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Title: GC/MS Analysis Based On Methods 8270C and 625

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1.0 Scope and Application

1.1 This method is based upon EPA Method SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.

1.1.1 The modifications presented in Appendix A may be followed for analysis of wastewater by Method 625.

1.1.2 The modifications presented in Appendix B may be followed for analysis of wastewater using method 8270 (Best Practices). The best practices method provides for lower reporting limits in aqueous samples.

1.1.3 Direct injection of a sample may be used in limited applications.

1.1.4 Refer to Tables 1, 2 and 3 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. This method may be amenable to additional compounds. If non-standard analytes are required, they must be validated by the procedures described in Section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

1.2.1 Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography.

1.2.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.2.3 N-Nitrosodimethylamine may be difficult to separate from the solvent under the instrument conditions in the method.

1.2.4 N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.

1.2.5 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.2.6 Pyridine may perform poorly at the injection port temperatures described in the method.

1.2.7 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as 3/4-methylphenol.

1.2.8 Hexachlorophene and famphur analyses are not quantitatively reliable by this method.

1.2.9 Kepone should be analyzed by GC/ECD.

1.2.10 The calibration standard includes azobenzene. Azobenzene is a decomposition product of 1, 2-Diphenylhydrazine. In the calibrations standard both compounds are included, however the concentration for 1, 2-Diphenylhydrazine is adjusted to compensate for the decomposition using a factor of 1.011 determined as follows:

A/B where A = the molecular weight of Azobenzene and B = the molecular weight of 1, 2-diphenylhydrazine.

1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 – 3.3 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 4 - 200 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1, 2, and 3 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

2.0 Summary of Method

2.1 Aqueous samples are extracted with methylene chloride using a continuous extractor or a separatory funnel. TestAmerica Denver performs extractions using a one liter sample aliquot or a 250 mL sample aliquot. The extract is dried, concentrated to 1 mL and analyzed by GC/MS.

2.1.1 Samples extracted using 250 mL are analyzed following the Large Volume Injection (LVI) procedures described in this SOP.

2.1.2 LVI is used only for aqueous samples. It is utilized to maintain reporting limits while reducing the initial volume of the sample required for extraction. The extraction procedure is the same with the only adjustments to sample size, spike concentrations and volume of extraction solvent.

2.2 Solid samples are extracted with methylene chloride / acetone using sonication or Soxhlet extraction. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS.

2.3 Waste dilution is used for samples that are miscible with the solvent.

2.4 Extraction and cleanup procedures are detailed in the following SOPs:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series
DV-OP-0007	Concentration and Cleanup of Organic Extracts, SW846 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0010	Soxhlet Extraction of Solid Samples, SW846 3540C
DV-OP-0012	Waste Dilution Preparation for Non-Aqueous Waste Samples, SW-846 3580A
DV-OP-0016	Ultrasonic Extraction of Solid Samples, SW846 3550B and 3550C

2.5 Sample extracts are analyzed by capillary GC/MS. The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) where the analytes are separated and then introduced into the mass spectrometer. The mass spectra are produced by electron impact. Qualitative identification is accomplished by comparing the extract spectra with the spectra of standards. Identification of target analytes of the analytes in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique, with a single characteristic ion and a minimum of a five-point calibration curve.

2.6 For LVI the GC/MS analysis is the same with the exception of the injection liner, injection volume and standard concentrations.

3.0 Definitions

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCCs is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / matrix spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.

3.3.1 Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica QC Program document (DV-QA-003P) for further details of the batch definition.

3.3.2 Some clients or programs (e.g., DoD) specify the frequency of the MS/MSD relative to the field sampling and do not link it to the QC batch in the lab.

3.4 Method Blank (MB) - An analytical control consisting of all reagents, internal standards and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.5 Laboratory Control Sample (LCS) - A blank matrix (reagent water or Ottawa Sand) spiked with the analytes of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked analytes demonstrates that the laboratory techniques for this method are acceptable.

3.6 Matrix Spike (MS) – An aliquot of a matrix (water or soil) fortified (spiked) with known amounts of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7 Matrix Spike Duplicate (MSD) - A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method by measuring the relative percent difference (RPD) between the MS and MSD results.

