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

TestAmerica Chicago

Data File:	\\ChromNA\Chicago\ChromData\CMS16\20151120-34653.b\16C1120.D				
Injection Date:	20-Nov-2015 08:32:30	Instrument ID:	CMS16	Operator ID:	BDW
Lims ID:	CCVIS			Worklist Smp#:	3
Client ID:					
Purge Vol:	5.000 mL	Dil. Factor:	1.0000	ALS Bottle#:	2
Method:	8260s16_test	Limit Group:	MSVOA_8260_ICAL_SOIL_LOW		



(046-001)

Attachment 1.

Example: Initial Calibration Guides, Method Listings, Concentrator Conditions, Flow Settings (047-001 to 047-004)

TestAmerica Chicago CHI-22-20-061/F-03/14

8260LOW LEVEL SOIL ICAL LIST#1

ICAL LEVELS	CONC SS	CONC	FILE	AMOUNT INJECTED	MEOH
1		5	A	1UL IS + 5.0 UL LOW 8260/624 STD, LOW ACROLEIN	94 UL
2	12	20	B	1UL IS + 2UL LOW SS + 20.0 UL LOW 8260/624 STD, LOW ACROLEIN	77 UL
3	30	50	C	1UL IS + 5UL LOW SS + 2.5 UL 8260 GAS, MEGA, KET, VA/CEVE, ACROLEIN	84 UL
4	60	100	D	1UL IS + 10UL LOW SS + 5.0 UL 8260 GAS, MEGA, KET, VA/CEVE, ACROLEIN	69 UL
5	90	150	E	1UL IS + 15UL LOW SS + 7.5 UL 8260 GAS, MEGA, KET, VA/CEVE, ACROLEIN	54 UL
6	120	200	F	1UL IS + 20UL LOW SS + 10.0 UL 8260 GAS, MEGA, KET, VA/CEVE, ACROLEIN	39 UL

8260/ 624 IS	250/ 5000
8260/ 624 LOW SS	30
LOW 8260/ 624 STD	5/10/25/50/100/125
LOW ACROLEIN STD	200
8260/ 624 GAS STD	100
8260/ 624 MEGAMIX STD	100/200/500/1000/20000/2500
8260/ 624 KETONE STD	100
8260/ 624 VA/ CEVESTD	100
8260/ 624 ACROLEIN STD	4000

(047-001)

TestAmerica Chicago CHI-22-20-083/F-03/14

8260LOW LEVEL SOIL ICAL LIST#2

ICAL LEVELS	CONC SS	CONC	FILE	AMOUNT INJECTED	MEOH
1		5	A	1UL IS + 5.0 UL LOW 8260 ADDS, STD2, POLAR, CYCLOHEXANONE	84 UL
2	12	20	B	1UL IS + 20 UL LOW 8260 ADDS, STD2, POLAR, CYCLOHEXANONE	39 UL
3	30	50	C	1UL IS + 2.5 UL 8260 ADDS, STD2, POLAR, CYCLOHEXANONE	91.5 UL
4	60	100	D	1UL IS + 5 UL 8260 ADDS, STD2, POLAR, CYCLOHEXANONE	84 UL
5	90	150	E	1UL IS + 7.5 UL 8260 ADDS, STD2, POLAR, CYCLOHEXANONE	76.5 UL
6	120	200	F	1UL IS + 10 UL 8260 ADDS, STD2, POLAR, CYCLOHEXANONE	69 UL

8260/ 624 IS	250/ 5000
LOW 8260/ 624 ADDS	5/ 10/50/125
LOW 8260/624 STD2	5
LOW POLAR ADD	5/ 50/ 250
LOW CYCLOHEXANONE	4000
8260/ 624 ADDS	100/ 200/1000/ 2500
8260/ 624 STD2	100
8260/ 624 POLAR ADDS	100/ 1000/ 5000
8260 CYCLOHEXANONE	4000

(047-002)

TestAmerica Chicago CHI-22-20-059/F-03/14

8260W/ 8260HL/624 - 5ML WATER - LIST#1

ICAL LEVELS	CONC SS	CONC	FILE	AMOUNT INJECTED	MEOH
1		0.25	A	5UL IS + 2.5 UL LOW LOW 8260/624 STD	92.5 UL
2		1	B	5UL IS + 1.0 UL LOW 8260/624 STD, LOW ACROLEIN	94 UL
3		2	C	5UL IS + 2.0 UL LOW 8260/624 STD, LOW ACROLEIN	93 UL
4		5	D	5UL IS + 5.0 UL LOW 8260/624 STD, LOW ACROLEIN	90 UL
5	12	20	E	5UL IS + 2UL SS + 20.0 UL LOW 8260/624 STD, LOW ACROLEIN	73 UL
6	30	50	F	5UL IS + 5UL SS + 2.5 UL 8260 GAS, MEGA, KET, VA/CEVE, ACROLEIN	80 UL
7	60	100	G	5UL IS + 10UL SS + 5.0 UL 8260 GAS, MEGA, KET, VA/CEVE, ACROLEIN	65 UL
8	90	150	H	5UL IS + 15UL SS + 7.5 UL 8260 GAS, MEGA, KET, VA/CEVE, ACROLEIN	50 UL
9	120	200	I	5UL IS + 20UL SS + 10.0 UL 8260 GAS, MEGA, KET, VA/CEVE, ACROLEIN	35 UL

8260/ 624 LOW IS	50/1000
8260/ 624 LOW SS	30
8260/ 624 LOW IS/SS	50/1000/30
LOW LOW 8260/ 624 STD	0.5/1/2.5/5/10/12.5
LOW 8260/ 624 STD	5/10/25/50/100/125
LOW ACROLEIN STD	200
8260/ 624 GAS STD	100
8260/ 624 MEGAMIX STD	100/200/500/1000/20000/2500
8260/ 624 KETONE STD	100
8260/ 624 VA/ CEVESTD	100
8260/ 624 ACROLEIN STD	4000

(047-003)

TestAmerica Chicago CHI-22-20-060/F-03/14

8260 WATER ICAL LIST#2

ICAL LEVELS	CONC	FILE	AMOUNT INJECTED	MEOH
2	1	B	5UL LOW IS + 1UL LOW ADDS, LOW STD2, LOW POLAR, LOW CYCLOHEXANONE	92UL
3	2	C	5UL LOW IS + 2UL LOW ADDS, LOW STD2, LOW POLAR, LOW CYCLOHEXANONE	89UL
4	5	D	5UL LOW IS + 5UL LOW ADDS, LOW STD2, LOW POLAR, LOW CYCLOHEXANONE	80UL
5	20	E	5UL LOW IS + 20UL LOW ADDS, LOW STD2, LOW POLAR, LOW CYCLOHEXANONE	35UL
6	50	F	5UL LOW IS + 2.5UL 8260 ADDS, STD2, POLAR, CYCLOHEXANONE	87.5UL
7	100	G	5UL LOW IS + 5UL 8260 ADDS, STD2, POLAR, CYCLOHEXANONE	80UL
8	150	H	5UL LOW IS + 7.5UL 8260 ADDS, STD2, POLAR, CYCLOHEXANONE	72.5UL
9	200	I	5UL LOW IS + 10UL 8260 ADDS, STD2, POLAR, CYCLOHEXANONE	65UL

8260/ 624 IS	250/ 5000
LOW 8260/ 624 ADDS	5/ 10/50/125
LOW 8260/624 STD2	5
LOW POLAR ADD	5/ 50/ 250
LOW CYCLOHEXANONE	4000
8260/ 624 ADDS	100/ 200/1000/ 2500
8260/ 624 STD2	100
8260/ 624 POLAR ADDS	100/ 1000/ 5000
8260 CYCLOHEXANONE	4000

(047-004)

Example: Volatiles Method for 5972

GC Oven Parameters

Initial Temperature = 40 °C
Initial Time = 2.0 minutes
Detector A Temperature = 180 °C
Detector B Temperature = 250 °C
Oven Equib. Time = 0.50 min.

Ramp Rate (°C/min.)	Final Temp. (°C)	Final Time (min.)
7.0	65	0.00
12.0	165	0.00
20.0	212	5.00

Run Time = 21.25 min.

Inlet Pressure Program

Gas = Helium
Column length = 75 m
Column Diameter = 0.530 mm
Initial Pressure = 3 psi
Rate (psi/min) = 0.00
Initial Time = 7.0 min.
Oven Temp. 50 °C
Program Time = 7.0 min.

Scan Parameters

Mass Range = 35-260
Threshold = 150
Scans/sec = 1.9
EM Voltage = 1938
Solvent Delay (scan start time): before the elution of the first compound.
Run Time (scan stop time): until after the elution of last compound.

Example: Volatiles Method for 5973

GC Oven Parameters

Initial Temperature = 50 °C
Initial Time = 2.0 minutes
Aux Temperature = 250 °C
Oven Equib. Time = 0.50 min.

Ramp Rate (°C/min.)	Final Temp. (°C)	Final Time (min.)
15.0	220	0.00

Run Time = 13.33 min.

Inlet Pressure Program

Mode=split
Gas = Helium
Column length = 25 m
Column Diameter = 0.25 mm
Constant flow = 1.0 mL/min
Injection port temp. = 250 °C
Program Time = 7.0 min.
Split ratio = 80:1
Gas saver = off

Scan Parameters

Mass Range = 35-260
Threshold = 100
Scans/sec = 6
EM Voltage = 1938
Solvent Delay = 0.8 min. (scan start time): before the elution of the first compound.
Run Time (scan stop time): until after the elution of last compound.

Concentrator Conditions

Trap Temp. Prior to Purge	< 35
Desorb Preheat	250
Desorb	250
Bake	260
Purge Time	11 min
Desorb	0.5 - 2 min (inst. dependent)
Bake Time	4 min

Trap = Vocarb 3000

Flow Conditions

Purge Pressure	20 psi
Purge Flow Rate	~40 mLs/min

Flow Adjustment

Capillary Column: 5972/MSD's;

· Make-up gas off/separator pump on: flow through separator is 5-10 mLs/minutes.

· Open make-up gas: adjust until you achieve ~30 mLs/minute through the separator. (On MSD's - adjust to 0.5 torr on gauge)

(Flow into the Mass Spec is $\leq$ 1 mL/minute)

Approximate Vacuums

~5 x 10^{-6} torr

Example Archon Conditions

Transfer Line Temp	110 deg C
Soil Valve	95 deg C
Purge Pressure	25 psi
Purge Flow	~ 40 ml/min
Purge Time	11 min
Desorb	0.5 -2 min
SamplePre-heat(soils)	40 deg C ~ 2 min

Attachment 2.

Example: Target and Internal Standards; Initial Calibration (Form 6) (51-001 to 051-006)

TestAmerica Laboratories
Initial Calibration RRF Report

Method: \\ChromNA\Chicago\ChromData\CMS25\20151116-34579.b\8260W25cps.m
Instrument: CMS25 Lims Location: 500
Lock State: Initial Calib Locked Cpnd Order: Retention Time
Integrator: RTE Last Modified: 17-Nov-2015 10:08:39
No.Compounds:125

Initial Calibration Batches

Ical Batch: \\ChromNA\Chicago\ChromData\CMS25\20151113-34534.b
Inj Date : 13-Nov-2015 12:42:30, Sublist: chrom-8260W25cps*sub3

Limit Group: MSVOA_8260_ICAL_WATER

Detector 1: MS SCAN

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	b	M1	M2	%RSD/R^2
1 Dichlorodifluoromethan		0.3966149	0.2976011	0.2861239	0.2834346	0.3674132	0.3505291	0.3409182	0.3387940		0.3326786		Avg 12.2
2 Chloromethane		0.6724588	0.4676534	0.3693743	0.3337206	0.3986149	0.3980569	0.3891150	0.3779356	0.1908163	0.3831313		Linr 0.998
3 Vinyl chloride	0.3628101	0.3873248	0.3215913	0.3027708	0.3189223	0.3771277	0.3617825	0.3526995	0.3465227		0.3479502		Avg 8.1
4 Butadiene		0.4284553	0.4010441	0.3737846	0.3767313	0.4540248	0.4359413	0.4253770	0.4199596		0.4144147		Avg 6.8
5 Bromomethane		0.1507851	0.2164898	0.1664526	0.1520234	0.1787863	0.1900418	0.1927839	0.1838658		0.1789036		Avg 12.4
6 Chloroethane		0.2270908	0.2003292	0.1864031	0.1931828	0.2151840	0.2111469	0.2042085	0.1941865		0.2039665		Avg 6.5
7 Dichlorofluoromethane		0.5764613	0.5332213	0.5038482	0.5021176	0.5376329	0.5108964	0.5040755	0.4938368		0.5202613		Avg 5.3
8 Trichlorofluoromethane		0.6061578	0.4976604	0.4977540	0.4986424	0.5574423	0.5393644	0.5211443	0.5084136		0.5283224		Avg 7.2
9 Ethanol		0.6003503	0.4424719	0.2945803	0.2179101	0.2061537	0.2089814	0.1953868	0.2132973	16.467769	0.2051891		Linr 0.993
10 Ethyl ether		0.1725414	0.1344913	0.1385955	0.1426572	0.1455978	0.1477155	0.1413198	0.1464009		0.1461649		Avg 7.9
11 Acrolein		0.0177186	0.0157199	0.0154400	0.0169678	0.0166907	0.0177980	0.0171517	0.0180012		0.0169360		Avg 5.6
12 1,1-Dichloroethene		0.3285671	0.2830605	0.2724921	0.2702967	0.2851507	0.2845141	0.2737864	0.2781922		0.2845075		Avg 6.6
13 1,1,2-Trichloro-1,2,2-		0.3748587	0.3071278	0.3036528	0.3060727	0.3268891	0.3256078	0.3142998	0.3191208		0.3222037		Avg 7.1
14 Acetone				0.0483529	0.0363606	0.0343080	0.0331656	0.0323962	0.0332831	0.0795428	0.0324444		Linr 1.000
15 Iodomethane		0.3708091	0.3880848	0.3922757	0.4227748	0.4664004	0.4505584	0.4567113	0.4273966		0.4218764		Avg 8.3
16 Carbon disulfide		1.2308958	0.9739148	0.9019615	0.8942929	0.9652733	0.9515329	0.9169819	0.9245502		0.9699254		Avg 11.3
17 Isopropyl alcohol		2.2003114	1.3369311	1.2105927	1.0821573	1.1389559	1.0720711	0.9975896	1.0923433	9.4159245	1.0554916		Linr 0.998
18 Acetonitrile		0.0164047	0.0145565	0.0147598	0.0164046	0.0143515	0.0150063	0.0148970	0.0161287		0.0153136		Avg 5.6
19 3-Chloro-1-propene		0.1690477	0.1586358	0.1443851	0.1519990	0.1515980	0.1495272	0.1446734	0.1435562		0.1516778		Avg 5.7
20 Methyl acetate		0.1743359	0.1164486	0.0958526	0.0919221	0.0979772	0.0992649	0.0938981	0.1017934	0.2505600	0.0977136		Linr 0.998
21 Methylene Chloride				0.2984728	0.2581373	0.2546949	0.2465285	0.2366663	0.2357732		0.2550455		Avg 9.1
22 TBA-d9 (IS)	78751	81310	84598	77804	75533	73008	82861	79543	81230				
23 2-Methyl-2-propanol		1.9997540	1.6980307	1.4544239	1.2275429	1.2615330	1.1639131	1.1690784	1.1722455	8.4681621	1.2036636		Linr 0.997
24 Acrylonitrile		0.0464583	0.0450991	0.0410719	0.0422279	0.0431557	0.0445934	0.0420428	0.0448250		0.0436843		Avg 4.2
25 trans-1,2-Dichloroethe		0.3318226	0.2961741	0.2830608	0.2911281	0.3054701	0.2990757	0.2951620	0.2975342		0.2999284		Avg 4.8
26 Methyl tert-butyl ethe		0.3758115	0.3748170	0.3460879	0.3789684	0.3935251	0.4056436	0.3912209	0.4110730		0.3846434		Avg 5.3
27 Hexane		0.6663448	0.5233476	0.4743233	0.4814771	0.5207421	0.5273653	0.5098440	0.5173309		0.5275969		Avg 11.3
28 1,1-Dichloroethane		0.5294551	0.5103883	0.4859344	0.4978507	0.5123749	0.5024758	0.4945089	0.4954337		0.5035527		Avg 2.7
29 Vinyl acetate		0.2629013	0.2583375	0.2268175	0.2594852	0.2590704	0.2586224	0.2861246	0.2896045		0.2626204		Avg 7.4

(05-001)

Method: \\ChromNA\Chicago\ChromData\CMS25\20151116-34579.b\8260W25cps.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	b	M1	M2	%RSD/R^2
31 Isopropyl ether		0.9263964	0.8937486	0.9216222	0.9523844	0.9135780	0.9819747	0.9573037	1.0044499		0.9439323		Avg 3.9
30 2-Chloro-1,3-butadiene		0.4947591	0.4681182	0.4797449	0.4688948	0.5047970	0.5152932	0.5175266	0.5375172		0.4983314		Avg 5.0
32 Tert-butyl ethyl ether		74.740529	77.096134	79.982101	83.928915	80.291435	89.725584	81.714791	79.732510		80.901500		Avg 5.6
33 2,2-Dichloropropane		0.4822901	0.4179375	0.4017378	0.4150610	0.4306904	0.4305764	0.4255926	0.4321849		0.4295088		Avg 5.5
34 cis-1,2-Dichloroethene		0.3261850	0.3060864	0.2908550	0.2983994	0.3072608	0.3003687	0.2966504	0.2978624		0.3029585		Avg 3.5
35 2-Butanone (MEK)				0.0480802	0.0484753	0.0521207	0.0523552	0.0492890	0.0524958		0.0504694		Avg 4.1
36 Propionitrile		0.0159070	0.0134589	0.0147439	0.0158130	0.0147808	0.0156162	0.0155244	0.0162105		0.0152569		Avg 5.8
37 Ethyl acetate		0.1090880	0.1011520	0.1070628	0.1059948	0.1121614	0.1157865	0.1192049	0.1223278		0.1115973		Avg 6.4
38 Methacrylonitrile		0.0854806	0.0780802	0.0813706	0.0849988	0.0875432	0.0900140	0.0912963	0.0934438		0.0865284		Avg 5.9
39 Chlorobromomethane		0.1280760	0.1334500	0.1281552	0.1306073	0.1319846	0.1304380	0.1269717	0.1281509		0.1297292		Avg 1.7
40 Tetrahydrofuran			0.0497352	0.0350177	0.0343408	0.0339168	0.0352219	0.0331309	0.0354978	0.0586366	0.0333787		Linr 0.995
41 Chloroform		0.5562137	0.4873624	0.4540199	0.4671009	0.4716813	0.4548582	0.4477431	0.4491397		0.4735149		Avg 7.6
$ 42 Dibromofluoromethane					0.2731711	0.2892200	0.2880848	0.2743402	0.2747413		0.2799115		Avg 2.9
43 1,1,1-Trichloroethane		0.5165125	0.4660721	0.4493209	0.4590217	0.4827829	0.4753360	0.4631911	0.4670859		0.4724154		Avg 4.3
44 Cyclohexane		0.7045374	0.5826672	0.5643574	0.5639298	0.5860625	0.5854652	0.5677592	0.5747397		0.5911898		Avg 7.9
46 Carbon tetrachloride		0.5293757	0.4566226	0.4453757	0.4636703	0.4814041	0.4782251	0.4686804	0.4761408		0.4749368		Avg 5.3
45 1,1-Dichloropropene		0.4310756	0.3858864	0.3737365	0.3918383	0.4036948	0.3997363	0.3909447	0.3960513		0.3966205		Avg 4.2
47 Isobutyl alcohol			0.0025841	0.0034711	0.0036625	0.0037083	0.0041554	0.0039180	0.0044473	-0.0738144	0.0040406		Linr 0.996
$ 48 1,2-Dichloroethane-d4					0.2107703	0.2206083	0.2213692	0.2084793	0.2118892		0.2146233		Avg 2.8
49 Benzene	1.3635973	1.1510964	1.0405240	0.9880184	1.0288790	1.0690065	1.0553546	1.0422887	1.0406353		1.0866000		Avg 10.4
50 1,2-Dichloroethane		0.2797346	0.2739581	0.2483718	0.2696877	0.2775608	0.2814960	0.2715435	0.2810658		0.2729273		Avg 4.0
51 Isooctane		1.3534066	1.2747668	1.2890494	1.1869185	1.2886728	1.2934703	1.3180502	1.3416535		1.2932485		Avg 3.9
52 Tert-amyl methyl ether		58.043591	55.713392	58.433335	60.448818	57.135444	63.601012	58.745221	56.258969		58.547473	\	Avg 4.3
54 n-Heptane		0.7722676	0.6138313	0.6262299	0.6435853	0.6710112	0.6735917	0.6488172	0.6586371		0.6634964		Avg 7.3
* 53 Fluorobenzene (IS)	604724	629704	648183	623541	615777	601987	612126	649449	595671				
55 n-Butanol			0.0019066	0.0016577	0.0016370	0.0020011	0.0020074	0.0021061			0.0018860		Avg 10.4
56 Trichloroethene	0.4610368	0.3824813	0.3628219	0.3525670	0.3596594	0.3790613	0.3748950	0.3634019	0.3680517		0.3782196		Avg 8.6
57 Ethyl acrylate		0.1732137	0.1398999	0.1440062	0.1434807	0.1454729	0.1558086	0.1557407	0.1603364		0.1522449		Avg 7.3
58 Methylcyclohexane		0.6262466	0.5040629	0.4918682	0.5102862	0.5333305	0.5363577	0.5216858	0.5315866		0.5319281		Avg 7.7
59 1,2-Dichloropropane		0.2489265	0.2439897	0.2348843	0.2471268	0.2556882	0.2520543	0.2469483	0.2509938		0.2475765		Avg 2.5
60 2,3-Dichloro-1-propene		0.3658092	0.3324228	0.3595402	0.3828447	0.3719181	0.3917624	0.3860558	0.4253537		0.3769634		Avg 7.2
* 61 1,4-Dioxane-d8	11318	10834	11604	10821	10539	10934	12424	13035	13724				
62 Dibromomethane		0.1341900	0.1219177	0.1132885	0.1149523	0.1165989	0.1177315	0.1127397	0.1160616		0.1184350		Avg 5.9
63 Methyl methacrylate		0.1228822	0.1321769	0.1124322	0.1157232	0.1229430	0.1263532	0.1278830	0.1311165		0.1239388		Avg 5.6
64 1,4-Dioxane				0.8880880	1.0985388	1.1209073	0.9826143	0.9470144	0.9033809		0.9900906		Avg 10.0
65 Dichlorobromomethane		0.3401598	0.3249468	0.3007982	0.3073434	0.3159420	0.3141690	0.3080124	0.3132979		0.3155837		Avg 3.9
66 2-Nitropropane		0.0440920	0.0585756	0.0479776	0.0489421	0.0527569	0.0550953	0.0579276	0.0596473		0.0531268		Avg 10.7
67 2-Chloroethyl vinyl et		0.0930596	0.0815742	0.0777976	0.0824690	0.0844802	0.0880031	0.0838952	0.0867303		0.0847512		Avg 5.4
68 cis-1,3-Dichloropropen		0.3615191	0.3356305	0.3167875	0.3383205	0.3539047	0.3529478	0.3477219	0.3512421		0.3447593		Avg 4.1
69 4-Methyl-2-pentanone (				0.0967859	0.0939301	0.1103396	0.1127701	0.1069820	0.1105342		0.1052236		Avg 7.5
$ 70 Toluene-d8 (Surr)					0.9943129	1.0482868	1.0342928	1.0023001	0.9998360		1.0158057		Avg 2.4
71 Toluene	0.8294693	0.7809225	0.6900443	0.6628915	0.6753419	0.6907309	0.6728386	0.6697123	0.6713383		0.7048099		Avg 8.4
72 trans-1,3-Dichloroprop		0.3398519	0.2999706	0.3047159	0.3415621	0.3551613	0.3678950	0.3613745	0.3736936		0.3430281		Avg 8.1
73 Ethyl methacrylate		0.2781370	0.2066476	0.1775406	0.1993083	0.1972136	0.2063518	0.1954265	0.2080529	0.0690402	0.1931227		Linr 0.991
74 1,1,2-Trichloroethane		0.2202005	0.1877601	0.1827998	0.1978766	0.2018718	0.2063074	0.1984795	0.2073158		0.2003264		Avg 5.8
75 Tetrachloroethene		0.6559834	0.5187710	0.5052232	0.5204151	0.5383151	0.5316446	0.5266313	0.5294837		0.5408084		Avg 8.8