3.8 Surrogates - Organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Surrogate recoveries are used to assess accuracy, method performance, and extraction efficiency for each sample analyzed. Each sample, blank, LCS, MS, and MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits for the project.

4.0 Interferences

4.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample. Cleanup procedures may help to eliminate select interferences, as follows:

4.1.1 Method 3660B, Sulfur Cleanup – If a sulfur peak is detected, copper or mercury can be used to treat the extract and remove the sulfur.

4.1.2 Other, more aggressive cleanup procedures listed in SW-846 may be used for select compounds listed in this procedure, but may cause degradation of some of the more reactive compounds. Consult with a technical expert in the laboratory for more difficult interference problems.

NOTE: Details concerning cleanup steps are described in the organic extraction SOPs (see Section 2.4).

4.2 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section (Section 9.2). Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.3 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 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, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

NOTE: Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

5.3.2 The gas chromatograph and mass spectrometer contain 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.

5.3.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.3.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power before performing any maintenance.

5.4 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 MSDS 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 MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Significant or Serious Hazard Rating

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Methanol	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.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system: an analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 30 m x 0.25 mm I.D., 0.5-µm film thickness fused-silica capillary column coated with 5% diphenyl/95% dimethyl polysiloxane (Restek Rtx®-5MS or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 u (previously "amu") every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) that meets all of the criteria in Table 5 when 25 ng of the GC/MS tuning standard is injected through the GC.

6.4 Autosampler: HP7683 Autosampler or equivalent, with appropriate sample trays.

6.5 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

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

6.7 Analytical Syringe: 5 µL or 10 µL Hamilton Laboratory grade syringes or equivalent. The 5 µL syringe is used for the Agilent ALS to be able to inject 0.5 µL and the 10 µL syringe is commonly used for the Large Volume Injection.

6.8 Sample Aliquot Equipment: Laboratory grade syringes and pipettes as appropriate to dilute and aliquot extracts. Also, amber glass aliquot vials with polytetrafluoroethylene (PTFE)-lined screw or crimp caps and suitable for use with the autosampler.

6.9 Carrier gas: Ultra high-purity helium.

6.10 Computer Software and Hardware

Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Methylene Chloride, equivalent to pesticide quality or better, used to dilute samples as needed.

7.2 Stock standards are received from the vendor in multiple mixtures for each set of calibration standards, for both primary ICAL standards and second source verification standards. Other standards may be used to accommodate client-specific target lists or reporting limit requirements.

7.2.1 Surrogate Standards:

Restek 567685	Surrogates at 5000 ug/mL

7.2.2 Internal Standards:

Restek 567684	Internal Standards at 2000 ug/mL

7.2.3 HSL (Primary) Standards:

Restek 567672	Mega Mix at 1000 ug/mL
Restek 567673	Amine Mix at 2000 ug/mL
Restek 567674	Benzoic Acid at 2000 ug/mL
Restek 567675	Indene at 2000 ug/mL
Restek 568023	Famphur at 2000 ug/mL
Restek 568033	HSL Custom Mix at 4000 ug/mL
Restek 568038	N-Nitrosodiphenylamine at 2000 ug/mL

7.2.4 AP9 Standards:

Restek 567678	Supplemental Standard 1 at 1000 ug/mL
Restek 567679	List 2 Standard 2 at 1000 ug/mL
Restek 567680	List 2 Standard 3 at 2000 ug/mL
Restek 567681	List 2 Standard 4 at 1000 ug/mL
Restek 567682	List 2 Standard 5 at 2000 ug/mL
Accustandard H-173N	Dibenz[a,j]acridine, neat

7.2.5 Benzaldehyde Standards:

Restek 567677	Benzaldehyde at 2000 ug/mL

7.2.6 Hexachlorophene Standards:

Restek 31811	Hexachlorophene at 2000 ug/mL

7.2.7 Custom Standards:

Restek 568748	Custom Stock 1 at 2000 ug/mL
Restek 568749	Custom Stock 2 at 2000 ug/mL

7.2.8 Refinery Standards:

Supelco 21579195B	Refinery Stock at 2000 ug/mL
SPEX S1200	Dibenzo(a,e)pyrene

7.2.9 PHE Standards:

Sigma Aldrich P23954	1,3-Phenylenediamine, neat
Supelco 442315	2,4-Xylidine, neat
Sigma Aldrich A88182	o-Anisidine, neat
Sigma Aldrich 103284	5-Methyl-o-Anisidine, neat

7.3 A minimum of seven calibration standards are typically prepared. Refer to Tables 10 and 11 for common calibration levels of the HSL and Appendix 9 analytes by the standard method. Other calibration compounds and levels may be used, depending on client requests and instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration. The low point should be at or below the reporting limit. A minimum five-point calibration curve is required when average response factors or linear regression curve fitting is used. Six calibration points are required for second-order curve fits. Table 12 provides calibration levels for many of the non-standard compounds listed in Table 3. Tables LVI-1, LVI-2 and LVI-3 provide calibration levels for LVI.

7.4 Initial Calibration Verification (ICV) Standards: For each curve, containing all components from a second source (an alternate vendor, a unique lot from the same vendor, or the same source but prepared by an alternate analyst). The ICV concentration may vary on a per-compound basis, but is usually prepared at 80 or 100 µg/mL concentration for full scan or 25 µg/mL for LVI. The Initial Calibration Verification must be ≤ ½ the concentration of the highest calibration standard for each analyte.

7.5 Continuing Calibration Verification (CCV) Standards: Also prepared for each curve and containing all the same components as was calibrated for, from the same vendor source as the calibration standards (Section 7.2). The CCV is usually prepared at the 80 µg/mL concentration for full scan analysis, and 20 µg/mL for LVI. It must be ≤ ½ the concentration of the highest calibration standard for each analyte.

7.6 An internal standard (IS) solution is prepared. Compounds in the IS Mix are acenaphthene-d_{10}, chrysene-d_{12}, 1,4-dichlorobenzene-d_4, naphthalene-d_8, perylene-d_{12}, and phenanthrene-d_{10}.

7.6.1 Internal standards are added to all standards and extracts to result in a final concentration of 40 µg/mL. For example, if the volume of an extract aliquot used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added to the aliquot for the standard analysis. See Appendix B for the levels used for the 8270 best practice method.

7.7 Surrogate Standard Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOP numbers). Concentration levels for surrogate compounds are listed in Table 9.

Acid Surrogates	**Base Surrogates**
2-Fluorophenol	2-Fluorobiphenyl
2,4,6-Tribromophenol	Terphenyl-d_4
Phenol-d_5	Nitrobenzene-d_5
2-Chlorophenol-d_4[1]	1,2-Dichlorobenzene-d_4[1]

[1] These two surrogates are in the mix but are not evaluated or reported.

7.8 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT should also be included in the Tuning Standard at 50 µg/mL.

7.9 Laboratory Control Spiking Solution: Prepare as indicated in the SOP DV-OP-0020. The reported components of each LCS may vary and are selected by the client from the list of target analytes. For further information and to see current spike volumes and spiking

solution concentrations see WI-DV-009 and the LIMS Reagent module.

7.10 Matrix Spike Solution: Prepare as indicated in the SOP DV-OP-0020. The matrix spike compounds and levels are the same as the LCS compounds.

7.11 The standards listed in sections 7.1 to 7.10 must be stored at -10°C to -20°C if it can be demonstrated that analytes do not fall out of solution at these temperatures. If not stable, the standards should be stored between 0°C and 6°C. The standard stock solutions expire after one year from preparation date or at the earliest expiration date assigned by the vendor to any parent standard, whichever is earlier. The continuing calibration standard should be replaced when there are visible signs of degradation or when the standard fails to meet QC criteria. The continuing calibration standard is stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Water	1 liter amber	1 liter	Cool, ≤6°C and not frozen	7 Days	40 Days from extraction	40 CFR Part 136.3, SW-846
Water[1]	250 mL amber	250 mL	Cool, ≤6°C and not frozen	7 Days	40 Days from extraction	40 CFR Part 136.3, SW-846
Soil	4 oz Jar	30 grams	Cool, <6°C and not frozen	14 Days	40 Days from extraction	SW-846