(05-003)

Method: \\ChromNA\Chicago\ChromData\CMS25\20151116-34579.b\8260W25cps.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9	b	M1	M2	%RSD/R^2
76 1,3-Dichloropropane		0.3293561	0.3040216	0.3124775	0.3324082	0.3409000	0.3446196	0.3338384	0.3471844		0.3306007		Avg 4.6
77 2-Hexanone				0.0811148	0.0818860	0.0920651	0.0962997	0.0935299	0.0967549		0.0902751		Avg 7.8
78 n-Butyl acetate		0.2434059	0.2057332	0.2195050	0.2292512	0.2392511	0.2524694	0.2539463	0.2570653		0.2375784		Avg 7.7
79 Chlorodibromomethane		0.3145572	0.2881723	0.2754934	0.3036884	0.3145854	0.3244955	0.3158407	0.3301048		0.3083672		Avg 6.0
80 Ethylene Dibromide		0.1952207	0.2028498	0.1866806	0.2026813	0.2058291	0.2121137	0.2056056	0.2155649		0.2033182		Avg 4.5
* 81 Chlorobenzene-d5	468401	476384	493715	471554	461007	446521	450282	474504	434774				
82 1-Chlorohexane		0.9359429	0.7032522	0.5263746	0.4327230	0.4486035	0.4491927	0.4485704	0.4625050	0.5003594	0.4388359		Linr 0.998
83 Chlorobenzene		1.1540060	0.9762211	1.0162569	1.0552877	1.0815102	1.0645251	1.0541007	1.0586696		1.0575722		Avg 4.8
84 1,1,1,2-Tetrachloroeth		0.3638871	0.3387582	0.3355289	0.3685736	0.3833213	0.3845346	0.3861478	0.3891729		0.3687405		Avg 5.8
85 Ethylbenzene	0.7643024	0.6407646	0.5774080	0.5678883	0.5811246	0.6099624	0.6011733	0.5957119	0.5946957		0.6147812		Avg 9.7
86 m-Xylene & p-Xylene	1.6579811	1.5685875	1.3135108	1.2996391	1.3346164	1.3794805	1.3558870	1.3454913	1.3542743		1.4010520		Avg 8.9
87 o-Xylene	1.6071699	1.4431635	1.3039405	1.2686564	1.3421163	1.3912560	1.3612381	1.3559015	1.3607892		1.3815813		Avg 7.1
88 Styrene		1.1555804	1.0338961	1.0087074	1.0515079	1.1170404	1.0987559	1.0887678	1.0948924		1.0811435		Avg 4.4
89 Bromoform		0.1572261	0.1428456	0.1412352	0.1580291	0.1635713	0.1716491	0.1661770	0.1759178		0.1595814		Avg 7.8
90 Isopropylbenzene		3.9332609	3.4946058	3.4134676	3.6068152	3.7483105	3.6768702	3.6913366	3.6788911		3.6554448		Avg 4.3
91 Cyclohexanone		0.0098338	0.0085113	0.0072148	0.0078295	0.0081282	0.0082771	0.0088088	0.0106149		0.0086523		Avg *12.7 R7
$ 92 4-Bromofluorobenzene (					0.4436664	0.4655361	0.4566990	0.4443318	0.4433731		0.4507213		Avg 2.2
94 Bromobenzene		0.9092616	0.8241995	0.8109603	0.8767634	0.8824847	0.8655097	0.8733770	0.8687187		0.8639094		Avg 3.7
93 1,1,2,2-Tetrachloroeth		0.4180889	0.3810394	0.3511784	0.3630711	0.3694100	0.3765310	0.3671502	0.3824443		0.3761142		Avg 5.3
95 1,2,3-Trichloropropane		0.3960412	0.3557020	0.3440065	0.3762937	0.3862085	0.3928225	0.3875391	0.4029018		0.3801894		Avg 5.4
96 trans-1,4-Dichloro-2-b		0.1622952	0.1358829	0.1089723	0.1202303	0.1221777	0.1251654	0.1187072	0.1270168		0.1275560		Avg 12.5
97 N-Propylbenzene		4.6839019	3.8789713	3.9317724	4.0916928	4.2234053	4.1310091	4.1450941	4.1189497		4.1505996		Avg 5.9
98 2-Chlorotoluene		2.5685519	2.1844955	2.1134230	2.2026504	2.2767257	2.2097508	2.2401391	2.2317902		2.2534408		Avg 6.0
99 1,3,5-Trimethylbenzene		3.4243275	2.8013978	2.7993459	2.9483198	3.0229015	2.9450727	2.9632742	2.9456027		2.9812803		Avg 6.6
100 4-Chlorotoluene		3.0431889	2.4798377	2.4979201	2.5732637	2.6661777	2.6117365	2.6347035	2.6190040		2.6407290		Avg 6.6
101 4-Isopropyltoluene		3.1479153	2.5503716	2.5889831	2.7734537	2.8751026	2.7937089	2.7975484	2.7767355		2.7879774		Avg 6.6
102 Pentachloroethane		0.1677071	0.1694036	0.1660472	0.1746404	0.1984938	0.1920066	0.1848028	0.1871908		0.1800366		Avg 6.8
103 1,2,4-Trimethylbenzene		3.3998302	2.8093218	2.7795104	2.8978201	2.9871217	2.8839913	2.9054156	2.8782658		2.9426596		Avg 6.6
104 sec-Butylbenzene		4.5485538	3.8328944	3.8146856	4.0050796	4.1602537	4.0500989	4.0239804	4.0010599		4.0545758		Avg 5.7
105 1,3-Dichlorobenzene		2.1306609	1.6590622	1.7200736	1.7206924	1.7528725	1.7064744	1.6967720	1.6961168		1.7603406		Avg 8.6
106 tert-Butylbenzene		3.8373128	3.3448314	3.3863373	3.4885418	3.6266393	3.5235428	3.4660087	3.4589410		3.5165194		Avg 4.4
* 107 1,4-Dichlorobenzene-d4	241113	244924	255551	244007	236338	229068	231961	238806	220774				
108 1,4-Dichlorobenzene		2.0841159	1.6644427	1.6517149	1.6729747	1.6929252	1.6411121	1.6356066	1.6342583		1.7096438		Avg 8.9
109 1,2,3-Trimethylbenzene		2.5035220	2.4880214	2.4372575	2.5391690	2.7525360	2.8125781	2.8359774	2.9443901		2.6641814		Avg 7.3
110 Benzyl chloride		0.1772784	0.1406817	0.1490542	0.1561080	0.1796848	0.1840063	0.1887842	0.1897844		0.1706728		Avg 11.2
112 n-Butylbenzene		3.4324933	2.8557900	2.8616802	2.9935516	3.1183797	3.0108273	2.9403672	2.9363365		3.0186782		Avg 6.2
111 1,2-Dichlorobenzene		1.7182881	1.4164492	1.4200822	1.4609902	1.4766401	1.4449304	1.4212457	1.4409940		1.4749525		Avg 6.8
113 1,2-Dibromo-3-Chloropr			0.0611424	0.0505313	0.0522874	0.0500070	0.0505882	0.0511852	0.0542274		0.0528527		Avg 7.4
114 1,3,5-Trichlorobenzene		1.2625733	1.2980933	1.3073277	1.2678072	1.3155379	1.3515759	1.2964180	1.3663107		1.3082055		Avg 2.8
115 1,2,4-Trichlorobenzene		1.1832242	1.0283662	1.0102169	1.0485301	1.0610343	1.0083980	0.9905083	0.9911221		1.0401750		Avg 6.1
116 Hexachlorobutadiene		0.8465891	0.7638397	0.7557980	0.7863632	0.8114141	0.7497640	0.7200978	0.7195458		0.7691765		Avg 5.7
117 Naphthalene		1.6441835	1.3522741	1.3534038	1.3740067	1.4063946	1.4145438	1.3477900	1.4341023		1.4158374		Avg 6.9
118 1,2,3-Trichlorobenzene		0.9507031	0.8211668	0.8128865	0.8449657	0.8613949	0.8347998	0.8041060	0.8216547		0.8439597		Avg 5.5
119 2-Methylnaphthalene		0.8061028	0.8559348	0.8212305	0.8748342	0.9337490	0.9858350	0.9464046	0.9969092		0.9026250		Avg 8.1

(60150)

ICalib Error Legend

R7, Calibration Average RF < Min. RF Limit

TestAmerica Laboratories
Initial Calibration RRF Report

Method: \\ChromNA\Chicago\ChromData\CMS16\20151120-34653.b\8260s16_test.m
Instrument: CMS16 Lims Location: 500
Lock State: Initial Calib Locked Cpnd Order: Retention Time
Integrator: RTE Last Modified: 23-Nov-2015 14:01:01
No.Compounds:125

Initial Calibration Batches

Ical Batch: \\ChromNA\Chicago\chromdata\CMS16\20150407-30118.b
Inj Date : 07-Apr-2015 13:08:30, Sublist: chrom-8260s16_test*sub1

Limit Group: MSVOA_8260_ICAL_SOIL_LOW

Detector 1: MS SCAN

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Lvl 6 b	M1	M2	%RSD/R^2
1 Dichlorodifluoromethan	0.2163475	0.1689497	0.1762375	0.2131963	0.2017249	0.1981674		0.1957706		Avg 9.9
2 Chloromethane	0.3958770	0.3315092	0.3286952	0.3405917	0.3226140	0.3077987		0.3378476		Avg 9.0
3 Vinyl chloride	0.4095082	0.3510121	0.3535987	0.3642230	0.3424229	0.3282193		0.3581640		Avg 7.8
4 Butadiene	0.3307733	0.3740739	0.3676184	0.3831320	0.3546257	0.3469133		0.3595228		Avg 5.3
5 Bromomethane	0.2460552	0.2318635	0.2054311	0.1997210	0.1934919	0.1880657		0.2107714		Avg 10.9
6 Chloroethane	0.4139305	0.3488451	0.2980467	0.3029216	0.2792216	0.2722328	0.8713257	0.2768436		Linr 0.998
7 Dichlorofluoromethane	0.6943932	0.6749488	0.6963777	0.6084156	0.6388821	0.5623208		0.6458897		Avg 8.2
8 Trichlorofluoromethane	0.6042817	0.5049986	0.5791623	0.5152663	0.5102618	0.4639465		0.5296528		Avg 9.8
9 Ethanol	0.0833061	0.0828460	0.0743571	0.0904397	0.0709174	0.0626398		0.0774177		Avg 13.0
10 Ethyl ether	0.2507724	0.2105733	0.2174248	0.2165536	0.2104068	0.2014306		0.2178602		Avg 7.9
11 Acrolein	0.0256881	0.0219661	0.0223224	0.0232886	0.0235064	0.0214789		0.0230418		Avg 6.6
12 1,1-Dichloroethene	0.1896921	0.2097705	0.2075287	0.2258280	0.2117519	0.2162082		0.2101299		Avg 5.7
13 1,1,2-Trichloro-1,2,2-	0.2633804	0.2276363	0.2245362	0.2408071	0.2253642	0.2289016		0.2351043		Avg 6.4
14 Acetone	0.0845446	0.0624195	0.0621443	0.0667060	0.0684775	0.0604393	0.1046729	0.0623669		Linr 0.995
15 Iodomethane	0.3924085	0.3472515	0.3360264	0.3701925	0.3496075	0.3578730		0.3588932		Avg 5.6
16 Carbon disulfide	0.7006192	0.6204182	0.6393585	0.7221199	0.6737779	0.6841172		0.6734018		Avg 5.6
17 Isopropyl alcohol	0.5792486	0.6218803	0.5782830	0.7232335	0.7051207	0.7177311		0.6542495		Avg 10.5
18 3-Chloro-1-propene	0.1508103	0.1261715	0.1239496	0.1433110	0.1265240	0.1401704		0.1351561		Avg 8.2
19 Acetonitrile	0.0181169	0.0185266	0.0187821	0.0195658	0.0204817	0.0216646		0.0195230		Avg 6.9
20 Methyl acetate	0.1636229	0.1427656	0.1425982	0.1585831	0.1533210	0.1417733		0.1504440		Avg 6.3
21 Methylene Chloride	0.2592008	0.2219025	0.2208614	0.2217767	0.2267476	0.2318837		0.2303955		Avg 6.4
* 22 TBA-d9 (IS)	145517	137872	150509	175124	200258	180989				
23 2-Methyl-2-propanol	1.4790216	1.1420518	1.2288830	1.2912428	1.2076277	1.2488187		1.2662743		Avg 9.1
24 Acrylonitrile	0.0746161	0.0699663	0.0682833	0.0776933	0.0739878	0.0708097		0.0725594		Avg 4.8
25 trans-1,2-Dichloroethe	0.2690514	0.2289412	0.2289682	0.2521442	0.2386547	0.2448876		0.2437745		Avg 6.3
26 Methyl tert-butyl ethe	0.6462505	0.5591693	0.5621534	0.6341560	0.6205715	0.6125497		0.6058084		Avg 6.1
27 Hexane	0.5113433	0.4338281	0.4289565	0.4565772	0.4207899	0.4261102		0.4462675		Avg 7.7
28 1,1-Dichloroethane	0.5299518	0.4545806	0.4492891	0.4887838	0.4532647	0.4597065		0.4725961		Avg 6.7
29 Vinyl acetate	0.2408004	0.2305546	0.2353101	0.2842267	0.2881563	0.2695715		0.2581033		Avg 9.9
30 Isopropyl ether	0.8689723	0.9173039	0.8690336	0.9162490	0.8717129	0.8941926		0.8895774		Avg 2.6
31 2-Chloro-1,3-butadiene	0.4029172	0.4548197	0.4071200	0.4562669	0.4383650	0.4368484		0.4327229		Avg 5.3
32 Tert-butyl ethyl ether	47.677446	49.041510	50.338149	59.229560	52.657717	52.851035		51.965903		Avg 7.9
34 2,2-Dichloropropane	0.9623347	0.4097501	0.2948284	0.2729890	0.2496092	0.2427162	3.6775220	0.2266028		Linr 0.999
35 cis-1,2-Dichloroethene	0.2715140	0.2374905	0.2447115	0.2676409	0.2470786	0.2511769		0.2532687		Avg 5.3
36 2-Butanone (MEK)	0.1253515	0.0875692	0.0794876	0.0936336	0.0902812	0.0842107	0.1509413	0.0860849		Linr 0.996
37 Ethyl acetate	0.1212516	0.1226450	0.1136739	0.1203646	0.1212786	0.1261828		0.1208994		Avg 3.4
38 Propionitrile	0.0240099	0.0260660	0.0266868	0.0262739	0.0259888	0.0280429		0.0261781		Avg 5.0
39 Chlorobromomethane	0.1302594	0.1047627	0.1043773	0.1124584	0.1092665	0.1078794		0.1115006		Avg 8.7
40 Methacrylonitrile	0.1140676	0.1259855	0.1151588	0.1256612	0.1242062	0.1251219		0.1217002		Avg 4.5
41 Tetrahydrofuran	0.0786569	0.0556457	0.0578168	0.0610146	0.0608856	0.0545793	0.2105095	0.0563166		Linr 0.994
42 Chloroform	0.4719411	0.3979147	0.3969203	0.4261429	0.3975504	0.4029431		0.4155687		Avg 7.2
43 1,1,1-Trichloroethane	0.3618684	0.3214019	0.3245319	0.3534492	0.3359333	0.3397136		0.3394830		Avg 4.7
$ 44 Dibromofluoromethane		0.2197039	0.2254832	0.2188926	0.2227074	0.2198977		0.2213370		Avg 1.2
45 Cyclohexane	0.5310790	0.4604488	0.4587791	0.4895244	0.4591461	0.4575472		0.4760874		Avg 6.2

(051-004)

Method: \\ChromNA\Chicago\ChromData\CMS16\20151120-34653.b\8260s16_test.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
46 Carbon tetrachloride	0.3324555	0.2917640	0.2872102	0.3172624	0.2973929	0.3030090		0.3048490		Avg 5.6
47 1,1-Dichloropropene	0.3390630	0.2997044	0.3010085	0.3235950	0.3032682	0.3080779		0.3124528		Avg 5.0
$ 48 1,2-Dichloroethane-d4		0.2573295	0.2599612	0.2521842	0.2541402	0.2512951		0.2549820		Avg 1.4
49 Benzene	1.0959585	0.9485307	0.9247733	0.9789333	0.9112476	0.9163161		0.9626266		Avg 7.3
50 Isobutyl alcohol	0.4947679	0.5252415	0.5300951	0.5738829	0.5259768	0.5733452		0.5372182		Avg 5.8
51 1,2-Dichloroethane	0.3435373	0.3160477	0.3113473	0.3405227	0.3217735	0.3210839		0.3257187		Avg 4.1
52 Isooctane	1.0169763	0.9990623	0.8933100	1.0960650	1.0527270	1.0081609		1.0110502		Avg 6.7
53 Tert-amyl methyl ether	33.301904	37.700360	39.055391	45.741155	41.716038	41.741734		39.876097		Avg 10.6
* 55 Fluorobenzene (IS)	499121	547245	566975	596107	610674	629451				
54 n-Heptane	0.5156095	0.4304907	0.4278283	0.4627377	0.4288614	0.4338630		0.4498984		Avg 7.7
56 Trichloroethene	0.2568596	0.2272290	0.2254644	0.2447197	0.2290505	0.2348090		0.2363554		Avg 5.2
57 n-Butanol	0.0039752	0.0039740	0.0041303	0.0042659	0.0048112	0.0051043		0.0043768		Avg 10.8
58 Methylcyclohexane	0.4750628	0.3943795	0.3960699	0.4233832	0.3988695	0.3978446		0.4142683		Avg 7.6
59 Ethyl acrylate	0.2074203	0.2417317	0.2226686	0.2584345	0.2584979	0.2708199		0.2432622		Avg 10.0
60 1,2-Dichloropropane	0.2763525	0.2360314	0.2390154	0.2600530	0.2416294	0.2455202		0.2497670		Avg 6.2
* 61 1,4-Dioxane-d8	9112	9362	9614	10756	12782	13624				
62 Dibromomethane	0.1447404	0.1177211	0.1147833	0.1250945	0.1205730	0.1196264		0.1237565		Avg 8.7
63 1,4-Dioxane	1.1171484	1.5778107	1.3179377	1.5628039	1.1613568	1.0157648	-71.73959	1.7980088	-0.0001894	Quad 0.990
64 Methyl methacrylate	0.1494998	0.1803824	0.1682308	0.1862515	0.1887347	0.1948322		0.1779886		Avg 9.3
65 Dichlorobromomethane	0.3013431	0.2793276	0.2838347	0.3152919	0.3000700	0.3060691		0.2976561		Avg 4.6
66 2-Nitropropane	0.0583163	0.0708003	0.0664394	0.0806985	0.0824491	0.0870728		0.0742961		Avg 14.7
67 2-Chloroethyl vinyl et	0.1261252	0.1301193	0.1294887	0.1531667	0.1524399	0.1465561		0.1396493		Avg 8.9
68 cis-1,3-Dichloropropen	0.4729202	0.4554551	0.4559609	0.5291329	0.5015864	0.5126190		0.4879457		Avg 6.3
69 4-Methyl-2-pentanone (	0.2474528	0.2399120	0.2558840	0.2903805	0.2815994	0.2598865		0.2625192		Avg 7.5
$ 70 Toluene-d8 (Surr)		1.1994255	1.2229534	1.1661769	1.1481238	1.1819616		1.1837283		Avg 2.4
71 Toluene	0.9051093	0.8370712	0.8120702	0.8903215	0.8101209	0.8394925		0.8490309		Avg 4.7
72 trans-1,3-Dichloroprop	0.3801069	0.3567397	0.3715491	0.4342418	0.4115765	0.4207990		0.3958355		Avg 7.8
73 Ethyl methacrylate	0.2874914	0.2883961	0.2951857	0.3580440	0.3442421	0.3470319		0.3200652		Avg 10.3
74 1,1,2-Trichloroethane	0.2553665	0.2375420	0.2318962	0.2558059	0.2399354	0.2377258		0.2430453		Avg 4.1
75 Tetrachloroethene	0.4074093	0.3495894	0.3361810	0.3694234	0.3432029	0.3512810		0.3595145		Avg 7.2
76 1,3-Dichloropropane	0.4866703	0.4450813	0.4283558	0.4666654	0.4386037	0.4330653		0.4497403		Avg 5.0
78 2-Hexanone	0.1757098	0.1625555	0.1755141	0.2011016	0.1936162	0.1771260		0.1809372		Avg 7.7
79 Chlorodibromomethane	0.2775497	0.2548297	0.2573323	0.2937465	0.2787367	0.2826636		0.2741431		Avg 5.5
80 n-Butyl acetate	0.2061432	0.2687259	0.2551865	0.2915035	0.2968460	0.3044706	-0.4397450	0.2918558		Linr 0.997
81 Ethylene Dibromide	0.2439411	0.2301495	0.2308172	0.2540315	0.2418890	0.2382284		0.2398428		Avg 3.7
* 82 Chlorobenzene-d5	350014	375276	391486	404640	425937	432277				
83 Chlorobenzene	1.0140469	0.9242076	0.8928956	0.9491395	0.8654110	0.8922903		0.9229985		Avg 5.8
84 1-Chlorohexane	0.4001320	0.4059301	0.3688673	0.4153236	0.4019006	0.3891325		0.3968810		Avg 4.1
85 1,1,1,2-Tetrachloroeth	0.3331932	0.2827911	0.2894811	0.3320978	0.3072942	0.3173190		0.3103627		Avg 6.8
86 Ethylbenzene	0.5147641	0.4872108	0.5012722	0.5414171	0.4927171	0.5146930		0.5086791		Avg 3.8
87 m-Xylene & p-Xylene	1.3311654	1.2123961	1.1985440	1.2842268	1.1662190	1.1949745		1.2312543		Avg 5.1
88 o-Xylene	1.2748541	1.2184393	1.2417194	1.3197116	1.2005168	1.2391419		1.2490639		Avg 3.4
89 Styrene	1.0533683	1.0040783	1.0281507	1.1302483	1.0503185	1.0814799		1.0579407		Avg 4.2
90 Bromoform	0.1633693	0.1568409	0.1554487	0.1819238	0.1758512	0.1784306		0.1686441		Avg 6.8
91 Isopropylbenzene	3.3942652	3.0110295	2.9436509	3.0134157	2.6464704	2.6761155		2.9474912		Avg 9.3
92 Cyclohexanone	0.0146422	0.0177646	0.0187236	0.0145943	0.0184491	0.0195958		0.0172950		Avg 12.5
$ 93 4-Bromofluorobenzene (		0.4211603	0.4483814	0.4489277	0.4402026	0.4470736		0.4411491		Avg 2.7
95 Bromobenzene	0.7954083	0.6988080	0.6833964	0.7339343	0.6538263	0.6826754		0.7080081		Avg 7.1
96 1,1,2,2-Tetrachloroeth	0.6609795	0.5550438	0.5521600	0.5750328	0.5168010	0.5025023		0.5604199		Avg 10.0
97 1,2,3-Trichloropropane	0.1898499	0.1715494	0.1620326	0.1730596	0.1583662	0.1501424		0.1675000		Avg 8.3
98 trans-1,4-Dichloro-2-b	0.1834126	0.2159431	0.2064130	0.2247593	0.2055113	0.2069663		0.2071676		Avg 6.7
99 N-Propylbenzene	4.0145087	3.6637975	3.5145293	3.5459569	3.0778638	3.0595684		3.4793708		Avg 10.5
100 2-Chlorotoluene	2.2919287	2.0905892	2.0089962	2.0595606	1.8021509	1.8599176		2.0188572		Avg 8.7
101 4-Chlorotoluene	2.6852575	2.4207306	2.3414535	2.4900538	2.2171202	2.2848461		2.4065769		Avg 6.9
102 1,3,5-Trimethylbenzene	2.8003744	2.5219602	2.4874114	2.5921191	2.2907516	2.3556530		2.5080450		Avg 7.2
103 tert-Butylbenzene	2.2795570	2.1188224	2.0904690	2.1458684	1.8964481	1.9265822		2.0762912		Avg 6.9
104 Pentachloroethane	0.3679645	0.2901439	0.3204402	0.4080686	0.4161027	0.4008175		0.3672562		Avg 14.1
105 1,2,4-Trimethylbenzene	2.7347945	2.5432499	2.5139824	2.5952775	2.2831721	2.3553537		2.5043050		Avg 6.5
106 sec-Butylbenzene	3.5754148	3.2869107	3.2651718	3.2919182	2.8592521	2.8636148		3.1903804		Avg 8.8
107 1,3-Dichlorobenzene	1.5480690	1.3542863	1.3357144	1.4041428	1.2454894	1.2867100		1.3624020		Avg 7.8
108 4-Isopropyltoluene	2.9972652	2.7720944	2.7508430	2.8221168	2.4652281	2.4899745		2.7162537		Avg 7.5
* 109 1,4-Dichlorobenzene-d4	172117	193228	208557	225141	243593	247259				