[1] Samples extracted using 250 mL are analyzed following the LVI procedure.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Method Blank (MB)

For aqueous sample batches, the method blank is reagent water; for solid sample batches, the method blank is clean sand. In either case, the method blank is free of the analytes of interest and is spiked with the surrogates. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than ½ of the reporting limit or less than 10% of the analyte concentration found in the associated samples, whichever is higher. When a compound is above ½ the reporting limit a NCM needs to be completed.

NOTE: All programs require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: Re-preparation and reanalysis of all samples associated with an unacceptable method blank. If the analyte was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.3 Instrument Blank

Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank. (See Section 9.2.)

9.4 Laboratory Control Sample (LCS)

The LCS is prepared using reagent water for aqueous methods and Ottawa sand for solid sample methods. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. The LCS is spiked with the compounds as described in Section 7.8 unless specified by a client or agency. The compounds is typically spiked at a concentration equivalent to 80 µg/L, depending on the analyte, unless a special QAS states a specific level. (The LVI method uses the same concentration of analytes for the LCS.) Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Acceptance Criteria: All analytes must be within established control limits. See Quality Assurance Program DV-QA-003P for details on establishing control limits.

Corrective Action: If any analyte in the LCS is outside the laboratory-established historical control limits or project-specific control limits, as

applicable, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be documented and reviewed with the client before reporting.
- If re-extraction and reanalysis of the batch are not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The matrix spike is a second aliquot of one of the samples in the batch. The matrix spike duplicate is a third aliquot of the same sample. The MS and MSD are spiked with the same analytes as the LCS (see Sections 7.8 and 7.9). An MS/MSD pair is prepared and analyzed with every batch of samples.

Acceptance Criteria: The percent recovery (%R) must fall within either historical limits or project-specific limits, as applicable. The relative percent difference (RPD) between the MS and MSD results should be less than or equal to the established historical or project-specific limit. See Quality Assurance Program Policy DV-QA-003P for details on establishing control limits.

Corrective Action: If any individual recovery or RPD fails the acceptance criteria, then corrective action must occur. Initially check the recovery of the analyte in question in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is considered to be in control and analysis may proceed.

- If the recovery for any analyte fails acceptance criteria for the MS, MSD, and the LCS, the laboratory operation is considered to be out of out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.
- The MS/MSD pair must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be over-range or be diluted to concentrations below the calibration range.

9.6 Surrogates

9.6.1 Each sample, blank, and QC sample is spiked with the surrogate standards. Surrogate compounds must be spiked at 100 µg/L. Compounds in the Primary Standard are each assigned to a surrogate based on the AFCEE QAPP, or if not specified therein, the retention time proximity to the nearest surrogate. The compounds routinely included in the surrogate spiking solution, along with their routine concentrations, are listed in Table 9. For the Best Practice method, see Table B-4 in Appendix B.

Acceptance Criteria: Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

Corrective Action: If any surrogates are outside of the limits, then the following corrective actions must take place (except for dilutions):

* Check all calculations for error.
* Ensure that instrument performance is acceptable.
* Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
* Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if none of the above resolves the problem.

NOTE: The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.6.2 If the sample with failed surrogate recoveries was a sample used for an MS/MSD pair and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample and the MS and the MSD do not require reanalysis. This phenomenon indicates a possible matrix problem.

9.6.3 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate).

9.6.4 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effects.

9.6.5 If the sample cannot be re-analyzed for some reason (e.g., not enough volume for re-extraction), then any detected associated compounds in the sample are flagged as estimated due to surrogate failure.

9.7 Internal Standards

9.7.1 The peak areas of the internal standards are monitored in all field samples and QC samples.

Acceptance Criteria: The peak area for each internal standard should be between 50% and 200% of the peak area for the designated midpoint calibration ("ICIS") standard in the initial calibration. This is typically the 80 ppm Primary (HSL) standard for standard 8270C and the 20 ppm Primary (HSL) standard for LVI.