(051-005)

Method: \\ChromNA\Chicago\ChromData\CMS16\20151120-34653.b\8260s16_test.m

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	b	M1	M2	%RSD/R^2
110 1,4-Dichlorobenzene	1.5300144	1.3584412	1.3493734	1.4205366	1.2666811	1.3225186		1.3745942		Avg 6.6
111 1,2,3-Trimethylbenzene	2.4297731	2.7460684	2.4924749	2.7582938	2.7494555	2.6677210		2.6406311		Avg 5.5
112 Benzyl chloride	0.1377203	0.1546330	0.1488575	0.1819179	0.1947012	0.1985654	-0.4815161	0.1928063		Linr 0.994
113 1,2-Dichlorobenzene	1.3996089	1.2381841	1.2372954	1.2747858	1.1452295	1.1719902		1.2445157		Avg 7.2
114 n-Butylbenzene	2.7962003	2.6172430	2.5986054	2.6614679	2.3219736	2.3889717		2.5640770		Avg 6.9
115 1,2-Dibromo-3-Chloropr	0.0993255	0.0836357	0.0793271	0.0853062	0.0793845	0.0734420		0.0834035		Avg 10.6
116 1,3,5-Trichlorobenzene	1.0103484	1.1293857	1.0153754	1.0893060	1.1367861	1.1389113		1.0866855		Avg 5.5
117 1,2,4-Trichlorobenzene	1.0289615	0.8655082	0.8829197	0.8688776	0.7410616	0.7656264		0.8588258		Avg 11.9
118 Hexachlorobutadiene	0.6235124	0.5249739	0.5174998	0.5069009	0.4466425	0.4540909	0.7686130	0.4749069		Linr 0.996
119 Naphthalene	1.6297422	1.6195842	1.6732144	1.6384812	1.4482811	1.3740187		1.5638870		Avg 7.8
120 1,2,3-Trichlorobenzene	0.8802499	0.7848739	0.7853931	0.7488037	0.6478470	0.6463854		0.7489255		Avg 12.0
121 2-Methylnaphthalene	0.6305298	0.8338886	1.0044565	0.9366549	1.2271984	1.2075112	-1.2498125	0.8742396	0.0018273	Quad 0.995

(051-006)

Target and Internal Standards

Fluorobenzene	**1,4-Dioxane-d8**
Acetone	1,4-Dioxane
Acetonitrile	
Acrolein	**Chlorobenzene-d_5**
Acrylonitrile	Bromoform
Benzne	Bromofluorobenzene (surrogate)
Bromochloromethane	2-Chloroethyl vinyl ether
Bromomethane	Chlorodibromomethane
Butadiene	Chlorobenzene
2-Butanone	
Carbon disulfide	cis-1,3-Dichloropropene
Carbon Tetrachloride	1,3-Dichloropropane
2-Chloro-1,3-butadiene	Ethylbenzene
Chloroethane	Ethylene dibromide
Chloroform	Ethyl methacrylate
Chloromethane	2-Hexanone
3-Chloro-1-propene	4-Methyl-2-pentanone
Cyclohexane	n-Butyl acetate
Dibromofluoromethane (surr)	2-Nitropropane
Dibromomethane	Styrene
Dichlorobromomethane	1,1,1,2-Tetrachloroethane
Dichlorodifluoromethane	Tetrachloroethene
1,1-Dichloroethane	Toluene
1,2-Dichloroethane	Toluene-d_8 (surrogate)
1,2-Dichloroethane-d4 (surr)	trans-1,3-Dichloropropene
1,1-Dichloroethene	1,1,2-Trichloroethane
1,2-Dichloroethane (total)	m,p-Xylene
cis-1,2-Dichloroethene	o-Xylene
trans-1,2-Dichloroethene	Xylene (total)
Dichlorofluoromethane	
1,1-Dichloropropene	**1,4-Dichlorobenzene-d_4**
1,2-Dichloropropane	Benzyl chloride
2,2-Dichloropropane	Bromobenzene
Ethyl Acetate	n-Butylbenzene
Ethyl Acrylate	sec-Butylbenzene
Ethyl Ether	tert-Butylbenzene
Hexane	2-Chlorotoluene
Iodomethane	4-Chlorotoluene
Isopropyl ether	Cyclohexanone
Methacrylonitrile	1,2-Dibromo-3-chloropropane
Methyl Acetate	1,2-Dichlorobenzene
Methylcyclohexane	1,3-Dichlorobenzene
Methylene chloride	1,4-Dichlorobenzene
Methyl methacrylate	Hexachlorobutadiene
Methyl-tert-butyl ether	Isopropyl benzene
n-Butanol	p-Isopropyltoluene
n-Heptane	2-Methylnaphthalene
Propionitrile	Naphthalene
Tert-amyl methyl ether	Pentachloroethane
Tert-butyl ethyl ether	n-Propylbenzene
Tetrahydrofuran	trans-1,4-Dichloro-2-butene
1,1,1-Trichloroethane	1,1,2,2-Tetrachloroethane
Trichloroethene	1,2,3-Trichlorobenzene
Trichlorofluoromethane	1,2,4-Trichlorobenzene
1,1,2-Trichloro-1,2,2-trifluoroethane	1,3,5-Trichlorobenzene
Vinyl acetate	1,2,3-Trichloropropane
Vinyl Chloride	Trimethylbenzene (total)
	1,2,3-Trimethylbenzene
TBA-d9	1,2,4-Trimethylbenzene
Ethanol	1,3,5-Trimethylbenzene
Isobutyl alcohol	
Isopropyl alcohol	
2-Methyl-2-propanol	

Attachment 3.

Example: Sample Run Log; Corrective Action/Qualification Report; GC/MS VOA Maintenance Logbook; Sample Tracking Sheet; GC/MS VOA- ICOC Form (053-001 to 053-006)

TestAmerica Laboratories
Worklist Run Log Report

Worklist Name: 111615_INST25PM Worklist Num: 34579
Instrument: CMS25 Method: 8260W25cps
Batch Directory: \\ChromNA\Chicago\ChromData\CMS25\20151116-34579.b
Anaylysis Type: VOA Creator: Alikpala, Elaine M
Inj Volume: 5.00 Inj Vol Units: mL
Run Reagents:
8260LOW IS/SS_00083, Amount Added: 5.00 , Units: uL

Lab ID	Worklist ID	Sample Type	Inj Date/Time	File Name	Vial	Dil Factor	Client ID	Fract
BFB	500-0034579-001	BFB	16-Nov-2015 21:05:30	25B1116P.D	28	1.0		voaWater
CCVIS	500-0034579-002	CCVIS	16-Nov-2015 21:37:30	25C1116P.D	29	1.0		voaWater
CCV	500-0034579-003	CCV	16-Nov-2015 22:05:30	25D1116P.D	30	1.0		voaWater
LCS	500-0034579-004	LCS	16-Nov-2015 22:33:30	25S1116P.D	31	1.0		voaWater
BLANK	500-0034579-005	Client	16-Nov-2015 23:01:30	POS32P.D	32	1.0		voaWater
MB	500-0034579-006	MB	16-Nov-2015 23:28:30	25M1116P.D	33	1.0		voaWater
500-103669-A-1	500-0034579-007	Client	16-Nov-2015 23:56:30	103669-01.D	34	1.0	Trip Blank	voaWater
500-103669-A-2	500-0034579-008	Client	17-Nov-2015 00:24:30	103669-02.D	34	1.0	PMW-03-EQB	voaWater
500-103669-A-10	500-0034579-009	Client	17-Nov-2015 00:52:30	103669-10.D	35	1.0	MW-03	voaWater
500-103669-A-11	500-0034579-010	Client	17-Nov-2015 01:19:30	103669-11.D	36	1.0	S111-PP-3B	voaWater
500-103669-A-12	500-0034579-011	Client	17-Nov-2015 01:47:30	103669-12.D	37	1.0	MW-21	voaWater
500-103669-A-13	500-0034579-012	Client	17-Nov-2015 02:15:30	103669-13.D	38	1.0	MW-22	voaWater
500-103669-A-14	500-0034579-013	Client	17-Nov-2015 02:43:30	103669-14.D	39	1.0	PZ-1	voaWater
500-103669-A-8	500-0034579-014	Client	17-Nov-2015 03:11:30	103669-08.D	40	1.0	IW-01	voaWater
500-103669-A-8	500-0034579-015	Client	17-Nov-2015 03:39:30	103669-08A.D	41	5.0	IW-01	voaWater
500-103669-A-3	500-0034579-016	Client	17-Nov-2015 04:07:30	103669-03.D	42	2.0	PMW-03	voaWater
500-103669-A-3	500-0034579-017	Client	17-Nov-2015 04:35:30	103669-03A.D	43	20.0	PMW-03	voaWater
500-103669-A-4	500-0034579-018	Client	17-Nov-2015 05:03:30	103669-04.D	44	2.0	MW-19	voaWater
500-103669-A-4	500-0034579-019	Client	17-Nov-2015 05:31:30	103669-04A.D	45	20.0	MW-19	voaWater
500-103669-A-5	500-0034579-020	Client	17-Nov-2015 05:59:30	103669-05.D	46	5.0	PMW-02	voaWater
500-103669-A-5	500-0034579-021	Client	17-Nov-2015 06:27:30	103669-05A.D	47	50.0	PMW-02	voaWater
500-103669-A-6	500-0034579-022	Client	17-Nov-2015 06:55:30	103669-06.D	48	5.0	PMW-02 DUP	voaWater
500-103669-A-6	500-0034579-023	Client	17-Nov-2015 07:22:30	103669-06A.D	49	50.0	PMW-02 DUP	voaWater
500-103669-A-7	500-0034579-024	Client	17-Nov-2015 07:50:30	103669-07.D	50	5.0	IW-02	voaWater
500-103669-A-7	500-0034579-025	Client	17-Nov-2015 08:18:30	103669-07A.D	51	50.0	IW-02	voaWater
500-103669-A-2 MS	500-0034579-026	MS	17-Nov-2015 08:46:30	103669-02S.D	52	1.0		voaWater
500-103669-A-2 MSD	500-0034579-027	MSD	17-Nov-2015 09:14:30	103669-02T.D	53	1.0		voaWater

(053-001)

TestAmerica Laboratories
Worklist Run Log Report

Worklist Name: 040715ICAL16 Worklist Num: 30118
Instrument: CMS16 Method: 8260s16_test
Batch Directory: \\ChromNA\Chicago\ChromData\CMS16\20150407-30118.b
Anaylysis Type: VOA Creator: Werner, Brian D
Inj Volume: 5.00 Inj Vol Units: mL
Run Reagents:
INST16 IS_00012, Amount Added: 0.87 , Units: uL

Lab ID	Worklist ID	Sample Type	Cal Lvl	Inj Date/Time	File Name	Vial	Dil Factor	Fract
BFB	500-0030118-001	BFB	0	07-Apr-2015 11:43:30	16B0407.D	1	1.0	voaSoilLL
CCVIS	500-0030118-002	CCVIS	0	07-Apr-2015 12:20:30	16C0407.D	2	1.0	voaSoilLL
MB	500-0030118-003	MB	0	07-Apr-2015 12:44:30	16M0407.D	3	1.0	voaSoilLL
STD1	500-0030118-004	IC	1	07-Apr-2015 13:08:30	16I0407A.D	4	1.0	voaSoilLL
STD2	500-0030118-005	IC	2	07-Apr-2015 13:32:30	16I0407B.D	5	1.0	voaSoilLL
STD3	500-0030118-006	ICIS	3	07-Apr-2015 13:56:30	16I0407C.D	6	1.0	voaSoilLL
STD4	500-0030118-007	IC	4	07-Apr-2015 14:20:30	16I0407D.D	7	1.0	voaSoilLL
STD5	500-0030118-008	IC	5	07-Apr-2015 14:44:30	16I0407E.D	8	1.0	voaSoilLL
STD6	500-0030118-009	IC	6	07-Apr-2015 15:08:30	16I0407F.D	9	1.0	voaSoilLL
STD1AP	500-0030118-010	IC	1	07-Apr-2015 15:32:30	16J0407A.D	10	1.0	voaSoilLL
STD2AP	500-0030118-011	IC	2	07-Apr-2015 15:57:30	16J0407B.D	11	1.0	voaSoilLL
STD3AP	500-0030118-012	IC	3	07-Apr-2015 16:21:30	16J0407C.D	12	1.0	voaSoilLL
STD4AP	500-0030118-013	IC	4	07-Apr-2015 16:45:30	16J0407D.D	13	1.0	voaSoilLL
STD5AP	500-0030118-014	IC	5	07-Apr-2015 17:09:30	16J0407E.D	14	1.0	voaSoilLL
STD6AP	500-0030118-015	IC	6	07-Apr-2015 17:32:30	16J0407F.D	15	1.0	voaSoilLL
BLANK	500-0030118-016	Client	0	07-Apr-2015 17:56:30	BLA01.D	16	1.0	voaSoilLL
ICV	500-0030118-017	ICV	0	07-Apr-2015 18:20:30	16ICV0407.D	17	1.0	voaSoilLL
CV	500-0030118-018	ICV	0	07-Apr-2015 18:45:30	16ICV0407A.D	18	1.0	voaSoilLL

(053-002)

Instrument ID# _____

TestAmerica Chicago

CHI-22-20-082/D-09/13

Corrective Action/Qualification Report GC/MS VOA and GC VOA

Analytical Methods
___SW846 8260B
___40CFR 624
___Other _____

CHROM Batch:_____
___8015B_GRO
___8015C_GRO
___WI_GRO

Tune Criteria
Description of Situation:_____
_____
Action Taken:_____
_____
Demonstration of Control:_____
_____
_____

Initial Calibration Criteria
Description of Situation:_____
_____
Action Taken:_____
_____
Demonstration of Control_____
_____

ICV Criteria Eval _____

Continuing Calibration Criteria
Description of Situation:_____
_____
Action Taken:_____
_____
Demonstration of Control:_____
_____

Internal Standards (CCV)
Description of situation:_____
_____
Action Taken:_____
_____
Demonstration of Control:_____
_____

(053-003)

Analyst Signature / Date _____/_____

Method Blank
Description of situation:_____
_____
Action Taken:_____
_____
Demonstration of Control:_____
_____

LCS
Description of Situation:_____
_____
_____
Action Taken:_____
_____
_____
Demonstration of Control:_____
_____

Qualification of Data
Data Affected (Client/Sample #)_____
_____
_____
_____
Qualification:_____
_____
_____
Associated samples reanalyzed: Yes No (see below)
Explanation for no reanalysis/data MUST be qualified and narrated:_____
_____
_____
_____
_____

Client contacted/NCM # _____
_____

Instrument Preventative Maintenance was performed per SOP: _____
Purge-line or sample transfer line rinses within the concentrator and vial autosampler.

Reviewer Signature/date_____/_____

TestAmerica Chicago
GC/MS VOA Maintenance Logbook **Page No.**_____
Instrument No. MS28

Date Problem was identified: _____ Entry No.:_____

Analyst:_____

Description of Problem/Situation:
Tune Criteria Failed: _____ (Filename)
ICAL Criteria Failed: _____ (Date performed)
CCV Criteria Failed: _____ (Filename)

Description of Problem:

Action Taken 1: _____ (Date) _____ (Initials)

Action Taken 2: _____ (Date) _____ (Initials)

Action Taken 3: _____ (Date) _____ (Initials)

Action Taken 4: _____ (Date) _____ (Initials)

Demonstration of Return to Control:
Tune Passed Criteria: _____(Filename)
ICAL Passed Criteria: _____ (Date performed)
CCV Passed Criteria: _____ (Filename)
Contamination is no longer present _____ (Method Blank Filename)

Analyst:_____ Date:_____

Reviewer Signature:_____ Date: _____

(053004)

TestAmerica Chicago

GC/MS Volatiles: Sample Tracking Sheet **Screener:** ______________ **Job #**

Sample File Name	Dilution	pH	Action	Inst./Date	Chrom Batch	MeOH Lot #	Analytical Batch	misc info

Reviewed by: ______________________________ Date: ______________

CHI-22-20-075/D-09/11

(053-005)

TestAmerica Chicago

GC/MS Volatiles: Internal Chain of Custody Form

ICOC Job Number:

Date	Time Out	Time In	ICOC Initials	Misc. Info

(053006)

CHI-22-20-074/A-01/08

Attachment 4.

Example: Continuing Calibration Evaluation and Acceptance Criteria (Form 7) (054-001 to 054-012)

Report Date: 01-Dec-2015 13:37:27 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

TestAmerica Chicago
ICV, ICal Verification Report

Data File: \\ChromNA\Chicago\ChromData\CMS25\20151113-34534.b\25S1109ICV1.D
Lims ID: ICV
Client ID:
Sample Type: ICV
Inject. Date: 13-Nov-2015 21:30:30 ALS Bottle#: 21 Worklist Smp#: 22
Purge Vol: 5.000 mL Dil. Factor: 1.0000
Sample Info: ICV 1
Misc. Info.: 500-0034534-022
Operator ID: PF Instrument ID: CMS25
Sublist:
Raw Data: Smoothed

Method: \\ChromNA\Chicago\ChromData\CMS25\20151113-34534.b\8260W25cps.m
Limit Group: MSVOA_8260_ICAL_WATER
Last Update: 13-Nov-2015 21:44:28 Calib Date: 13-Nov-2015 20:34:30
Integrator: RTE ID Type: RT Order ID
Quant Method: Internal Standard Quant By: Initial Calibration
Last ICal File: \\ChromNA\Chicago\ChromData\CMS25\20151113-34534.b\25J1112I.D

Column 1 : Det: MS SCAN
Process Host: XAWRK013

First Level Reviewer: alikpalae Date: 13-Nov-2015 22:29:16

Start Cal Date: 13-Nov-2015 12:42:30
End Cal Date: 13-Nov-2015 20:34:30

Compound	Amount Added	Amount Detected	%Drift	Max. %Drift	%Rec	%Rec Limits
1 Dichlorodifluorome	50.0	46.1	-7.9	30.0	92.1	
2 Chloromethane	50.0	45.5	-8.9	30.0	91.1	
3 Vinyl chloride	50.0	49.2	-1.6	30.0	98.4	
4 Butadiene	50.0	47.8	-4.4	30.0	95.6	
5 Bromomethane	50.0	57.2	14.5	30.0	114.5	
6 Chloroethane	50.0	49.5	-1.0	30.0	99.0	
7 Dichlorofluorometh	50.0	47.6	-4.7	30.0	95.3	
8 Trichlorofluoromet	50.0	49.1	-1.9	30.0	98.1	
10 Ethyl ether	50.0	50.8	1.5	30.0	101.5	
11 Acrolein	2000.0	2790.9	* 39.5	30.0	139.5	
12 1,1-Dichloroethene	50.0	46.9	-6.3	30.0	93.7	
13 1,1,2-Trichloro-1,	50.0	47.8	-4.4	30.0	95.6	
14 Acetone	50.0	62.2	24.5	30.0	124.5	
15 Iodomethane	50.0	46.9	-6.2	30.0	93.8	
16 Carbon disulfide	50.0	46.1	-7.7	30.0	92.3	
19 3-Chloro-1-propene	50.0	46.9	-6.2	30.0	93.8	
20 Methyl acetate	250.0	252.0	0.8	30.0	100.8	
21 Methylene Chloride	50.0	48.0	-4.1	30.0	95.9	

(054-001)

Report Date: 01-Dec-2015 13:37:27 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

Data File: \\ChromNA\Chicago\ChromData\CMS25\20151113-34534.b\25S1109ICV1.D

Compound	Amount Added	Amount Detected	%Drift	Max. %Drift	%Rec	%Rec Limits
23 2-Methyl-2-propano	500.0	491.4	-1.7	30.0	98.3	
24 Acrylonitrile	500.0	487.4	-2.5	30.0	97.5	
25 trans-1,2-Dichloro	50.0	49.6	-0.7	30.0	99.3	
26 Methyl tert-butyl	50.0	48.0	-3.9	30.0	96.1	
27 Hexane	50.0	48.5	-2.9	30.0	97.1	
28 1,1-Dichloroethane	50.0	46.5	-7.0	30.0	93.0	
29 Vinyl acetate	50.0	38.4	-23.1	30.0	76.9	
33 2,2-Dichloropropan	50.0	45.6	-8.9	30.0	91.1	
34 cis-1,2-Dichloroet	50.0	48.2	-3.5	30.0	96.5	
35 2-Butanone (MEK)	50.0	58.6	17.2	30.0	117.2	
39 Chlorobromomethane	50.0	49.9	-0.1	30.0	99.9	
40 Tetrahydrofuran	100.0	97.5	-2.5	30.0	97.5	
41 Chloroform	50.0	47.0	-6.0	30.0	94.0	
$ 42 Dibromofluorometha	30.0	27.3	-9.0	30.0	91.0	
43 1,1,1-Trichloroeth	50.0	47.9	-4.3	30.0	95.7	
44 Cyclohexane	50.0	46.2	-7.5	30.0	92.5	
46 Carbon tetrachlori	50.0	49.2	-1.6	30.0	98.4	
45 1,1-Dichloropropen	50.0	47.3	-5.5	30.0	94.5	
47 Isobutyl alcohol	1250.0	1222.7	-2.2	30.0	97.8	
$ 48 1,2-Dichloroethane	30.0	27.9	-7.1	30.0	92.9	
49 Benzene	50.0	45.8	-8.4	30.0	91.6	
50 1,2-Dichloroethane	50.0	48.7	-2.6	30.0	97.4	
54 n-Heptane	50.0	47.8	-4.4	30.0	95.6	
56 Trichloroethene	50.0	48.0	-4.0	30.0	96.0	
58 Methylcyclohexane	50.0	46.6	-6.7	30.0	93.3	
59 1,2-Dichloropropan	50.0	49.4	-1.2	30.0	98.8	
62 Dibromomethane	50.0	47.9	-4.2	30.0	95.8	
64 1,4-Dioxane	1000.0	1127.6	12.8	30.0	112.8	
65 Dichlorobromometha	50.0	47.5	-4.9	30.0	95.1	
67 2-Chloroethyl viny	50.0	49.2	-1.7	30.0	98.3	
68 cis-1,3-Dichloropr	50.0	47.9	-4.2	30.0	95.8	
69 4-Methyl-2-pentano	50.0	54.3	8.6	30.0	108.6	
$ 70 Toluene-d8 (Surr)	30.0	27.4	-8.7	30.0	91.3	
71 Toluene	50.0	46.1	-7.8	30.0	92.2	
72 trans-1,3-Dichloro	50.0	46.1	-7.9	30.0	92.1	
73 Ethyl methacrylate	50.0	47.2	-5.5	30.0	94.5	

(054-002)

Report Date: 01-Dec-2015 13:37:27 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

Data File: \\ChromNA\Chicago\ChromData\CMS25\20151113-34534.b\25S1109ICV1.D

Compound	Amount Added	Amount Detected	%Drift	Max. %Drift	%Rec	%Rec Limits
74 1,1,2-Trichloroeth	50.0	47.4	-5.2	30.0	94.8	
75 Tetrachloroethene	50.0	46.1	-7.9	30.0	92.1	
76 1,3-Dichloropropan	50.0	47.6	-4.9	30.0	95.1	
77 2-Hexanone	50.0	52.8	5.6	30.0	105.6	
79 Chlorodibromometha	50.0	47.8	-4.4	30.0	95.6	
80 Ethylene Dibromide	50.0	49.7	-0.7	30.0	99.3	
83 Chlorobenzene	50.0	47.2	-5.6	30.0	94.4	
84 1,1,1,2-Tetrachlor	50.0	48.1	-3.8	30.0	96.2	
85 Ethylbenzene	50.0	45.3	-9.4	30.0	90.6	
86 m-Xylene & p-Xylen	50.0	45.1	-9.9	30.0	90.1	
87 o-Xylene	50.0	44.5	-11.0	30.0	89.0	
88 Styrene	50.0	47.5	-5.0	30.0	95.0	
89 Bromoform	50.0	51.5	3.0	30.0	103.0	
90 Isopropylbenzene	50.0	45.9	-8.2	30.0	91.8	
$ 92 4-Bromofluorobenze	30.0	27.5	-8.5	30.0	91.5	
94 Bromobenzene	50.0	46.5	-7.0	30.0	93.0	
93 1,1,2,2-Tetrachlor	50.0	44.5	-11.0	30.0	89.0	
95 1,2,3-Trichloropro	50.0	46.3	-7.4	30.0	92.6	
96 trans-1,4-Dichloro	50.0	41.5	-17.0	30.0	83.0	
97 N-Propylbenzene	50.0	45.1	-9.8	30.0	90.2	
98 2-Chlorotoluene	50.0	45.0	-9.9	30.0	90.1	
99 1,3,5-Trimethylben	50.0	44.7	-10.6	30.0	89.4	
100 4-Chlorotoluene	50.0	44.5	-10.9	30.0	89.1	
101 4-Isopropyltoluene	50.0	46.4	-7.3	30.0	92.7	
103 1,2,4-Trimethylben	50.0	43.9	-12.2	30.0	87.8	
104 sec-Butylbenzene	50.0	45.5	-8.9	30.0	91.1	
105 1,3-Dichlorobenzen	50.0	44.8	-10.4	30.0	89.6	
106 tert-Butylbenzene	50.0	46.4	-7.2	30.0	92.8	
108 1,4-Dichlorobenzen	50.0	45.2	-9.5	30.0	90.5	
112 n-Butylbenzene	50.0	44.7	-10.7	30.0	89.3	
111 1,2-Dichlorobenzen	50.0	46.0	-8.0	30.0	92.0	
113 1,2-Dibromo-3-Chlo	50.0	46.1	-7.7	30.0	92.3	
115 1,2,4-Trichloroben	50.0	46.1	-7.8	30.0	92.2	
116 Hexachlorobutadien	50.0	45.5	-9.0	30.0	91.0	
117 Naphthalene	50.0	47.0	-6.0	30.0	94.0	
118 1,2,3-Trichloroben	50.0	45.5	-8.9	30.0	91.1	

(054-003)

Report Date: 01-Dec-2015 13:06:49 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

TestAmerica Chicago

CCVIS, Cal Verification ISTD Check Report

Data File: \\ChromNA\Chicago\ChromData\CMS25\20151116-34579.b\25C1116P.D
Lims ID: CCVIS
Client ID:
Sample Type: CCVIS
Inject. Date: 16-Nov-2015 21:37:30 ALS Bottle#: 29 Worklist Smp#: 2
Purge Vol: 5.000 mL Dil. Factor: 1.0000
Sample Info: CCVIS
Misc. Info.: 500-0034579-002
Operator ID: TT Instrument ID: CMS25
Sublist: chrom-8260W25cps*sub3
Raw Data: Smoothed

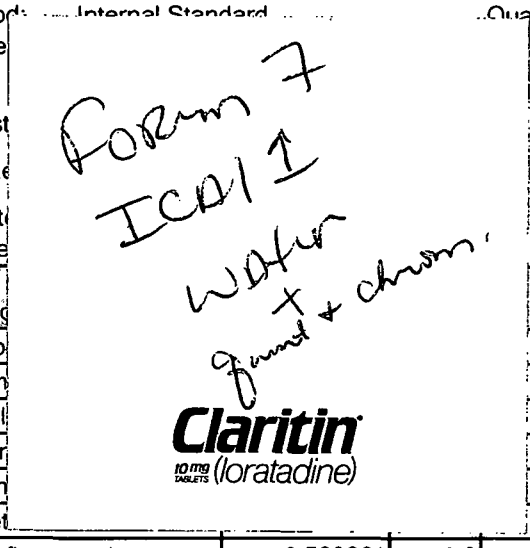
Method: \\ChromNA\Chicago\ChromData\CMS25\20151116-34579.b\8260W25cps.m
Limit Group: MSVOA_8260_ICAL_WATER
Last Update: 17-Nov-2015 10:08:39 Calib Date: 13-Nov-2015 20:34:30
Integrator: RTE ID Type: RT Order ID
Quant Method: Internal Standard Quant By: Initial Calibration
Last ICal File: 20151113-34534.b\25J1112I.D

Column 1 : Det: MS SCAN
Process Host:

First Level Re: e: 16-Nov-2015 22:42:16

Start Cal Date:
End Cal Date:

Form 7
ICAL 1
Water
+ Found + chrom.



Co			Ccal Amt	Ccal RF	Min. RRF	%D	Max. %D	%Rec
1 Dichloro				0.313129	0.010	-5.9	50	94
2 Chlorom			45.0	0.348773	0.100	-10	50	90
3 Vinyl chl				0.334673	0.010	-3.8	20	96
4 Butadier				0.408453	0.010	-1.4	50	99
5 Bromom				0.216300	0.010	20.9	50	121
6 Chloroet				0.195843	0.010	-4.0	50	96
7 Dichlorofluoromethane	0.520261	0.0		0.475328	0.010	-8.6	50	91
8 Trichlorofluoromethane	0.528322	0.0		0.503907	0.010	-4.6	50	95
10 Ethyl ether	0.146165	0.0		0.129839	0.010	-11.2	50	89
11 Acrolein	0.016936	0.0		0.014269	0.001	-15.8	50	84
12 1,1-Dichloroethene	0.284507	0.0		0.261912	0.010	-7.9	20	92
13 1,1,2-Trichloro-1,2,2-t	0.322204	0.0		0.308196	0.010	-4.3	50	96
14 Acetone	50.0	0.0	47.1	0.032146	0.010	-5.8	50	94
15 Iodomethane	0.421876	0.0		0.428013	0.010	1.5	50	101
16 Carbon disulfide	0.969925	0.0		0.842454	0.010	-13.1	50	87
19 3-Chloro-1-propene	0.151678	0.0		0.126984	0.010	-16.3	50	84
20 Methyl acetate	250.0	0.0	230.4	0.091054	0.010	-7.8	50	92
21 Methylene Chloride	0.255045	0.0		0.222529	0.010	-12.7	50	87
23 2-Methyl-2-propanol	500.0	0.0	481.5	1.176124	0.010	-3.7	50	96
24 Acrylonitrile	0.043684	0.0		0.039038	0.001	-10.6	50	89
25 trans-1,2-Dichloroethen	0.299928	0.0		0.280830	0.010	-6.4	50	94
26 Methyl tert-butyl ether	0.384643	0.0		0.331363	0.010	-13.9	50	86

(054-004)

Report Date: 01-Dec-2015 13:06:49 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

Data File: \\ChromNA\Chicago\ChromData\CMS25\20151116-34579.b\25C1116P.D

Compound	Standard RRF/Amt	DLT RT	Ccal Amt	Ccal RF	Min RRF	%D	Max. %D	%Rec
27 Hexane	0.527597	0.0		0.466714	0.010	-11.5	50	88
28 1,1-Dichloroethane	0.503553	0.0		0.456063	0.100	-9.4	50	91
29 Vinyl acetate	0.262620	0.0		0.207156	0.010	-21.1	50	79
33 2,2-Dichloropropane	0.429509	0.0		0.364012	0.010	-15.2	50	85
34 cis-1,2-Dichloroethene	0.302959	0.0		0.285354	0.010	-5.8	50	94
35 2-Butanone (MEK)	0.050469	0.0		0.050138	0.010	-0.7	50	99
39 Chlorobromomethane	0.129729	0.0		0.126878	0.010	-2.2	50	98
40 Tetrahydrofuran	100.0	0.0	93.8	0.031880	0.010	-6.2	50	94
41 Chloroform	0.473515	0.0		0.433734	0.010	-8.4	20	92
$ 42 Dibromofluoromethane	0.279911	0.0		0.259404	0.010	-7.3	50	93
43 1,1,1-Trichloroethane	0.472415	0.0		0.435741	0.010	-7.8	50	92
44 Cyclohexane	0.591190	0.0		0.532852	0.010	-9.9	50	90
46 Carbon tetrachloride	0.474937	0.0		0.445385	0.010	-6.2	50	94
45 1,1-Dichloropropene	0.396620	0.0		0.354382	0.010	-10.6	50	89
47 Isobutyl alcohol	1250.0	0.0	915.0	0.002899	0.001	-26.8	50	73
$ 48 1,2-Dichloroethane-d4 (	0.214623	0.0		0.193952	0.010	-9.6	50	90
49 Benzene	1.086600	0.0		0.961975	0.010	-11.5	50	89
50 1,2-Dichloroethane	0.272927	0.0		0.252879	0.010	-7.3	50	93
54 n-Heptane	0.663496	0.0		0.614470	0.010	-7.4	50	93
56 Trichloroethene	0.378220	0.0		0.356968	0.010	-5.6	50	94
58 Methylcyclohexane	0.531928	0.0		0.478222	0.010	-10.1	50	90
59 1,2-Dichloropropane	0.247577	0.0		0.224696	0.010	-9.2	20	91
62 Dibromomethane	0.118435	0.0		0.106971	0.010	-9.7	50	90
64 1,4-Dioxane	0.990091	0.0		1.053329	0.001	6.4	50	106
65 Dichlorobromomethane	0.315584	0.0		0.284394	0.010	-9.9	50	90
67 2-Chloroethyl vinyl eth	0.084751	0.0		0.055860	0.010	-34.1	50	66
68 cis-1,3-Dichloropropene	0.344759	0.0		0.298604	0.010	-13.4	50	87
69 4-Methyl-2-pentanone (M	0.105224	0.0		0.098209	0.010	-6.7	50	93
$ 70 Toluene-d8 (Surr)	1.015806	0.0		0.919795	0.010	-9.5	50	91
71 Toluene	0.704810	0.0		0.627974	0.010	-10.9	20	89
72 trans-1,3-Dichloroprope	0.343028	0.0		0.288540	0.010	-15.9	50	84
73 Ethyl methacrylate	50.0	0.0	40.5	0.157675	0.010	-19.1	50	81
74 1,1,2-Trichloroethane	0.200326	0.0		0.182905	0.010	-8.7	50	91
75 Tetrachloroethene	0.540808	0.0		0.477994	0.010	-11.6	50	88
76 1,3-Dichloropropane	0.330601	0.0		0.294625	0.010	-10.9	50	89
77 2-Hexanone	0.090275	0.0		0.078144	0.010	-13.4	50	87
79 Chlorodibromomethane	0.308367	0.0		0.289356	0.010	-6.2	50	94
80 Ethylene Dibromide	0.203318	0.0		0.187062	0.010	-8.0	50	92
83 Chlorobenzene	1.057572	0.0		0.982074	0.300	-7.1	50	93
84 1,1,1,2-Tetrachloroetha	0.368741	0.0		0.347761	0.010	-5.7	50	94
85 Ethylbenzene	0.614781	0.0		0.552201	0.010	-10.2	20	90
86 m-Xylene & p-Xylene	1.401052	0.0		1.214871	0.010	-13.3	50	87
87 o-Xylene	1.381581	0.0		1.225754	0.010	-11.3	50	89
88 Styrene	1.081144	0.0		1.001861	0.010	-7.3	50	93
89 Bromoform	0.159581	0.0		0.143497	0.100	-10.1	50	90

(054-005)

Report Date: 01-Dec-2015 13:06:49 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

Data File: \\ChromNA\Chicago\ChromData\CMS25\20151116-34579.b\25C1116P.D

Compound	Standard RRF/Amt	DLT RT	Ccal Amt	Ccal RF	Min. RRF	%D	Max. %D	%Rec
90 Isopropylbenzene	3.655445	0.0		3.407018	0.010	-6.8	50	93
$ 92 4-Bromofluorobenzene (S	0.450721	0.0		0.400134	0.010	-11.2	50	89
94 Bromobenzene	0.863909	0.0		0.818133	0.010	-5.3	50	95
93 1,1,2,2-Tetrachloroetha	0.376114	0.0		0.314975	0.300	-16.3	50	84
95 1,2,3-Trichloropropane	0.380189	0.0		0.315458	0.010	-17.0	50	83
96 trans-1,4-Dichloro-2-bu	0.127556	0.0		0.097003	0.010	-24.0	50	76
97 N-Propylbenzene	4.150600	0.0		3.735803	0.010	-10	50	90
98 2-Chlorotoluene	2.253441	0.0		2.020569	0.010	-10.3	50	90
99 1,3,5-Trimethylbenzene	2.981280	0.0		2.620233	0.010	-12.1	50	88
100 4-Chlorotoluene	2.640729	0.0		2.317541	0.010	-12.2	50	88
101 4-Isopropyltoluene	2.787977	0.0		2.662798	0.010	-4.5	50	96
103 1,2,4-Trimethylbenzene	2.942660	0.0		2.561417	0.010	-13.0	50	87
104 sec-Butylbenzene	4.054576	0.0		3.809062	0.010	-6.1	50	94
105 1,3-Dichlorobenzene	1.760341	0.0		1.597477	0.010	-9.3	50	91
106 tert-Butylbenzene	3.516519	0.0		3.267124	0.010	-7.1	50	93
108 1,4-Dichlorobenzene	1.709644	0.0		1.525398	0.010	-10.8	50	89
112 n-Butylbenzene	3.018678	0.0		2.603802	0.010	-13.7	50	86
111 1,2-Dichlorobenzene	1.474952	0.0		1.345817	0.010	-8.8	50	91
113 1,2-Dibromo-3-Chloropro	0.052853	0.0		0.042651	0.010	-19.3	50	81
115 1,2,4-Trichlorobenzene	1.040175	0.0		0.875028	0.010	-15.9	50	84
116 Hexachlorobutadiene	0.769176	0.0		0.717968	0.010	-6.7	50	93
117 Naphthalene	1.415837	0.0		1.264459	0.010	-10.7	50	89
118 1,2,3-Trichlorobenzene	0.843960	0.0		0.747016	0.010	-11.5	50	89
S 120 1,2-Dichloroethene, T	100.0		93.9			-6.1	50	94
S 121 Xylenes, Total	100.0		87.7			-12.3	50	88

(054-006)

Report Date: 01-Dec-2015 13:28:44 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

TestAmerica Chicago
ICV, ICal Verification Report

Data File: \\ChromNA\Chicago\ChromData\CMS16\20150407-30118.b\16ICV0407.D
Lims ID: ICV
Client ID:
Sample Type: ICV
Inject. Date: 07-Apr-2015 18:20:30 ALS Bottle#: 17 Worklist Smp#: 17
Purge Vol: 5.000 mL Dil. Factor: 1.0000
Sample Info: ICV
Misc. Info.: 500-0030118-017
Operator ID: BDW Instrument ID: CMS16
Sublist:

Method: \\ChromNA\Chicago\ChromData\CMS16\20150407-30118.b\8260s16_test.m
Limit Group: MSVOA_8260_ICAL_SOIL_LOW
Method Label: TAL Chicago VOA Report SW846 8260B
Last Update: 08-Apr-2015 08:35:57 Calib Date: 07-Apr-2015 17:32:30
Integrator: RTE ID Type: RT Order ID
Quant Method: Internal Standard Quant By: Initial Calibration
Last ICal File: \\ChromNA\Chicago\chromdata\CMS16\20150407-30118.b\16J0407F.D

Column 1 : Det: MS SCAN
Process Host: XAWRK013

First Level Reviewer: petruszakj Date: 01-Dec-2015 13:28:40

Start Cal Date: 07-Apr-2015 13:08:30
End Cal Date: 07-Apr-2015 17:32:30

Compound	Amount Added	Amount Detected	%Drift	Max %Drift	%Rec	%Rec Limits
1 Dichlorodifluorome	50.0	57.7	15.4	30.0	115.4	
2 Chloromethane	50.0	51.3	2.6	30.0	102.6	
3 Vinyl chloride	50.0	48.4	-3.2	30.0	96.8	
4 Butadiene	50.0	51.5	3.0	30.0	103.0	
5 Bromomethane	50.0	47.0	-6.1	30.0	93.9	
6 Chloroethane	50.0	49.5	-1.0	30.0	99.0	
7 Dichlorofluorometh	50.0	54.0	8.0	30.0	108.0	
8 Trichlorofluoromet	50.0	51.6	3.3	30.0	103.3	
10 Ethyl ether	50.0	53.4	6.8	30.0	106.8	
11 Acrolein	2000.0	2001.3	0.1	30.0	100.1	
12 1,1-Dichloroethene	50.0	53.3	6.7	30.0	106.7	
13 1,1,2-Trichloro-1,	50.0	49.2	-1.7	30.0	98.3	
14 Acetone	50.0	48.7	-2.7	30.0	97.3	
15 Iodomethane	50.0	55.3	10.5	30.0	110.5	
16 Carbon disulfide	50.0	52.8	5.6	30.0	105.6	
18 3-Chloro-1-propene	50.0	46.9	-6.2	30.0	93.8	
20 Methyl acetate	250.0	272.7	9.1	30.0	109.1	
21 Methylene Chloride	50.0	53.9	7.8	30.0	107.8	

(054-007)

Report Date: 01-Dec-2015 13:28:44 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

Data File: \\ChromNA\Chicago\ChromData\CMS16\20150407-30118.b\16ICV0407.D

Compound	Amount Added	Amount Detected	%Drift	Max. %Drift	%Rec	%Rec Limits
23 2-Methyl-2-propano	500.0	508.7	1.7	30.0	101.7	
25 trans-1,2-Dichloro	50.0	53.5	7.1	30.0	107.1	
24 Acrylonitrile	500.0	533.6	6.7	30.0	106.7	
26 Methyl tert-butyl	50.0	53.3	6.6	30.0	106.6	
27 Hexane	50.0	53.4	6.7	30.0	106.7	
28 1,1-Dichloroethane	50.0	53.7	7.4	30.0	107.4	
29 Vinyl acetate	50.0	42.1	-15.8	30.0	84.2	
34 2,2-Dichloropropan	50.0	53.6	7.2	30.0	107.2	
35 cis-1,2-Dichloroet	50.0	53.0	6.0	30.0	106.0	
36 2-Butanone (MEK)	50.0	48.1	-3.7	30.0	96.3	
39 Chlorobromomethane	50.0	52.8	5.7	30.0	105.7	
41 Tetrahydrofuran	100.0	106.2	6.2	30.0	106.2	
42 Chloroform	50.0	52.9	5.8	30.0	105.8	
43 1,1,1-Trichloroeth	50.0	52.0	4.0	30.0	104.0	
$ 44 Dibromofluorometha	30.0	28.7	-4.4	30.0	95.6	
45 Cyclohexane	50.0	51.8	3.6	30.0	103.6	
46 Carbon tetrachlori	50.0	51.3	2.6	30.0	102.6	
47 1,1-Dichloropropen	50.0	56.1	12.2	30.0	112.2	
$ 48 1,2-Dichloroethane	30.0	30.3	0.8	30.0	100.8	
49 Benzene	50.0	52.6	5.2	30.0	105.2	
50 Isobutyl alcohol	1250.0	1321.3	5.7	30.0	105.7	
51 1,2-Dichloroethane	50.0	51.3	2.5	30.0	102.5	
54 n-Heptane	50.0	51.3	2.5	30.0	102.5	
56 Trichloroethene	50.0	53.0	5.9	30.0	105.9	
58 Methylcyclohexane	50.0	51.5	3.1	30.0	103.1	
60 1,2-Dichloropropan	50.0	52.6	5.2	30.0	105.2	
62 Dibromomethane	50.0	53.0	6.0	30.0	106.0	
63 1,4-Dioxane	1000.0	830.5	-17.0	30.0	83.0	
65 Dichlorobromometha	50.0	51.7	3.3	30.0	103.3	
67 2-Chloroethyl viny	50.0	51.9	3.8	30.0	103.8	
68 cis-1,3-Dichloropr	50.0	54.6	9.1	30.0	109.1	
69 4-Methyl-2-pentano	50.0	52.2	4.4	30.0	104.4	
$ 70 Toluene-d8 (Surr)	30.0	29.3	-2.2	30.0	97.8	
71 Toluene	50.0	52.6	5.2	30.0	105.2	
72 trans-1,3-Dichloro	50.0	57.4	14.8	30.0	114.8	
73 Ethyl methacrylate	50.0	57.0	13.9	30.0	113.9	

(054-008)

Report Date: 01-Dec-2015 13:28:44 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

Data File: \\ChromNA\Chicago\ChromData\CMS16\20150407-30118.b\16ICV0407.D

Compound	Amount Added	Amount Detected	%Drift	Max. %Drift	%Rec	%Rec Limits
74 1,1,2-Trichloroeth	50.0	54.1	8.2	30.0	108.2	
75 Tetrachloroethene	50.0	51.7	3.5	30.0	103.5	
76 1,3-Dichloropropan	50.0	51.4	2.9	30.0	102.9	
78 2-Hexanone	50.0	52.3	4.7	30.0	104.7	
79 Chlorodibromometha	50.0	50.8	1.5	30.0	101.5	
81 Ethylene Dibromide	50.0	54.0	8.0	30.0	108.0	
83 Chlorobenzene	50.0	52.8	5.6	30.0	105.6	
85 1,1,1,2-Tetrachlor	50.0	53.0	6.1	30.0	106.1	
86 Ethylbenzene	50.0	52.6	5.3	30.0	105.3	
87 m-Xylene & p-Xylen	50.0	53.6	7.2	30.0	107.2	
88 o-Xylene	50.0	53.5	7.1	30.0	107.1	
89 Styrene	50.0	51.0	2.1	30.0	102.1	
90 Bromoform	50.0	48.5	-3.0	30.0	97.0	
91 Isopropylbenzene	50.0	53.5	6.9	30.0	106.9	
$ 93 4-Bromofluorobenze	30.0	29.7	-1.0	30.0	99.0	
95 Bromobenzene	50.0	52.6	5.2	30.0	105.2	
96 1,1,2,2-Tetrachlor	50.0	53.2	6.4	30.0	106.4	
97 1,2,3-Trichloropro	50.0	54.9	9.8	30.0	109.8	
98 trans-1,4-Dichloro	50.0	54.3	8.6	30.0	108.6	
99 N-Propylbenzene	50.0	53.4	6.9	30.0	106.9	
100 2-Chlorotoluene	50.0	53.3	6.7	30.0	106.7	
101 4-Chlorotoluene	50.0	51.9	3.8	30.0	103.8	
102 1,3,5-Trimethylben	50.0	52.9	5.8	30.0	105.8	
103 tert-Butylbenzene	50.0	54.4	8.9	30.0	108.9	
105 1,2,4-Trimethylben	50.0	53.3	6.6	30.0	106.6	
106 sec-Butylbenzene	50.0	54.3	8.6	30.0	108.6	
107 1,3-Dichlorobenzen	50.0	52.4	4.8	30.0	104.8	
108 4-Isopropyltoluene	50.0	52.5	5.1	30.0	105.1	
110 1,4-Dichlorobenzen	50.0	52.9	5.7	30.0	105.7	
113 1,2-Dichlorobenzen	50.0	53.6	7.2	30.0	107.2	
114 n-Butylbenzene	50.0	52.3	4.6	30.0	104.6	
115 1,2-Dibromo-3-Chlo	50.0	54.3	8.5	30.0	108.5	
117 1,2,4-Trichloroben	50.0	53.4	6.7	30.0	106.7	
118 Hexachlorobutadien	50.0	53.2	6.5	30.0	106.5	
119 Naphthalene	50.0	59.1	18.1	30.0	118.1	
120 1,2,3-Trichloroben	50.0	55.0	10.0	30.0	110.0	

(054-009)

Report Date: 01-Dec-2015 13:24:10 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

TestAmerica Chicago

CCVIS, Cal Verification ISTD Check Report

Data File: \\ChromNA\Chicago\ChromData\CMS16\20151120-34653.b\16C1120.D
Lims ID: CCVIS
Client ID:
Sample Type: CCVIS
Inject. Date: 20-Nov-2015 08:32:30 ALS Bottle#: 2 Worklist Smp#: 3
Purge Vol: 5.000 mL Dil. Factor: 1.0000
Sample Info: CCVIS
Misc. Info.: 500-0034653-003
Operator ID: BDW Instrument ID: CMS16
Sublist: chrom-8260s16_test*sub1

Method: \\ChromNA\Chicago\ChromData\CMS16\20151120-34653.b\8260s16_test.m
Limit Group: MSVOA_8260_ICAL_SOIL_LOW
Method Label: TAL Chicago VOA Report SW846 8260B
Last Update: 23-Nov-2015 14:01:01 Calib Date: 07-Apr-2015 17:32:30
Integrator: RTE ID Type: RT Order ID
Quant Method: Internal Standard Quant By: Initial Calibration
Last ICal File: \\ChromNA\Chicago\chromdata\CMS16\20150407-30118.b\16J0407F.D

Column 1 : Det: MS SCAN
Process Host: XAWRK013

First Level Reviewer: wernerb Date: 20-Nov-2015 08:51:06

Start Cal Date: 07-Apr-2015 13:08:30
End Cal Date: 07-Apr-2015 17:32:30

Compound	Standard RRF/Amt	DLT RT	Ccal Amt	Ccal RF	Min. RRF	%D	Max. %D	%Rec
1 Dichlorodifluoromethane	0.195771	0.0		0.234092	0.010	19.6	50	120
2 Chloromethane	0.337848	0.0		0.353366	0.100	4.6	50	105
3 Vinyl chloride	0.358164	0.0		0.334044	0.010	-6.7	20	93
4 Butadiene	0.359523	0.0		0.404819	0.010	12.6	50	113
5 Bromomethane	0.210771	0.0		0.130793	0.010	-37.9	50	62
6 Chloroethane	50.0	0.0	37.3	0.223826	0.010	-25.4	50	75
7 Dichlorofluoromethane	0.645890	0.0		0.542295	0.010	-16.0	50	84
8 Trichlorofluoromethane	0.529653	0.0		0.477978	0.010	-9.8	50	90
10 Ethyl ether	0.217860	0.0		0.214934	0.010	-1.3	50	99
11 Acrolein	0.023042	0.0		0.028032	0.001	21.7	50	122
12 1,1-Dichloroethene	0.210130	0.0		0.222255	0.010	5.8	20	106
13 1,1,2-Trichloro-1,2,2-t	0.235104	0.0		0.236450	0.010	0.6	50	101
14 Acetone	50.0	0.0	50.1	0.064624	0.010	0.3	50	100
15 Iodomethane	0.358893	0.0		0.314569	0.010	-12.4	50	88
16 Carbon disulfide	0.673402	0.0		0.684458	0.010	1.6	50	102
18 3-Chloro-1-propene	0.135156	0.0		0.131360	0.010	-2.8	50	97
20 Methyl acetate	0.150444	0.0		0.159023	0.010	5.7	50	106
21 Methylene Chloride	0.230395	0.0		0.219209	0.010	-4.9	50	95
23 2-Methyl-2-propanol	1.266274	0.0		1.207212	0.010	-4.7	50	95
24 Acrylonitrile	0.072559	0.0		0.069648	0.001	-4.0	50	96
25 trans-1,2-Dichloroethen	0.243775	0.0		0.230518	0.010	-5.4	50	95
26 Methyl tert-butyl ether	0.605808	0.0		0.612822	0.010	1.2	50	101

(054-010)

Report Date: 01-Dec-2015 13:24:10 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

Data File: \\ChromNA\Chicago\ChromData\CMS16\20151120-34653.b\16C1120.D

Compound	Standard RRF/Amt	DLT RT	Ccal Amt	Ccal RF	Min RRF	%D	Max %D	%Rec
27 Hexane	0.446268	0.0		0.455069	0.010	2.0	50	102
28 1,1-Dichloroethane	0.472596	0.0		0.459183	0.100	-2.8	50	97
29 Vinyl acetate	0.258103	0.0		0.309081	0.010	19.8	50	120
S 33 1,2-Dichloroethene, T	100.0		95.7			-4.3	50	96
34 2,2-Dichloropropane	50.0	0.0	39.8	0.253872	0.010	-20.4	50	80
35 cis-1,2-Dichloroethene	0.253269	0.0		0.245129	0.010	-3.2	50	97
36 2-Butanone (MEK)	50.0	0.0	47.2	0.084263	0.010	-5.6	50	94
39 Chlorobromomethane	0.111501	0.0		0.098390	0.010	-11.8	50	88
41 Tetrahydrofuran	100.0	0.0	114.3	0.066463	0.010	14.3	50	114
42 Chloroform	0.415569	0.0		0.408149	0.010	-1.8	20	98
43 1,1,1-Trichloroethane	0.339483	0.0		0.338091	0.010	-0.4	50	100
$ 44 Dibromofluoromethane	0.221337	0.0		0.221203	0.010	-0.06	50	100
45 Cyclohexane	0.476087	0.0		0.512226	0.010	7.6	50	108
46 Carbon tetrachloride	0.304849	0.0		0.303986	0.010	-0.3	50	100
47 1,1-Dichloropropene	0.312453	0.0		0.339881	0.010	8.8	50	109
$ 48 1,2-Dichloroethane-d4 (	0.254982	0.0		0.275565	0.010	8.1	50	108
49 Benzene	0.962627	0.0		0.931985	0.010	-3.2	50	97
50 Isobutyl alcohol	0.537218	0.0		0.580613	0.001	8.1	50	108
51 1,2-Dichloroethane	0.325719	0.0		0.321457	0.010	-1.3	50	99
54 n-Heptane	0.449898	0.0		0.502600	0.010	11.7	50	112
56 Trichloroethene	0.236355	0.0		0.220720	0.010	-6.6	50	93
58 Methylcyclohexane	0.414268	0.0		0.429683	0.010	3.7	50	104
60 1,2-Dichloropropane	0.249767	0.0		0.227525	0.010	-8.9	20	91
62 Dibromomethane	0.123756	0.0		0.110395	0.010	-10.8	50	89
63 1,4-Dioxane	1000.0	0.0	781.5	1.217768	0.001	-21.8	50	78
65 Dichlorobromomethane	0.297656	0.0		0.275235	0.010	-7.5	50	92
67 2-Chloroethyl vinyl eth	0.139649	0.0		0.101556	0.010	-27.3	50	73
68 cis-1,3-Dichloropropene	0.487946	0.0		0.492218	0.010	0.9	50	101
69 4-Methyl-2-pentanone (M	0.262519	0.0		0.254785	0.010	-2.9	50	97
$ 70 Toluene-d8 (Surr)	1.183728	0.0		1.375074	0.010	16.2	50	116
71 Toluene	0.849031	0.0		0.874036	0.010	2.9	20	103
72 trans-1,3-Dichloroprope	0.395835	0.0		0.422045	0.010	6.6	50	107
73 Ethyl methacrylate	0.320065	0.0		0.347270	0.010	8.5	50	108
74 1,1,2-Trichloroethane	0.243045	0.0		0.236202	0.010	-2.8	50	97
75 Tetrachloroethene	0.359514	0.0		0.346696	0.010	-3.6	50	96
76 1,3-Dichloropropane	0.449740	0.0		0.446509	0.010	-0.7	50	99
S 77 Xylenes, Total	100.0		105.7			5.7	50	106
78 2-Hexanone	0.180937	0.0		0.181753	0.010	0.5	50	100
79 Chlorodibromomethane	0.274143	0.0		0.255739	0.010	-6.7	50	93
81 Ethylene Dibromide	0.239843	0.0		0.225203	0.010	-6.1	50	94
83 Chlorobenzene	0.922998	0.0		0.880968	0.300	-4.6	50	95
85 1,1,1,2-Tetrachloroetha	0.310363	0.0		0.291338	0.010	-6.1	50	94
86 Ethylbenzene	0.508679	0.0		0.502446	0.010	-1.2	20	99
87 m-Xylene & p-Xylene	1.231254	0.0		1.289386	0.010	4.7	50	105
88 o-Xylene	1.249064	0.0		1.333070	0.010	6.7	50	107

(054-011)

Report Date: 01-Dec-2015 13:24:10 Chrom Revision: 2.2 08-Oct-2015 07:17:48

Preliminary Report

Data File: \\ChromNA\Chicago\ChromData\CMS16\20151120-34653.b\16C1120.D

Compound	Standard RRF/Amt	DLT RT	Ccal Amt	Ccal RF	Min. RRF	%D	Max. %D	%Rec
89 Styrene	1.057941	0.0		1.002864	0.010	-5.2	50	95
90 Bromoform	0.168644	0.0		0.139077	0.100	-17.5	50	82
91 Isopropylbenzene	2.947491	0.0		3.150121	0.010	6.9	50	107
$ 93 4-Bromofluorobenzene (S	0.441149	0.0		0.521416	0.010	18.2	50	118
95 Bromobenzene	0.708008	0.0		0.685054	0.010	-3.2	50	97
96 1,1,2,2-Tetrachloroetha	0.560420	0.0		0.570580	0.300	1.8	50	102
97 1,2,3-Trichloropropane	0.167500	0.0		0.172434	0.010	2.9	50	103
98 trans-1,4-Dichloro-2-bu	0.207168	0.0		0.247971	0.010	19.7	50	120
99 N-Propylbenzene	3.479371	0.0		3.882792	0.010	11.6	50	112
100 2-Chlorotoluene	2.018857	0.0		2.228897	0.010	10.4	50	110
101 4-Chlorotoluene	2.406577	0.0		2.649009	0.010	10.1	50	110
102 1,3,5-Trimethylbenzene	2.508045	0.0		2.684174	0.010	7.0	50	107
103 tert-Butylbenzene	2.076291	0.0		2.242577	0.010	8.0	50	108
105 1,2,4-Trimethylbenzene	2.504305	0.0		2.717355	0.010	8.5	50	109
106 sec-Butylbenzene	3.190380	0.0		3.454994	0.010	8.3	50	108
107 1,3-Dichlorobenzene	1.362402	0.0		1.406987	0.010	3.3	50	103
108 4-Isopropyltoluene	2.716254	0.0		2.941298	0.010	8.3	50	108
110 1,4-Dichlorobenzene	1.374594	0.0		1.413669	0.010	2.8	50	103
113 1,2-Dichlorobenzene	1.244516	0.0		1.272554	0.010	2.3	50	102
114 n-Butylbenzene	2.564077	0.0		2.881151	0.010	12.4	50	112
115 1,2-Dibromo-3-Chloropro	0.083403	0.0		0.095986	0.010	15.1	50	115
117 1,2,4-Trichlorobenzene	0.858826	0.0		0.869253	0.010	1.2	50	101
118 Hexachlorobutadiene	50.0	0.0	54.9	0.536630	0.010	9.8	50	110
119 Naphthalene	1.563887	0.0		1.671750	0.010	6.9	50	107
120 1,2,3-Trichlorobenzene	0.748925	0.0		0.749463	0.010	0.07	50	100

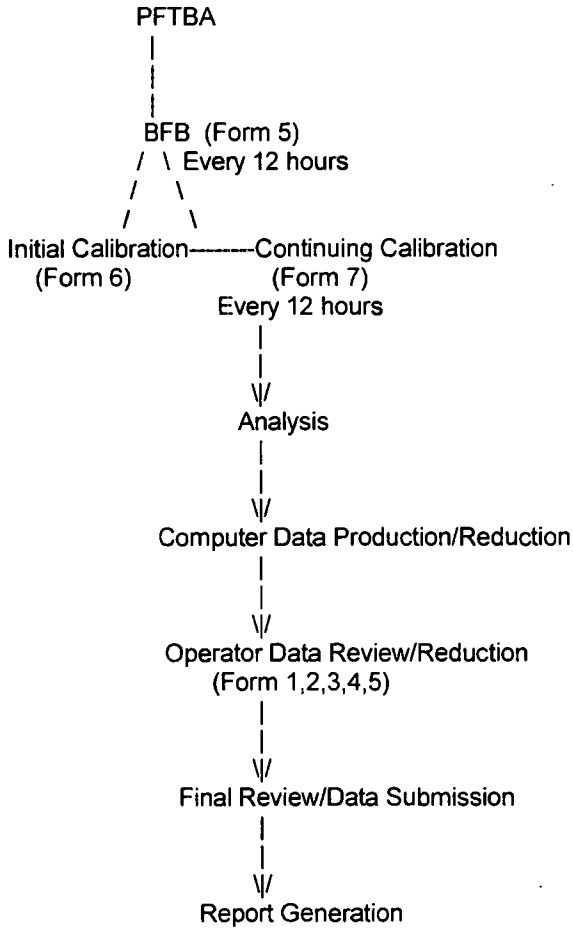
(054-012)

Attachment 5.

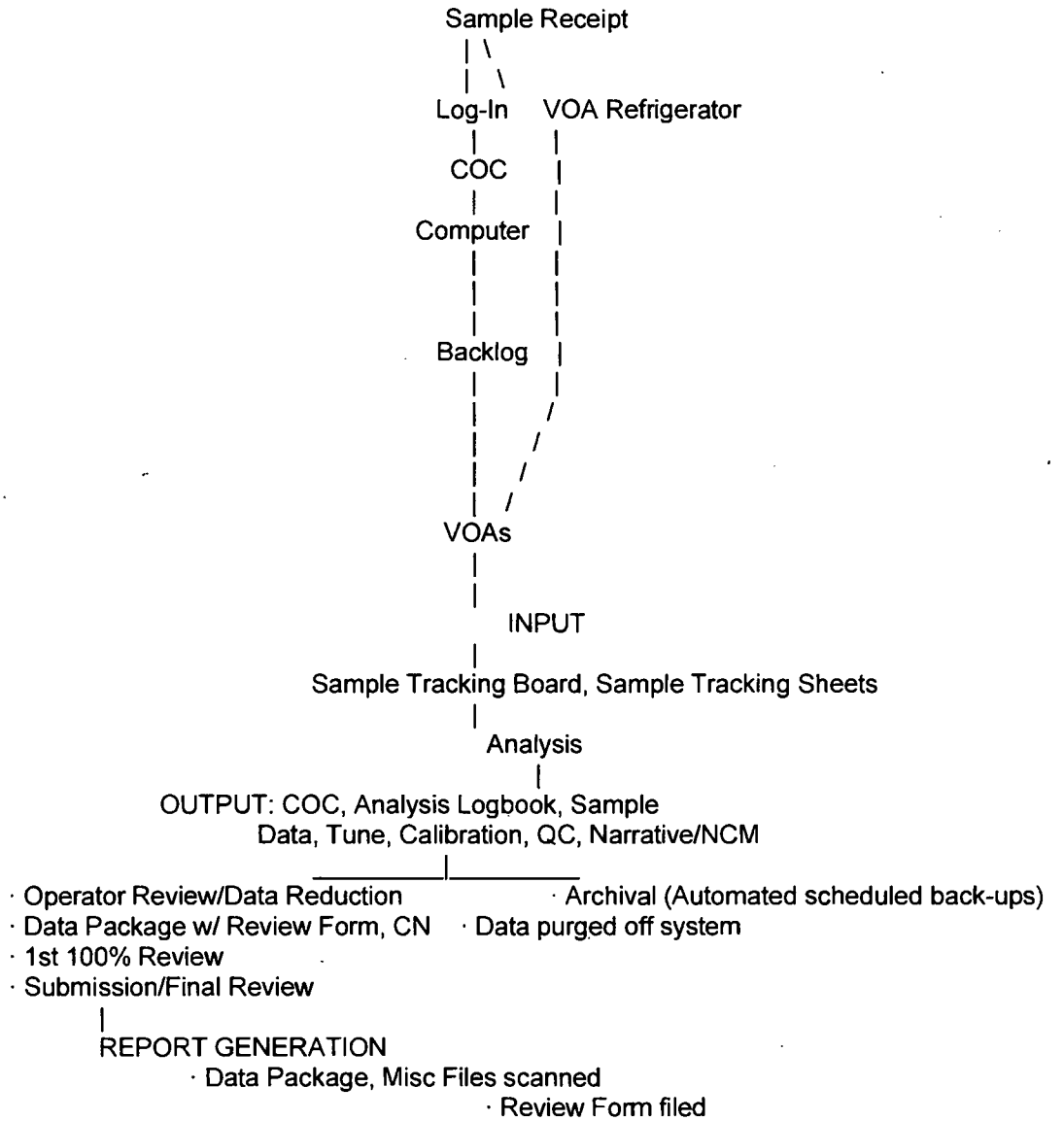
Example: Analysis and Sample Tracking Flowcharts

Analysis Scheme Flowchart

(Terms defined in the Section 9)



Sample Tracking Flowchart



Attachment 6.

Example: Data Review Checklist (058-001 to 058-004)

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Chicago

GCMS Volatile ICAL Data Review Checklist

Instrument: **Date:**	**Worklist #:**	**LIMS Batch Number:** Water _____ MeOH _____ 624 _____
Analyst/1st Reviewer: **Date:**	**QC Type:** Standard ICAL – 8260B / 624	**Analytical Method / Matrix (check)** ___ 8260B/624: Water/MeOH (5-ml purge) ___ 8260B Soil_Low Level (5 ml purge)
2nd Reviewer: **Date:**		

Review Items	NA	Yes	No	2nd Rev	If No, why is data reportable?
A. Tune / Calibration					
1. Did BFB meet tune criteria?					
2. Were all standards injected within 12 hours of the BFB? (or 24 hours for 624?)					
3. ICAL date and instrument ID verified?		X			
4. Were ≥ 5 levels of each compound / surrogate analyzed?					
5. Was low level standard at or below RL?					
6. If calibration points removed, were reasons for removal documented? Did sufficient calibration points remain? (removal from middle of curve not allowed)					(e.g.; some points <RL removed)
7. Do the average RFs meet minimum RF requirements? (624 – not method defined) (8260B-SPCCs = Chloromethane, 1,1-Dichloroethane, Bromoform ≥0.1; Chlorobenzene, 1,1,2,2-Tetrachloroethane ≥0.3) (8260C- all cmpds have min RFs defined in method/SOP)					
8. Did the calibration %RSD meet method requirements? (624: Method Table limits and ≤35% all other cmpds) (8260B: ≤30% for CCCs & ≤15% for all other cmpds/surrogates) (8260C: ≤20% for all cmpds/surrogates)					
9. Was a linear or quadratic regression fit used for analytes that exceeded the %RSD requirements?					
10. If regression fit used, is correlation coefficient ≥0.990? (Is the best regression fit used: LS1 vs. LS2; QS1 vs. QS2) Review % Error					
11. Does the low point of a linear regression fit meet the ±30% read-back criteria? (8260C required) (8260B, 624-recommended)	X				Analyze a CCVL for WI and SC samples per tune.
12. At least 6 consecutive points used for quadratic curves? (Note: SC and WI – Does not allow the use of quadratic curves without the analysis of a low level verification per tune.)					
13. For quadratic – examine plot: Is a tangent's slope to the curve entirely positive or negative and continuous? (does not flatten or recurve within the range of calibration)					

(058-001)

14. For quadratic – evaluate curve fitting errors: Does each point fall within criteria when 'read-back' against the curve? (TA requirement – CA-Q-S-005);(recommended ±30% read-back criteria) (Chrom Report = Details of Calibration per Analyte)	X				Analyze a CCVL for WI and SC samples per tune.
15. Is the concentration intercept <RL for each cmpd? ("X" intercept in Chrom)					
16. Were manual integrations performed correctly and properly documented? (MI is electronically dated/initialed and reason given in Chrom; 2nd review of all MIs required)					
17. Was the high point checked for detector saturation?					
18. Isomeric pairs checked for elution order/correct peak assignment? • Vinyl Acetate / Isopropyl Ether • 1,2- & 1,3-& 1,4-Dichlorobenzene • Ethylbenzene / Xylenes • 1,3,5- & 1,2,4-Trimethylbenzene / Isopropylbenzene / sec-butylbenzene • 2- & 4-Chlorotoluene / n-Propylbenzene • MIBK / 2-Hexanone • Methyl Methacrylate / Ethyl Methacrylate • 1,1-Dichloroethene / cis-1,2- & trans-1,2-Dichloroethene • 1,2,3- & 1,2,4-Trichlorobenzene • 1,1-Dichloropropene / cis-1,3- & trans-1,3-Dichloropropene / 1,2,3-Trichloropropane • Chlorobenzene-d5 / 1,1,1,2-Tetrachloroethane • Trichlorofluoromethane / Freon 113 • Hexane / Vinyl Acetate (Chrom: View/Documents/Methods/Isomers)					
19. Was the 2nd source initial calibration verification standard (ICV) within required criteria? (624 = Method does not require ICV) (8260B = ±30% all compounds) (8260C = ±30%, poor performers <50%)					ICV TALs Batch: Water _____ MeOH _____
20. If any criteria from items 1-18 were not met, was a NCM generated & approved by supervisor?					
21. All points are in the most recent active calibration event? [Calibration Events –'Fix ICAL linkage' if needed]					
22. Is the ICAL locked in CHROM? Is the ICAL set as most recent? Is the ICAL locked in TALs?					

Comments: _____

(058-002)

TestAmerica Chicago
GCMS Volatiles DATA REVIEW CHECKLIST

Site Name:_____ Primary Reviewer:_____ Review Date:_____

JOB Number: _____ Secondary Reviewer:_____ Review Date:_____

Method: a) 8260B_____ 624 _____ 5030_____ Encores: 5035–High _____ 5035-Low_____ TCLP _____

Matrix: WATER / SOIL / SPLP-TCLP / Other (_____) Report Type: Level 1 2 3 4

TASK	PRI REV	SEC REV	COMMENTS
Inst# Date Worklist Analytical Batch _____ _____ _____ _____ _____			Associated Samples: Calib. ID: _____ _____ _____ _____ _____ _____
Sample Hold Time: _____ NCM Ref Number: _____			Date Analyzed: _____
Data Summaries: Executive Summary Are all flags correctly assigned? Y N Sample Data Sheets present? Y N Surrogate page present? Y N QC Data Summaries all present? Y N QC Associated Summary All samples associated? Y N Are 5035 samples linked to prep batch? Y N NA Are all dilutions appearing on summary? Y N NA			
FORM 1: IF original and re-run are to be reported in LIMS: Appropriate suffixes present Re-Analyzed (RA) Re-Extracted (RE) Dilution (DL) NCM Ref Number: _____			Smp # Original Dilution Comments
FORM 2: Surrogate Recoveries Within Limits Statistical Limits _____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			List sample numbers and surrogates:
FORM 3: MS/MSD Recoveries Acceptable Statistical Limits _____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			Sample _____ _____ MS MS MSD MSD RPD RPD
FORM 3: LCS Recoveries Acceptable (LCD if no MS/MSD) Statistical Limits _____ AFCEE/LCG/DOD/QAPP:_____ NCM Ref Number: _____			Control Compounds: Full / Other Batch # _____ Batch # _____ Batch # _____ Batch # _____

(058-003)

TASK	PRI REV	SEC REV	COMMENTS
Method Blank Detection Limits Met (< 1/2 RL for AFCEE / DoD QSM) NCM Ref Number: _____			
FORM 5: Tuning Criteria Met/Matches LIMS Analysis Batches ICAL Form 5 OK? Yes No Tunes: _____ _____ _____ _____ _____ _____ _____ _____ _____ NCM Ref Number: _____			Tune time met? Yes No
RAW DATA: 1) Raw Data Verified/Complete			
2) Screening Data match analysis data?			
Form 6: Initial Calibration Criteria met and Complete?			
Form 7: CCV criteria met method criteria NCM Ref Number: _____			
ICV (ICAL Spike Required): Yes No Control Limit applied: _____ NCM Ref Number: _____			
QC Raw data present and complete 1) Tune Yes No 2) Blank Yes No 3) LCS/LCSD Yes No 4) MS/MSD Yes No NA MRL Check Required: Yes No Control Limit Applied: _____			
Manual Integration reports (before and afters) present, analyst initials are present and the reasons for the MI are correctly documented and approved?			
Prep Log page Present / Verified			
NCM's reviewed and verified? Yes No			
ICOC Required/Properly Documented Yes No			
Additional Comments:			
Manual Calculation of On Column result: Response Factor (Smp) x Concentration of IS IS Response Factor (Smp) Cmpd. RRF (Cont.Calib)			Sample : _____ Compound: _____

(058-004)

Attachment 7.

CA-Q-P-003: TestAmerica Corporate Policy Calibration Curves and the Selection of Calibration Points (059-001 to 059-021)

Title: Calibration Curves and the Selection of Calibration Points

Approvals (Signature/Date):

Raymond J. Frederici — 2/17/2015	Larry Penfold — 2/17/2015
Raymond J. Frederici — Date	Larry Penfold — Date
Exec. Director of Quality & EHS	Quality Compliance Director

1.0 PURPOSE

1.1 This Policy provides guidance on the various types of calibration models used at TestAmerica Laboratories, the basic formulae and calculations, and guidelines for review of calibration data. This Policy is to be used in conjunction with the laboratories analytical method Standard Operating Procedures (SOPs) to clarify the calculations of the calibration curve and sample concentrations.

1.2 This policy further describes the requirements for the determination of the number of points and removal of points from calibration curves.

1.3 Requirements for continuing calibration verification are described in each laboratory's analytical method SOPs.

2.0 SCOPE

2.1 This policy applies to all multi-level initial calibrations. Procedures stated in this policy regarding the selection and removal of calibration points are required at all TestAmerica facilities.

2.2 The calibration models defined herein may be used at any TestAmerica laboratory. This policy pertains to the basic calculations involved in the various calibration models. There are two basic forms of linear calibrations and one basic form of non-linear calibration models used by TestAmerica. Whether a linear or non-linear model is used is analyte dependent based on the instrument responses for that analyte. In addition, the calculations vary based upon the differences between internal and external calibration techniques.

2.3 Additional variables are whether to "force" the curve through zero or not, and optional use of a "weighting" factor.

2.4 Appendix 1 details specific calculations for unweighted and weighted curves and Relative Standard Error (RSE). Refer to the specific method SOPs on the procedure to calibrate an instrument and verify the calibration.

2.5 If the laboratory chooses to use a calibration model that is not provided for in this procedure, it must be clearly defined in the laboratory's SOP and verified that it meets regulatory requirements where appropriate. This calibration procedure must also be peer reviewed by the Technical Services Director or a Quality Director prior to use.

2.6 For sample calculations, the laboratory must use the most recent initial calibration obtained prior to the analytical batch.

3.0 SAFETY

3.1 There are no specific safety hazards associated with this Policy.

3.2 During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Laboratory Health & Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Data Quality Objectives (DQOs)** are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application.

4.2 **External Standard Calibration** – Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

4.3 **Internal Standard Calibration** – Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

4.4 **Instrument Response** – Instrument response is normally expressed as either peak area or peak height; however, it may also reflect a numerical representation of some type of count on a detector (e.g., photomultiplier tube or diode array detector) and is used in this policy to represent all types.

4.5 **Origin** – The point in a coordinate system where the axes of the system intersect (e.g., x = 0 and y = 0 in a two-dimensional system).

4.6 **Concentration Axis** – The concentration axis may vary by software (e.g.; Target uses the 'y' axis for concentration). All discussion in this policy describes the 'x' as the concentration axis.

5.0 PROCEDURE

5.1 **Calibration Curves** – Extrapolation of the calibration to concentrations or instrument signals above or below those of the actual calibration standards is not appropriate and may lead to significant quantitative errors, regardless of the calibration model chosen. Analytes that exceed the upper calibration level should be diluted and rerun where possible. In the instance where a method has other requirements or there is insufficient sample left for reanalysis, results above the upper calibration level must be qualified to that effect. Analytes below the lower calibration level must be qualified if reported.

NOTE: Some metals analyses use a single point calibration and perform periodic linear range verifications.

Refer to CA-Q-WI-013, Calibration Spreadsheet, for additional validation support.

5.1.1 External Standard Calibration Procedure

5.1.1.1 External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample instrument responses (InstrRsp) are compared to responses of the standards. The ratio of the detector response to the amount (mass or concentration) of analyte in the calibration standard is defined as the **Calibration Factor (CF)**.

5.1.1.2 The advantages of external standard calibration are that it is simple to perform this type of calibration and it can be applied to a wide variety of specific analytical methods. Its primary disadvantage is that it is greatly affected by the stability of the detector system.

(059-003)

$$CF = \frac{InstrRsp}{Concentration}$$

5.1.1.3 A CF is calculated for each standard in the calibration. The average CF is used for quantitation if the %RSD meets the criteria stated in the specified method (e.g., 10%, 20%, etc.). A %RSD within the method criteria assumes that the calibration is linear and goes through zero (the origin).

$$Avg_{CF} = \frac{\sum CF}{n} \quad n = \text{number of CFs}$$

$$\%\ RSD = \frac{SD_{CF}}{Avg_{CF}} \times 100 \quad SD_{CF} = \text{Standard Deviation of CFs}$$

5.1.1.4 To quantify sample concentrations ($C_{(sample)}$) using the Avg CF:

$$C_{(sample)} = \frac{InstrRsp}{Avg_{CF}}$$

5.1.2 Internal Standard Calibration Procedure

5.1.2.1 The advantages of internal standard calibration includes the fact that it automatically adjusts for routine variation in the response of the analytical system, as well as variations in the exact volume of sample or sample extract introduced into the analytical system. In addition to normalizing the response (peak area) of the target compound to the response of the internal standard in that sample or extract for that injection, for chromatographic methods, the retention times of the target compound and the internal standard may be used to calculate the relative retention time (RRT) of the target compound.

5.1.2.2 The RRT evaluation allows the analyst to compensate for modest shifts in the chromatographic conditions that can occur due to interferences and simple day-to-day instrument variability. Many methods that employ internal standard calibration use more than one internal standard, and the target compounds are related to the internal standards on the basis of the similarity of their respective chromatographic retention times.

$$RRT = \frac{(RetentionTime_{analyte})}{(RetentionTime_{IS})}$$

5.1.2.3 Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the instrument response of the target compound in the sample or sample extract to the instrument response of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

NOTE: **Isotope dilution calibration** is essentially a special case of internal standard calibration for select GC/MS applications. In isotope dilution, the internal standards are stable isotopically-labeled analogs of the target analytes *and* they are added to the sample prior to any

sample handling steps, including sample extraction. The calculations are the same as with internal standard calibrations.

$$RF = \frac{(R_S * C_{IS})}{(R_{IS} * C_S)}$$ Where: R_S = Response of Standard

R_{IS} = Response of Internal Standard
C_S = Concentration of Standard
C_{IS} = Concentration of Internal Standard

NOTE: Response factors for GC/MS methods may also be calculated using the sums of the areas of two ions (m/zs) for each target analyte and each internal standard. The SOP for the analytical method must stipulate which is used.

5.1.2.4 A RF is calculated for each standard in the calibration. The average RF is used for quantitation if the % Relative Standard Deviation (RSD) meets the criteria stated in the specified method (e.g., 10%, 20%, etc.). A %RSD within the method criteria assumes that the calibration is linear and goes through zero.

$$\text{Avg RF} = \frac{\sum RF}{n}$$ where n = number of RFs

$$\%RSD = \frac{SD_{RF}}{Avg_{RF}} \times 100$$ where SD = Standard Deviation of RFs.

5.1.2.5 To quantify sample concentrations ($C_{(sample)}$) using the Avg RF:

$$C_{(sample)} = \frac{R_S * C_{IS}}{Avg_{RF} * R_{IS}}$$

5.1.3 Linear Calibrations using a Least Squares Regression

The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or response ratio) of a standard or sample and the x axis represents the concentration. Either way, the regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. As a general rule, in order to be used for quantitative purposes, $r \geq 0.990$ for organics and $r \geq 0.995$ for inorganics. Any exceptions to this must be technically sound, well documented and compliant with method or regulatory requirements.

5.1.4 External Standard Calibration

In most cases, the instrument software performs the calibration and calculates the results. For hand calculations, each standard is entered into a calculator or an Excel spreadsheet (refer to CA-Q-WI-013 on the Oasis QA webpage - Calibration) using the instrument response as the 'y' coordinate and the concentration as the 'x' coordinate. The origin is not included in the curve for organics and no zero point is used. For some inorganic methods, a zero concentration point is

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used but the curve is not forced through the origin. The regression function will result in the following equation as well as the correlation coefficient (r). If "r" is acceptable, then the curve may be used.

$y = mx + b$ Where: y = peak response
x = concentration
m = slope of the line
b = y intercept

To calculate sample results (C_{sample}) from the above equation, the following equation is used.

$$C_{(sample)} = \frac{y - b}{m}$$

5.1.5 Internal Standard Calibration

In most cases the instrument software performs the calibration and calculates the results, but each standard may be entered into a calculator or an Excel spreadsheet (refer to CA-Q-WI-013 on the Oasis QA webpage - Calibration) using the Response Ratio as the 'y' coordinate and the concentration as the 'x' coordinate. The origin is not included in the curve for organics and no zero point is used. For some inorganic methods, a zero concentration point is used but the curve is not forced through the origin. The regression function will result in the following equation as well as the correlation coefficient (r). If "r" is acceptable, then the curve may be used.

5.1.5.1 Resp. Ratio = $\frac{R_S * C_{IS}}{R_{IS}}$ Where: R_S = Response of Sample or Standard
C_{IS} = Concentration of Internal Standard
R_{IS} = Response of Internal Standard

5.1.5.2 $y = mx + b$ Where: y = Resp. Ratio
x = concentration
m = slope of the line
b = y intercept

5.1.5.3 To calculate sample results (C_{sample}) from the above equation the following equation is used.

$$C_{(sample)} = \frac{y - b}{m}$$

5.1.6 Non-Linear Calibrations Curves

5.1.6.1 Non-Linear polynomial curves may also be performed based on the analyte response on a given column. Some methods recognize the fact that not all analytes respond in a linear fashion. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fits may be indicated. Another term for this type of curve is a quadratic or 2nd order curve. There are limitations on the use of quadratic curves:

5.1.6.2 They may not be used to mask instrument problems that can be corrected by maintenance.

5.1.6.3 They may not be used to compensate for detector saturation.

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5.1.6.4 If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1st order fit or average RF.

5.1.6.5 TestAmerica does not utilize 3rd order (a.k.a., cubic fit) curves.

5.1.6.6 The 2nd order curves are a mathematical calculation of a curved line over two axis. The 'y' axis represents the instrument response (or Response ratio) of a standard or sample and the 'x' axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature of the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, $r^2 \geq 0.990$.

5.1.6.7 2nd order or quadratic curves should only be used in circumstances where a linear curve is clearly not appropriate.

5.1.6.8 Use of 2nd order curves requires the additional data review evaluations described in Section 5.1.11.9.

5.1.7 External Standard Calibrations

Sample concentration (x) is calculated from the following quadratic equations: $y = ax^2 + bx + c$

The equation above is further reduced to:

5.1.7.1 $0 = ax^2 + bx + (c - y)$ then

5.1.7.2 $0 = x^2 + bx/a + (c-y)/a$

Where:
y = analyte response
x = analyte concentration
a, b, and c are values from the polynomial curve equation
a = generally defines the curvature
b = is representative of the slope
c = the y intercept

5.1.7.3 Finally, sample concentration (x) is determined by:

$$x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}$$

Where: a = 1

b = b/a in 5.7.2
c = (c-y)/a in 5.7.2

The instrument software uses the basic quadratic formulae. Results can be verified manually by entering the original y, a, b, and c values in the Excel spreadsheet created for quadratic curves. X will be calculated based on the above equations.

5.1.8 Internal Standard Calibrations – The basic equations remain the same as it is for the External Calibration; however, the "y" coordinate is based on the Response ratio.

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5.1.9 Forcing Through Zero

5.1.9.1 Forcing through zero was not permitted by SW846 Method 8000B, but was recognized as valid and desirable in some cases in SW846 Method 8000C and 8000D. Though this Policy allows the use of forcing through zero for most methods, ***caution*** should be used if referencing Method 8000B when using a curve that is forced through zero. SW846 has professed to be a "guidance document"; however, there are regulators that take strict positions to the QC requirements to a referenced method and may not allow the practice when referencing Method 8000B.

5.1.9.2 ***Forcing through zero:*** Do not include the origin (0,0) as an extra calibration point unless required by the method. However, most data systems and many commercial software packages will allow the analyst to "force" the regression through zero. Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 ***before*** the regression is calculated, thereby setting the bias to favor the low end of the calibration range by "pivoting" the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression though zero for some calibrations

NOTE: The use of a linear regression or forcing the regression through zero may **NOT** be used as a rationale for reporting unqualified results below the calibration range demonstrated by the analysis of the standards. If it is necessary to report unqualified results at lower concentrations, then the analyst should run a calibration that reaches those lower concentrations.

5.1.10 Curve Weighting – A weighted curve is often recommended; unweighted regressions are likely to have high relative errors at the lower concentration levels and are strongly discouraged. Weighted regressions should be used wherever possible.

5.1.10.1 From Method 8000C: "Weighting may significantly improve the ability of the regression to fit the linear model to the data."

5.1.10.2 Weighted curves follow the same basic model as the unweighted curves; however, to improve linearity at the low of the calibration curve, the square of the residuals in the calculation (refer to Attachment 1) is based on $1/x$ or $1/x^2$ rather than x (where x is the concentration reading). When applying weighted regressions, the model ($1/x$ or $1/x^2$) that best minimizes the fitting errors (i.e., magnitude of the residuals) should be used.

5.1.10.3 A concentration at the origin (blank standard) cannot be included when an inversely weighted fit is used for calibration, the mathematics will produce an infinite, incalculable result.

5.1.11 Basic Elements of Calibration Review

5.1.11.1 All laboratories are required to use a data review checklist to document first and second level review of initial calibrations.

5.1.11.2 It is normally preferable to use the simplest mathematical algorithm that represents the relationship within acceptable limits of error.

5.1.11.3 Instrument signals for all samples must be no greater than the signal for the highest calibration standard.

5.1.11.4 Low Calibration Point

- The lowest calibration level must be at or below the RL, with the exception of single-point ICP calibrations that include an RL check standard. If this requirement is not met, the instrument must be recalibrated to include a lower concentration standard.
- The lowest calibration level must be higher than the MDL.
- A read-back of the low calibration point through the calibration equation should fall within the method defined limit or the expected value, or within +/-50% if no limit is stated in the method.

5.1.11.5 Minimum Number of Calibration Points – SOP/method requirements must be met. If this requirement is not met, the problem needs to be investigated and fixed, and the instrument recalibrated.

5.1.11.6 Linearity – SOP/method requirements must be met. If this requirement is not met, the problem needs to be investigated and fixed, and the instrument recalibrated.

5.1.11.7 X-intercept (concentration intercept) – Absolute value should be < RL, preferably < ½ reporting limit (RL), when reporting to the RL. An exception can be made for common lab contaminants with small residuals (fitting errors) at the lowest calibration point.

5.1.11.8 Increasing Response with Increasing Concentration – The instrument signal should increase with every increase in standard concentration. The opposite is true for instruments that produce a signal that is inversely proportional to concentration, such as ion selective electrode.

5.1.11.9 Additional Checks Required for 2nd Order Curves

- Curve fitting errors must be evaluated to confirm that the quadratic is the most accurate fit to the data. This is most commonly done by "reading back" each of the calibration standard points through the calibration equation.
- The calibration plot must be inspected to ensure that that the curve does not flatten out (i.e., slope = 0) at a level below the highest calibration standard.
- The calibration plot must be visually inspected to be certain that the function does not recurve (i.e., change from a positive slope to a negative slope or vice versa) within the range of calibration standard concentrations.

5.2 Selection of Calibration Points

5.2.1 If the number of data points required for an initial calibration is defined in the method, Quality Assurance (QA) plan, published report, or previously approved SOP, then that requirement will be used for the defined procedure.

5.2.2 In the cases not defined in Section 5.2.1, the number of data points will be determined by the technical manager based on the DQOs for precision and accuracy to be met by the method.

5.2.2.1 When used for regulatory purposes, the minimum number of calibration points determined by the technical manager shall be three (3), except in cases where reference methods using similar technology use a single point and blank (ICP and ICP/MS primarily), or where the need is to demonstrate that the result is above or below a specific concentration limit.

5.2.2.2 Non-detects may use a single point at the reporting level.

Examples:

A. Required to analyze a new pesticide in water and a published method does not exist. The data will be used to screen samples by HPLC-UV at a waste site for further remediation, using DQOs that require precision/accuracy of ± 50%.

The technical manager selects 2 data points to represent the range of the expected concentration of pesticide and based on 4 Laboratory Control Samples (LCS), the recoveries ranged from 78-104%. Therefore, 2 data points are sufficient for initial calibration for this method.

Note: Calibration curves with less than 3 points should only be used after discussion with the client that the DQOs will be met. There must be indication in the final report to the client to reflect the calibration and/or analysis limitations.

B. Same compound as above but being measured in the laboratory for meeting a regulatory limit of 0.05 mg/L in water. Precision and accuracy of ± 20% required.

A 5-point calibration is used, based on similar requirements in published methods with similar objectives and the high level of precision and accuracy required.

As noted above for methods where the technical manager selects the number of data points to meet DQOs for precision and accuracy, the 4 LCS's used in the demonstration of capability will be used to assure those DQOs are met. The SOP will then be approved by the QA Manager.

5.2.3 Removal of Points from a Calibration Curve

5.2.3.1 Removal or replacement of points from the middle of a calibration (i.e., levels other than the highest or lowest) is not permitted unless an injection or instrument problem confined to that point can be clearly documented as described below. The failed standard must be re-run within 24-hours and before any samples and inserted into the initial calibration. If not useful, recalibration is required. Removal of points for individual analytes from levels other than the highest and lowest is not permitted in any event.

5.2.3.2 If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run (refer to Sec. 5.2.3.3), the level may be excluded if the curve still has sufficient levels, or the run may be repeated once only. The whole level (all compounds) must be removed or replaced. The curve is evaluated with the level removed or replaced. If the curve still fails to meet criteria, then corrective action must be taken and the whole curve reanalyzed. Corrective action may include, but is not limited to, instrument maintenance and/or re-preparation of standards.

5.2.3.3 One of the following conditions must be satisfied to allow removal or replacement of a level:

- The data file is corrupted and unusable or the run is interrupted before completion.
- The analyst observes and documents a problem such as leaking of a purge vessel.
- For internal standard methods, the recovery of the internal standard is less than 70% or greater than 130% of the recovery in the other standards (all internal standards show the same bias in the standard in question), or the amount of analyte recovered is less than 70% or greater than 130% of the expected values; indicating an improperly made up standard (all analytes in a spike mix must show the bias).

(059-010)

- For external standard methods, the unit response of the analyte is less than 70% or greater than 130% of the average unit response for the analyte in the other calibration standards; indicating an improperly prepared standard or bad injection. (All analytes in spike mix must show the bias to demonstrate a standard is bad, all analytes in calibration standard must show bias to demonstrate bad injection.)

5.2.3.4 When using autosamplers with discrete sample pathways for different samples (such as 16 port purge and trap autosamplers), the level to be replaced must be reanalyzed on the same port or that port must be excluded from sample analysis until corrective action is performed and verified by successful analysis of a continuing calibration standard on that port.

5.2.3.5 The reason for replacing the level **must** be documented in the run log. The fact that the curve passes criteria with the level removed is **not** alone sufficient evidence to document an injection or instrument problem confined to the level.

5.2.3.6 Removal of the highest or lowest levels is permitted, but the calibration range must be adjusted accordingly. If the lowest level is removed then the reporting limit is raised to be equivalent to the lowest level used in the calibration curve. In any event the number of levels remaining in the calibration must be at least that required by the method.

5.2.3.7 Removal of the highest or lowest point is permitted on a compound specific basis. This may be necessary when strongly responding and poorly responding analytes are included in the same standard mix at the same level. Each compound must have at least the minimum number of calibration levels required by the method.

6.0 RESPONSIBILITIES

6.1 **Supervisors** are responsible for the initial and annual training of all analysts performing linear or non-linear calibration on the procedures defined in this Policy; as well as the Linear Regression spreadsheet (CA-Q-WI-013).

NOTE: An exception to the training is the study of the calculations.

6.2 **All Analysts** are to attend a training session on this Policy as conducted by his/her supervisor on the topics/forms defined in Section 5.1. All analysts utilizing methods involving multi-point calibrations are required to follow this policy (Sec. 5.2).

6.3 **Quality Assurance Manager** ensures that each analyst performing linear or non-linear regressions is trained by their supervisor on this Policy.

7.0 REFERENCES/CROSS-REFERENCES

7.1 EPA Method 8000B, EPA SW-846 Update III, December 1996.

7.2 EPA Method 8000C, EPA SW-846 Update III, March 2003.

7.3 EPA May 2012 Method Update Rule – discussion of RSE

7.4 Calibration Spreadsheet, Form No. CA-Q-WI-013.

(059-011)

8.0 ATTACHMENTS

Attachment 1: Detailed Calculations (Informational Only).

9.0 REVISION HISTORY

- Revision 0, dated 13 February 2015
  - Initial Release – Combination of CA-Q-S-005, Calibration Curves; and CA-T-P-002, Selection of Calibration Points.
  - The derivation of equations for linear regressions with 1/X weighting added to Attachment 1.

(059-012)

Attachment 1.

Detailed Calculations

The linear regression formulas on the following pages are derived using the method of least squares. This method is applied to determine the mathematical relationship between two variables, using data from the analysis of known standards, thus allowing the value of one variable to be predicted from a second measured variable.

In these examples the known standard concentrations are assumed to represent the independent variable (*x*) with the measured response as the dependent variable (*y*). The integer value symbolized by *n* represents the number of points in the calibration. A basic knowledge of calculus and matrix solutions is required to understand these derivations.

The intent of this procedure is to define a relationship that minimizes the sum of the squared deviations between all measured and predicted values in the calibration. Taking the derivative of this sum relative to each of the coefficients in the selected calibration equation produces a set of equations, which can be solved for the formulas defining the values of the coefficients.

Included are the derivations for the three most common curve types:

Single factor	$y = Cx$
Linear	$y = C_1 x + C_0$
Quadratic	$y = C_2 x^2 + C_1 x + C_0$

Where, C, C1, C0, C2 are constants

The curve equations represent the best fit relationship between the response and amount for the calibration data. This equation will normally be used to calculate sample amounts (*x*) from measured response (*y*). In the case of the quadratic curve type, the quadratic formula is used

$$x = \frac{\sqrt{C_1^{\,2} + 4C_2(y - C_0)} - C_1}{2C_2}$$

In this case the analyst must ensure that only the single valid root is reported.

In practical applications the calculations for Linear or higher order curves should be performed using validated spreadsheets or other computer applications due the complexity of the formulas.

Coefficients for internal standard calibration may be calculated using the same equations by substituting relative response for *y*:

$$y_R = y_{analyte}\left[x_{is} / y_{is} \right] \text{ (Relative Response)}$$

x_{is} = Internal standard amount
y_{is} = Internal standard response

(059013)

Single Factor (Weighting = 1)

The sum of the squares is defined as

$$S^2 = \sum_{i=1}^{n} (Cx_i - y_i)^2$$

Expanding

$$S^2 = C^2 \sum x_i^2 - 2C \sum x_i y_i + \sum y_i^2$$

Then take the derivative

$$\frac{\partial S^2}{\partial C} = C \sum x_i^2 - \sum x_i y_i$$

Setting this equal to zero gives the regression equation

$$\sum x_i y_i = C \sum x_i^2$$

The Single Factor Coefficient is then calculated as follows:

$$C = \frac{\sum x_i y_i}{\sum x_i^2}$$

Instrument Calibration

Linear (Weighting = 1/x)

The weighted sum of squares is

$$S^2 = \sum \frac{1}{x}(C_1 x + C_0 - y)^2$$

The regression equations are:

$$\sum y = C_1 \sum x + nC_0$$

$$\sum (y/x) = nC_1 + C_0 \sum (1/x)$$

Replacing the coefficients of C_1 & C_0 respectively by the elements to the left of the equal sign gives the matrices below. The Determinants are solved to give ***R***, R_0 & R_1.

$$R = \begin{vmatrix} \sum x & n \\ n & \sum (1/x) \end{vmatrix} \qquad R_0 = \begin{vmatrix} \sum x & \sum y \\ n & \sum (y/x) \end{vmatrix} \qquad R_1 = \begin{vmatrix} \sum y & n \\ \sum (y/x) & \sum (1/x) \end{vmatrix}$$

Note: The determinant of a 2x2 matrix can be calculated as $(x_{1,2}\, x_{2,1} - x_{1,1}\, x_{2,2})$

The Regression coefficients are calculated as follows:

$$C_1 = R_1/R = \frac{n\sum \frac{y}{x} - \left(\sum y\right)\left(\sum \frac{1}{x}\right)}{n^2 - \left(\sum x\right)\left(\sum \frac{1}{x}\right)}$$

$$C_0 = R_0/R = \frac{n\sum \frac{y}{x} - \left(\sum y\right)\left(\sum \frac{1}{x}\right)}{n^2 - \left(\sum x\right)\left(\sum \frac{1}{x}\right)}$$

(059-015)

Single Factor (Weighting = $1/x^2$)

The weighted sum of squares is

$$S^2 = \sum_{i=1}^{n} \left(\frac{Cx_i - y_i}{x_i} \right)^2$$

Expanding

$$S^2 = nC^2 - 2C\sum \frac{y_i}{x_i} + \sum \frac{y_i^2}{x_i^2}$$

Take the derivative with respect to C

$$\frac{\partial S^2}{\partial C} = 2nC - 2\sum \frac{y_i}{x_i}$$

set it equal to zero to get the regression equation

$$\sum \frac{y_i}{x_i} = nC$$

The Weighted Single Factor Coefficient is then calculated as follows:

$$C = \frac{1}{n}\sum \frac{y_i}{x_i}$$

This Calibration is more commonly called Average.

Factors for each calibration level are calculated as:

$$C_i = \frac{y_i}{x_i}$$

The average coefficient is calculated as the average of C_i:

$$C = \frac{1}{n}\sum_{i=1}^{n} \frac{y_i}{x_i}$$

(059-016)

Linear (Weighting = 1)

The sum of the squares is defined as

$$S^2 = \sum (C_1 x + C_0 - y)^2$$

The regression equations are:

$$\sum y = C_1 \sum x + nC_0$$

$$\sum xy = C_1 \sum x^2 + C_0 \sum x$$

Replacing the coefficients of C_1 & C_0 respectively by the elements to the left of the equal sign gives the matrices below. The Determinants are solved to give ***R***, R_0 & R_1.

$$R = \begin{vmatrix} \sum x & n \\ \sum x^2 & \sum x \end{vmatrix} \qquad R_1 = \begin{vmatrix} \sum y & n \\ \sum xy & \sum x \end{vmatrix} \qquad R_0 = \begin{vmatrix} \sum x & \sum y \\ \sum x^2 & \sum xy \end{vmatrix}$$

The Regression coefficients are calculated as follows:

$$C_1 = R_1/R \qquad C_0 = R_0/R$$

Note: The determinant of a 2x2 matrix is calculated as ($x_{1,2}\ x_{2,1}$ - $x_{1,1}\ x_{2,2}$) giving the following coefficients:

$$C_1 = \frac{n\sum xy - \left(\sum x\right)\left(\sum y\right)}{n\sum x^2 - \left(\sum x\right)^2}$$

$$C_0 = \frac{\sum y \sum x^2 - \sum xy \sum x}{n\sum x^2 - \left(\sum x\right)^2}$$

Quadratic (weighting = $1/x^2$)

The weighted sum of squares is

$$S^2 = \sum\left(\frac{C_2x^2 + C_1x + C_0 - y}{x}\right)^2$$

The regression equations are:

$$\sum\left(y/x^2\right) = nC_2 + C_1\sum\left(1/x\right) + C_0\sum\left(1/x^2\right)$$

$$\sum\left(y/x\right) = C_2\sum x + nC_1 + C_0\sum\left(1/x\right)$$

$$\sum y = C_2\sum x^2 + C_1\sum x + nC_0$$

Replacing the coefficients of C_0, C_1 & C_2 respectively by the elements to the left of the equal sign gives the matrices below. The Determinants are solved to give $\boldsymbol{R}$, $\boldsymbol{R}_0$, $\boldsymbol{R}_1$ & $\boldsymbol{R}_2$.

$$R = \begin{vmatrix} n & \Sigma 1/x & \Sigma 1/x^2 \\ \Sigma x & n & \Sigma 1/x \\ \Sigma x^2 & \Sigma x & n \end{vmatrix} \qquad R_2 = \begin{vmatrix} \Sigma y/x^2 & \Sigma 1/x & \Sigma 1/x^2 \\ \Sigma y/x & n & \Sigma 1/x \\ \Sigma y & \Sigma x & n \end{vmatrix}$$

$$R_1 = \begin{vmatrix} n & \Sigma y/x^2 & \Sigma 1/x^2 \\ \Sigma x & \Sigma y/x & \Sigma 1/x \\ \Sigma x^2 & \Sigma y & n \end{vmatrix} \qquad R_0 = \begin{vmatrix} n & \Sigma 1/x & \Sigma y/x^2 \\ \Sigma x & n & \Sigma y/x \\ \Sigma x^2 & \Sigma x & \Sigma y \end{vmatrix}$$

The Regression coefficients are calculated as follows:

$C_2 = \boldsymbol{R}_2/\boldsymbol{R}$ $\qquad$ $C_1 = \boldsymbol{R}_1/\boldsymbol{R}$ $\qquad$ $C_0 = \boldsymbol{R}_0/\boldsymbol{R}$

Note: The determinant of a 3x3 matrix can be calculated by the formula

$$x_{1,1}\,(x_{2,2}\,x_{3,3} - x_{3,2}\,x_{2,3}) + x_{2,1}\,(x_{3,2}\,x_{1,3} - x_{1,2}\,x_{3,3}) + x_{3,1}\,(x_{1,2}\,x_{2,3} - x_{2,2}\,x_{1,3})$$

(059-018)

Quadratic (weighting = 1)

The sum of the squares is defined as

$$S^2 = \sum \left(C_2 x^2 + C_1 x + C_0 - y\right)^2$$

The regression equations are:

$$\sum y = C_2 \sum x^2 + C_1 \sum x + nC_0$$

$$\sum xy = C_2 \sum x^3 + C_1 \sum x^2 + C_0 \sum x$$

$$\sum x^2 y = C_2 \sum x^4 + C_1 \sum x^3 + C_0 \sum x^2$$

Replacing the coefficients of C_0, C_1 & C_2 respectively by the elements to the left of the equal sign gives the matrices below. The Determinants are solved to give $\boldsymbol{R}$, $\boldsymbol{R}_0$, $\boldsymbol{R}_1$ & $\boldsymbol{R}_2$.

$$R = \begin{vmatrix} \Sigma x^2 & \Sigma x & n \\ \Sigma x^3 & \Sigma x^2 & \Sigma x \\ \Sigma x^4 & \Sigma x^3 & \Sigma x^2 \end{vmatrix} \qquad R_2 = \begin{vmatrix} \Sigma y & \Sigma x & n \\ \Sigma xy & \Sigma x^2 & \Sigma x \\ \Sigma x^2 y & \Sigma x^3 & \Sigma x^2 \end{vmatrix}$$

$$R_1 = \begin{vmatrix} \Sigma x^2 & \Sigma y & n \\ \Sigma x^3 & \Sigma xy & \Sigma x \\ \Sigma x^4 & \Sigma x^2 y & \Sigma x^2 \end{vmatrix} \qquad R_0 = \begin{vmatrix} \Sigma x^2 & \Sigma x & \Sigma y \\ \Sigma x^3 & \Sigma x^2 & \Sigma xy \\ \Sigma x^4 & \Sigma x^3 & \Sigma x^2 y \end{vmatrix}$$

The Regression coefficients are calculated as follows:

$C_2 = \boldsymbol{R}_2/\boldsymbol{R}$ $\qquad$ $C_1 = \boldsymbol{R}_1/\boldsymbol{R}$ $\qquad$ $C_0 = \boldsymbol{R}_0/\boldsymbol{R}$

Note: The determinant of a 3x3 matrix can be calculated by the formula

$$x_{1,1}\,(x_{2,2}\,x_{3,3} - x_{3,2}\,x_{2,3}) + x_{2,1}\,(x_{3,2}\,x_{1,3} - x_{1,2}\,x_{3,3}) + x_{3,1}\,(x_{1,2}\,x_{2,3} - x_{2,2}\,x_{1,3})$$

(059-019)

Quadratic (weighting = $1/x^2$)

The weighted sum of squares is

$$S^2 = \sum\left(\frac{C_2x^2 + C_1x + C_0 - y}{x}\right)^2$$

The regression equations are:

$$\sum(y/x^2) = nC_2 + C_1\sum(1/x) + C_0\sum(1/x^2)$$

$$\sum(y/x) = C_2\sum x + nC_1 + C_0\sum(1/x)$$

$$\sum y = C_2\sum x^2 + C_1\sum x + nC_0$$

Replacing the coefficients of C_0, C_1 & C_2 respectively by the elements to the left of the equal sign gives the matrices below. The Determinants are solved to give ***R***, R_0, R_1 & R_2.

$$R = \begin{vmatrix} n & \Sigma 1/x & \Sigma 1/x^2 \\ \Sigma x & n & \Sigma 1/x \\ \Sigma x^2 & \Sigma x & n \end{vmatrix} \qquad R_2 = \begin{vmatrix} \Sigma y/x^2 & \Sigma 1/x & \Sigma 1/x^2 \\ \Sigma y/x & n & \Sigma 1/x \\ \Sigma y & \Sigma x & n \end{vmatrix}$$

$$R_1 = \begin{vmatrix} n & \Sigma y/x^2 & \Sigma 1/x^2 \\ \Sigma x & \Sigma y/x & \Sigma 1/x \\ \Sigma x^2 & \Sigma y & n \end{vmatrix} \qquad R_0 = \begin{vmatrix} n & \Sigma 1/x & \Sigma y/x^2 \\ \Sigma x & n & \Sigma y/x \\ \Sigma x^2 & \Sigma x & \Sigma y \end{vmatrix}$$

The Regression coefficients are calculated as follows:

$C_2 = R_2/R$ $\qquad$ $C_1 = R_1/R$ $\qquad$ $C_0 = R_0/R$

Note: The determinant of a 3x3 matrix can be calculated by the formula

$$x_{1,1}\,(x_{2,2}\,x_{3,3} - x_{3,2}\,x_{2,3}) + x_{2,1}\,(x_{3,2}\,x_{1,3} - x_{1,2}\,x_{3,3}) + x_{3,1}\,(x_{1,2}\,x_{2,3} - x_{2,2}\,x_{1,3})$$

(059-020)

Calibration Error Estimates

Standard deviation (Average)

$$s = \sqrt{\frac{\sum(x_i - \bar{x})^2}{(n-1)}}$$

The *standard deviation* is a measure of the absolute variation of the ***x*** values about the mean of the ***x*** values.

The **relative *standard error (RSE)*** is a relative measure of the variation of the x values about the predicted value of ***x***.

$$\%\,RSE = 100 \times \sqrt{\sum_{i=1}^{n}\left[\frac{x_i' - x_i}{x_i}\right]^2 / (n-p)}$$

Correlation Coefficient

$$r = \sqrt{\frac{\left[\sum xy - \left(\sum x \sum y\right)/n\right]^2}{\left[\sum x^2 - \left(\sum x\right)^2/n\right]\left[\sum y^2 - \left(\sum y\right)^2/n\right]}}$$

The *correlation coefficient* is a measure of the degree of relationship present between two variables.

y	=	Dependent variable (true)
$\hat{y}$	=	Calculated value
$n-P$	=	Degrees of freedom
n	=	number of data points
P	=	1 for single factor
	=	2 for linear
	=	3 for quadratic

(059-021)

Attachment 8.

CA-Q-S-002: TestAmerica Corporate SOP Acceptable Manual Integration Practices (060-001 to 060-015)

Title: Acceptable Manual Integration Practices

Approvals (Signature/Date):

Raymond J. Frederici 10/1/2014
Raymond J. Frederici Date
Corporate Quality Director

Larry Penfold 10/1/2014
Larry Penfold Date
Quality Compliance Director

Verl D. Preston 10/2/2014
Verl D. Preston Date
Quality Systems Director

1.0 PURPOSE

This Standard Operating Procedure (SOP) defines TestAmerica Laboratories, Inc. procedure for proper manual integration and the required documentation and reviews performed during the course of analyses. Willful failure to follow this procedure shall result in disciplinary action, up to and including termination.

2.0 SCOPE

This SOP applies to all analysts & data reviewers within laboratories that perform analytical procedures involving identification or quantitation based on peak analysis (e.g., GC, GC/MS, HPLC, IC, and alpha or gamma spectroscopy). Each laboratory may have locally controlled supplemental procedures or have an addendum to this SOP that describes additional details necessary to implement the processes defined herein. Any supplemental procedure or addendum must adhere to the requirements set forth in this SOP.

Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, on the down side, the technique could also be improperly used to make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, possible laboratory decertification, and potential legal consequences.

3.0 SAFETY

There are no specific safety hazards associated with this SOP.

During the course of performing this procedure it may be necessary to go into laboratory areas to consult with appropriate staff members, therefore employees performing this procedure must be familiar with the Corporate Safety Plan, and take appropriate precautions and wear appropriate attire and safety glasses.

4.0 DEFINITIONS

4.1 **Integration:** The determination of the area or height under a curve (peak).

4.2 **Manual integrations:** Any manual changes to automated peak integration settings. This can include changes to integration start times, integration stop times, baseline changes, on-the-fly changes to retention time (RT) windows assigned to target analytes, manual peak height or area measurements, or changes to automated mass spectrometer tuning algorithms. This does not include re-centering of RT windows following routine instrument maintenance.

4.3 **Chromatograms:** In the context of this procedure, chromatograms are not necessarily limited to the output of chromatography instruments or automated data systems. They can include strip charts, integrator printouts, computer screen dumps, or any graphic display of a continuous signal from an analytical detector.

4.4 **Chromatography:** A separation technique involving differential retention of components between stationary and mobile phases.

4.5 Baseline: The chromatographic signal plotted as a function of time (or counts as a function of energy for radiation counting) in the absence of signal construction from components of interest.

4.6 Peak: An increase in signal from baseline to a maximum and then back to baseline.

4.7 Coelution: The concurrent elution of two or more compounds to the detector at the same time.

4.8 Elution: The process of movement of compounds from the chromatographic system.

4.9 Carry-over: Carry-over results from system contamination from previous analyses and results in signal unrelated to the current analysis.

4.10 Peak Tailing: Peak tailing is a delayed return of a peak to chromatographic baseline and could be related to a delay of compound elution from the chromatographic system by adsorption or dead volume effects.

4.11 Interference Peaks or Fused Peaks: Peaks that partially or totally coelute with the peak of interest.

5.0 PROCEDURE

5.1 General Requirements

5.1.1 Audit trails or tracking systems MUST BE activated when available within the chromatographic system.

5.1.2 In some situations, manual integrations (MIs) are necessary to compensate for imperfect chromatography, but MIs must be performed ONLY when necessary. Baseline upsets, coelution, RT shifts, and peak shape variation can sometimes complicate automatic integration and analyst intervention through MI may be required to assure consistency between area assignment for standards and samples.

5.1.3 The same integration technique must be applied consistently to field samples and all calibration & QC standards within the same analytical batch affected by the MI. Consistency in integration between standards and samples is the most important consideration in quantitative chromatographic analysis.

5.2 Training Requirements

5.2.1 Initial MI training must be conducted for all new analysts and data reviewers using methods involving peak analysis.

5.2.2 On-going MI training must be conducted annually.

5.3 Reasons to Manually Integrate

MI is not to be used to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Manual integrations are acceptable in the following situations. Other situations may arise which require MI but the decisions for MI must be documented.

5.3.1 Undetected Peak

- A shift in RTs can result in undetected peaks or false positive identification of compounds. A common cause of RT shifts is analysis of highly contaminated samples. If significant RT shifts are observed in surrogate or internal standard (IS) compounds, then the potential of undetected peaks in samples must be carefully reviewed.

- Mass tuning changes that favor the light or heavy end of the mass spectra, or following highly contaminated samples, can sometimes cause the relative abundances of ions of compounds present to deviate from reference criteria, causing the peak to go undetected by the data system.

5.3.2 Incorrect Peak Integration

- Peak has a small amount of splitting and the whole peak area was not integrated.

- Peaks close in RTs utilizing the same quantitation ions often integrate together as one peak, for example: ethylbenzene, xylenes; dichlorobenzenes, benzo(b)fluoranthene and benzo(k)fluoranthene.

5.3.3 Baseline Correction

- Matrix interferences caused by contaminated samples may interfere with calibrated compounds.

5.3.4 Other Examples of Events Requiring Manual Integration

- Incorrectly identified peak, where the wrong peak is chosen by the data system. This can occur both with the primary and the secondary ions.

- To remove a shoulder from a peak or to integrate a peak that only appears as a shoulder.

5.4 Data Systems

The chromatography system's method integration parameters must be optimized to the greatest extent possible so that compounds are properly identified and integrated with minimal operator intervention. Ensure that all analytes in a midpoint standard have sufficient separation prior to calibration (e.g., minimum of a 25% peak/valley ratio, but there may be minor exceptions made on confirmation columns).

NOTE: Even when integration parameters work properly for calibration standards, the analyst must ensure the integration is appropriate on all samples. The analyst must not assume that the chromatography system will automatically apply the correct integration.

The following steps are required when setting the integration parameters to identify peaks as targets (determine the RT). The calibration standards must be reprocessed after updating the RT to demonstrate that the data system integration parameters are set properly:

5.4.1 Process the file using the current data system parameters for the mid-level standard.

5.4.2 Identify all of the target compounds and assign the correct RT to each target compound and the method (Calibration) file and save it.

5.4.2.1 The RTs may be updated daily by using the RTs in the ICV or daily CCV. If updating the RT is a daily procedure (in the method SOP), it need not be recorded as it is a standard procedure.

5.4.2.2 If the RTs are only updated as needed (compounds not identified correctly), record in the instrument maintenance logbook or run log.

5.4.2.3 If the RTs are shifting on any frequent basis (use analytical judgment) within a calibration, instrument problems may be indicated. Perform maintenance and recalibrate the instrument. This **does not** include shifts due to column trimming, adjustments to gas pressure, or instrument maintenance.

5.4.3 Re-process the data and confirm that all of the targets can be identified and properly integrated. If the targets cannot be identified and properly integrated, adjust the integration parameters and re-process the data. This is an important step. If the data system cannot reliably detect and integrate the targets in the mid-level standard, the probability of properly identifying and quantifying the targets in the remaining standards and samples are low.

5.4.4 Process the remaining calibration standards and confirm that the data system can routinely and properly identify the target compounds at each calibration level. Pay particular attention to the lowest level standard because this standard typically defines the reporting limit (RL). The method integration parameters must allow for detection of the target compounds down to the quantitation limit or reporting limit.

5.4.5 The integration parameters as well as major method parameters (those that pertain to calculations/quantitation, e.g., changes to curve fit type, quantitation ion, internal standard assignment) that are set at the initial calibration must remain in use until the next calibration is performed (no changes without recalibration), except as noted below.

NOTE: An individual sample may need to utilize a different quantitation ion in the case of matrix interference. This would require clear documentation on why the change was made.

5.4.6 Any MI of a chromatographic peak or group of peaks must be documented. In all instances where the data system report has been edited or where MI has been performed, the chromatographic system operator must clearly identify such edits or manual procedures as listed below:

5.4.7 MIs indicated on expanded scale "after" chromatograms. That is, the after chromatogram must be presented at sufficient scale expansion to allow data reviewers to independently evaluate the MI. Expanded scale "before" chromatograms are also required for all MIs on QC parameters (calibration standards, QC standards, surrogates, internal standards, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

5.4.7.1 Re-integration technique marked, if available, on the data system.

(060005)

5.4.7.2 Technical justification for MI.

- Either entered in the instrument audit trail for electronic review.
- Clearly marked on the "after" chromatogram, manually or electronically. The table below contains some example abbreviations that could be used to simplify documentation for the reason for MIs. Others means may be used.

Reasons for Manual Integration	Acronym/Code
Baseline	BL
Co-elution	CE
Contamination	CON
Wrong Retention Time	WRT
Tailing	T
Matrix Interference	MI
Splitting	S
No Spectral Match (for deletions)	NSM

5.4.7.3 Analyst's initials and date. (Data system application of analyst name or initials is acceptable so long as the data system allows the analyst to log in as himself or herself and has password control.)

5.4.8 Project specific, client, program or laboratory specific requirements for MI may exceed the requirements of this policy. In those instances, the more specific requirements will apply or written approval allowing a deviation from the requirement must be received. The following programs have specific documentation requirements.

5.4.8.1 Department of Defense (DoD) projects:

- Requiring the before and after printing of all chromatograms.
- Handwritten or electronic initialing and dating the changes made to the report.
- Hardcopy printout of the EICP of the quantitation ion displaying the MI included in the raw data for all standards and samples, this applies to internal standards and surrogates as well.
- For DoD, MIs need to be documented in the raw data ***and*** in the case narrative.

5.4.8.2 Ohio VAP projects:

- Requires the before and after printing of all chromatograms

5.4.9 Data reviewers shall confirm that documentation of MI is complete and that each MI is appropriate. This inspection shall be documented. At a minimum, the information required in Section 5.4.7 must be reviewed, and for DoD & OVAP projects all of the elements in Section 5.4.8 must be reviewed. Any deficiencies must be resolved with the analyst or their supervisor before the results are approved and released from the analytical department.

5.4.10 If integration indicates problems with analytical instrumentation, investigate the problem and take action to correct it. If poor chromatography routinely interferes with the ability to identify and quantify components; e.g., is a result of delayed system maintenance; and is not inherent in the system, such as [benzo(b)fluoranthene and benzo(k)fluoranthene merging with column age; and dichlorobutenes merging with column age], then instrument maintenance must be performed. In the case of isomeric pairs, if resolution does not meet method criteria, it may be more appropriate to report as totals instead of individual peaks.

5.4.11 MIs on some data systems (e.g., Multichrom) may consist of adjusting integration parameters on a sample-by-sample basis. Changes such as these must be well documented.

5.4.12 Acceptable manual integration techniques are detailed in Attachment 1, and include

- valley-to-valley
- drop-to-baseline
- peak skimming

5.4.13 Unacceptable manual integration techniques are detailed in Attachment 2, and include:

- peak shaving
- peak enhancing
- baseline manipulation
- baseline enhancement

5.5 Data Miner Software

5.5.1 Electronic data surveillance is performed using automated data mining software, such *Mint Miner*™ (works with EnviroQuant, Target, and Turbochrom) and *Audit Miner* (works with Chrom). This software allows the user to connect to a workstation or archived data source and evaluate the audit trails in the electronic data file for MIs. It identifies any changes that are made to raw data files so they can be reviewed to ensure integrations were performed in accordance with this SOP.

5.5.2 Any questions arising from review of the documentation must be investigated in the electronic record, when available, prior to data acceptance.

NOTE: Some integrations are not readily obvious on the printouts; in these cases the electronic record must be reviewed. Any failures to complete these requirements shall be described in a non-conformance report.

5.5.3 Any concerns about violations of this policy must be reported to the laboratory Quality Manager, Laboratory Director or the Corporate Quality Director.

(060-007)

6.0 RESPONSIBILITIES

6.1 **Analysts** are responsible for following this SOP and the TestAmerica Ethics and Data Integrity Policy, CW-L-P-004.

6.2 **Data reviewers** are responsible to ensure that all documentation is correct and the decisions to report the data are in accordance with this SOP.

7.0 REFERENCES / CROSS-REFERENCES

7.1 Acceptable Manual Integration Training Presentation. CA-Q-T-001.

7.2 Practical Use of Mint Miner and Audit Miner Training Presentation, CA-Q-T-018.

7.3 Data Mining Using Chrom Audit Miner Training Presentation, CA-Q-T-048

7.4 Ethics Policy, CW-L-P-004.

8.0 ATTACHMENTS

Attachment 1. Examples of Peak Shape and Proper Integration Documenting Manual Integrations.

Attachment 2. Examples of Improper Integration and Inflection Point Guidance.

9.0 REVISION HISTORY

- Revision 0; dated 3 October 2007.
  - Initial Release.
- Revision 0.1; dated 30 November 2007.
  - Moved the first sentence from Sec. 7.2 to be the last sentence in Sec. 6.3.
- Revision 0.2, dated 20 February 2008.
  - Section 2.3: Deleted the statement '..integration below the baseline is never acceptable.'
  - Section 4.2: Clarified that adjusting the actual retention time and not the RTW is a Manual Integration.
  - Section 6.2.2.1: Clarified that the RTs may also be updated by the daily CCV.
  - Section 6.2.4: Clarified reintegration of the low calibration point.
  - Section 6.2.7, Bullet Item 3.2: Clarified that the reason for the MI can documented either manually or electronically.
- Revision 1, dated 2 November 2009.
  - Section 2.1: Clarified annual training.
  - Section 5.2.10: Clarified Manual Integration documentation in the raw data & case narrative for DoD work.
- Revision 2, dated 20 April 2011.
  - Clarifications to separate policy from procedure and training requirements.
  - Simplified definition of manual integration.
  - Expanded discussion of undetected peaks to include all chromatography systems, rather than GC/MS only.
  - Expanded section on data miner software to include Chrom Audit Miner
  - Included reference to data miner training presentation.

(060-008)

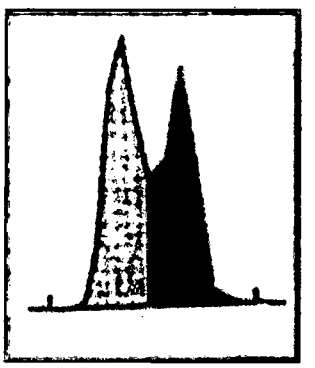
- Revision 3, dated 31 July 2012.
  - Procedure re-organization
  - Sec. 5.4.8 – Added requirement for OVAP-approved laboratories.
  - Attachment 1 - Updated MI examples
- Revision 3.1, dated 1 October 2014.
  - Sec. 5.4.7 – Included surrogates and internal standards to clarify that these QC materials require 'before' & 'after' chromatograms.
  - Sec. 5.4.7 – Removed redundant OVAP reference – defined in Section 5.4.8.
  - Sec. 7.3 – Added reference to 2014 Audit Miner training presentation.

(060-009)

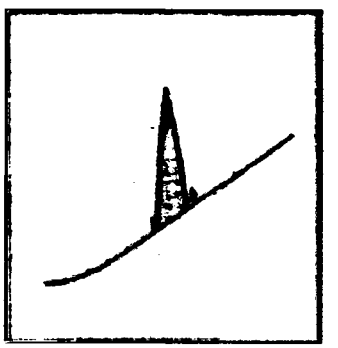
Attachment 1.

Examples of Peak Shape and Proper Integration Documenting Manual Integrations

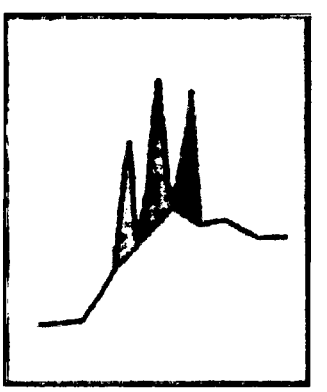
Drop to Common Baseline – One of the most commonly used integration techniques to integrate peak for partially resolved compounds or fused peaks. Fused peaks are near coeluters with some separation. They will occur in calibration mixes as well as real samples. To integrate, split the components with a vertical drop from the valley to the baseline.



Skimmed Peak – Integrating a peak riding on the shoulder of a larger peak. Aside from hydrocarbon analysis, which uses the total area within a window for quantitation, baseline variations are not integrated. Therefore, skim the baseline rise to integrate the peak properly. Use the tangent skimming functions of the integration software to correctly integrate small peaks on the tails of big peaks.



Valley-to-Valley Integration – Most often used for GC methods when peaks of interest are riding on mass of unresolved interference peaks, commonly seen in chromatograms for samples with high concentrations of petroleum hydrocarbons.



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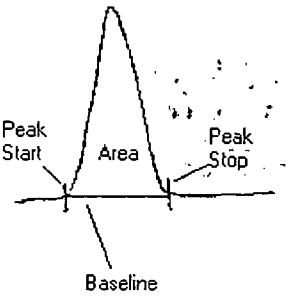
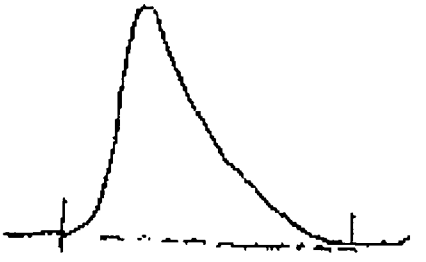
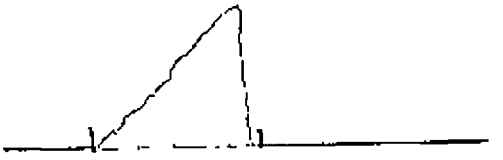
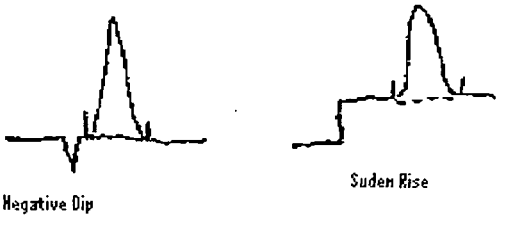
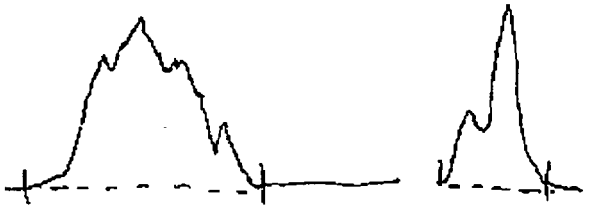
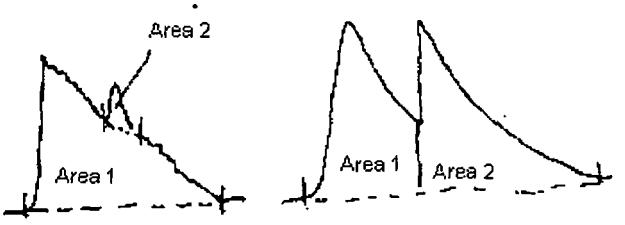
Ideal peak **(Gaussian Shape)** The start of integration is where the peak begins to rise from the background, and the end is where the peak returns to background.	
Tailing Peak Tailing peaks result from surface adsorption effects or dead volume in the instrument. When integrating tailing peaks, include only and all area that can be attributed to compound. If the baseline rises, the integration line should rise with the baseline.	

(060-011)

Fronting Peak Fronting peaks generally result from column overload and/or overcapacity. An example is benzoic acid on a 5% phenyl phase. In some cases, it is inherent in the analysis, in others, it is indicative of problems in the system. The analyst must have knowledge of their system. To integrate these peaks, it is important to have prior knowledge of how the compound acts when overloading versus co-eluting with a contaminate peak.	
Hydrocarbon Envelope For semivolatile hydrocarbon analysis, most fuels produce "humpograms" like this one. Integration shall be performed in accordance with method, program or client specific requirements.	
Peak near a baseline upset Negative dips are common in ECD analysis, and the sudden rises in baseline are common with programmable fluorescence detectors where gain/wavelength switching occurs. Often, the default integration will go from the base of the negative peak to the far side of the peak of interest. This is results in high bias and is incorrect. Similarly, a default integration using the low part of the baseline rise and the far side of the peak of interest will also result in high bias and must be corrected.	

(060-012)

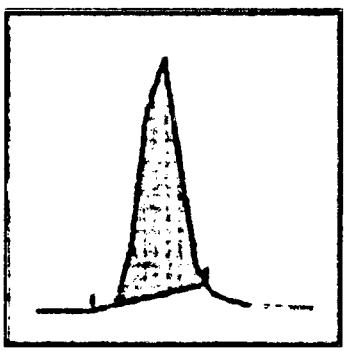
Split Peak Particularly in GC/MS analysis, low-level peaks may appear jagged or split. To integrate these properly, the analyst must have prior knowledge of the peak/compound chromatography.	
Fused tailing peaks These are very difficult to accurately quantify. It is best to do maintenance on the chromatography system to avoid this situation. To integrate these properly, the analyst should have prior knowledge of the compounds of interest. It is important to be consistent when integrating standards and samples.	

(060-013)

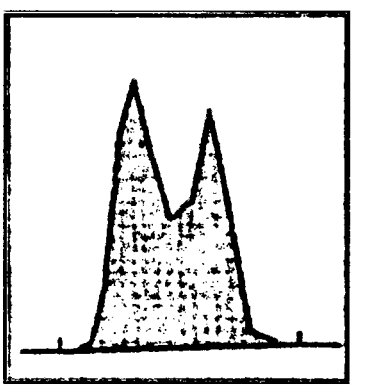
Attachment 2.

Examples of Improper Integration and Inflection Point Guidance

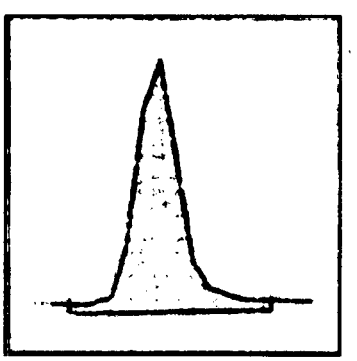
Peak Shaving – Intentionally removing peak areas inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.



Peak Enhancing – Improperly enhancing peak areas by combining multiple peaks to produce a value for a single compound (sometime called "mountain ranging") inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples. Peaks at low concentrations can combine with a noisy baseline unintentionally giving the appearance of improper peak enhancement – data must be rejected if the chromatogram is not clear enough that improper enhancement cannot be ruled out.

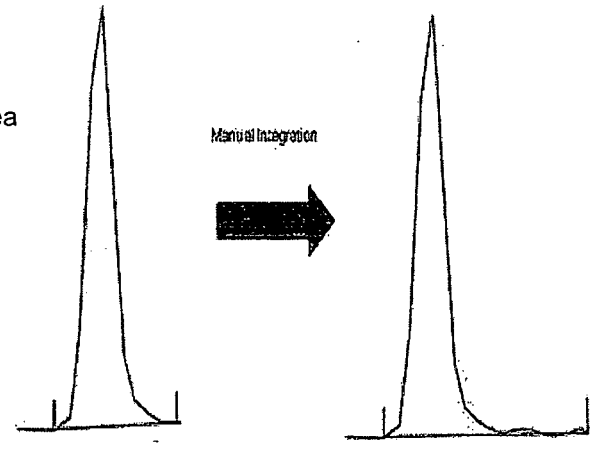


Baseline Manipulation – Artificially dropping a baseline to increase the area (sometimes called "boat anchoring" or a "leggo peak") in a manner inconsistent with valid chromatographic principles and the integration technique performed on the initial calibration, other QC samples, or field samples.

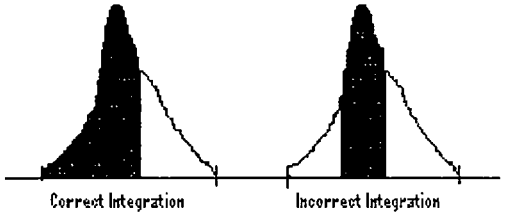


(060-014)

Baseline Extension – Improperly adding area by expanding the width of the integration range beyond the width of the peak inconsistent with valid chromatographic principles and the integration techniques performed on the initial calibration, other QC samples, or field samples.



An inflection point must be apparent when and if an integration point is drawn. For example:



When MS data has an inflection point in a peak, as shown in the example above, checking the underlying spectrum may be necessary to correctly manually integrate. On some chromatographic columns, for example, allyl chloride and carbon disulfide elute very close to each other, and they both share the primary characteristic ion mass 76. For both compounds mass 76 is the quantification mass. If two compounds can not be separated and a fused peak is going to be split at the inflection point, this can be correctly verified by looking at the spectra for each scan. In this example, the 76 peak may be fused, but the secondary ions will indicate which compound is present (carbon disulfide with secondary ion 78 and allyl chloride with secondary ions 41, 39 and 78).

Attachment 9.

List of Poor Purging or Poorly Performing Compounds

Acetone	Ethyl Acetate
Acetonitrile	2-Hexanone
Acrolein	Isobutyl Alcohol
Acrylonitrile	Methacrylonitrile
Bromomethane	Methyl Acetate
n-Butanol	4-Methyl-2-pentanone
2-Butanone (MEK)	2-Nitropropane
Carbon Disulfide	Propionitrile
Chloroethane	Tetrahydrafuran
2-Chloroethylvinyl ether	Trans-1,4-dichloro-2-butene
Chloromethane	1,1,2-Trichloro-1,2,2-trifluoromethane
Dichlorodifluoromethane	Trichlorofluoromethane
1,2-Dibromo-3-chloropropane	Vinyl Acetate
Ethyl Acetate	Isopropyl alcohol
Ethanol	2-Methyl-2-propanol
1,4-Dioxane	

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APPENDIX B

SCS Standard Procedures

Appendix B
SCS Standard Procedures

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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 BT^2).

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 PHOTOIONIZATION DETECTOR

A photoionization detector (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.