Corrective Action: If recovery for any internal standard is outside of the control limits, check for calculation errors or instrument problems, and reanalyze the associated samples at a dilution for the compounds quantified against the internal standard that was outside of control limits.

9.7.2 The retention times of the internal standards are monitored in all field samples and QC samples.

Acceptance Criteria: The retention time for any internal standard must be within ± 0.5 minutes of the last continuing calibration standard.

Corrective Action: If the retention time of any internal standard is greater than ± 0.5 minutes the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed if the retention time is outside of limits is required. If the retention time of any internal standard is between ± 0.1 minutes and ± 0.5 minutes, from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

9.8 2012 MUR Required QC Elements

The May 2012 EPA Method Update Rule (MUR) to 40 CFR Part 136 for compliance testing under the Clean Water Act (CWA) requires laboratories to include 12 QC elements when performing the published or approved methods. See Work Instruction WI-DV-0060, QC Requirements for Methods Designated in 40 CFR Part 136, for list of approved test procedures performed by TestAmerica-Denver and the required QC elements in each of these methods.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

Samples are prepared according to the following organic preparation SOPs, as applicable:

DV-OP-0006 Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series

DV-OP-0007 Concentration and Cleanup of Organic Extracts, SW846 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A and EPA 600 Series

DV-OP-0008 Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607

DV-OP-0010 Soxhlet Extraction of Solid Samples, SW846 3540C

DV-OP-0012 Waste Dilution Preparation for Non-Aqueous Waste Samples, SW-846 3580A

DV-OP-0016 Ultrasonic Extraction of Solid Samples, SW846 3550B AND 3550C

10.4 Sample Analysis Procedure

10.4.1 Calibrate the instrument as described in Section 11. Depending on the target compounds required by the client, it may be necessary to use more than one set of calibration standards.

10.4.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration verification (CCV) standard. Extracts are allowed to warm to room temperature before aliquotting. Excess extract is returned to the 0°C to 6°C refrigerator for storage.

10.4.3 Internal Standard

Add internal standard to an aliquot of the extract to result in a 40 ng/µL concentration (for example, 20 µL of internal standard solution at 400 µg/mL in 200 µL of extract). Mix thoroughly before injection into the instrument.

10.4.4 Inject the aliquot into the GC/MS system using the same injection technique as used for the standards.

10.4.4.1 For standard volume extracts the injection volume is 0.5 µL.

10.4.4.2 For LVI, the injection volume is 2.5 µL.

10.4.5 Qualitative identification of each analyte in the extract is evaluated by the data system using the criteria listed in Section 12.2. The data system will determine the concentration of each analyte using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration verification.

10.4.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst (see DV-QA-011P, *Acceptable Manual Integration Practices*) or automatically by the data system. The minimum documentation required includes the original data system peak integration, a similarly scaled hard copy showing the manual integration, the reason for the manual integration, analyst initials and date. All manual integrations must be reviewed by a second level reviewer. For Level 4 data packages (e.g., Federal projects or other client specified projects) second level review is documented with date and initials on the manual integration summary report. This documentation is then scanned and attached to the project in the LIMS to be included with the Level 4 data package.

10.4.7 Library searches of peaks present in the chromatogram that are not target compounds, i.e., Tentatively Identified Compounds (TIC), may be performed if required by the client. They are evaluated using the criteria in Section 12.3.

10.5 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a new aliquot of the extract is diluted and prepared (IS added to the aliquot) and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below approximately 50% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.5.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are significantly less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

10.5.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.6 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them between 0°C to 6°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

10.7 Retention Time Criteria for Samples

10.7.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.7.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

10.8 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP DV-WC-0023 for determination of percent moisture.

10.9 Data Review

10.9.1 All data are reviewed by the analyst and documented on a checklist, GCMS-BNA TALS Data Review. See SOP DV-QA-0020 and DV-WI-0025.

10.9.2 The data package and review checklist are submitted to a peer reviewer (or supervisor) for the level 2 review. All manual integrations must be evaluated by the peer reviewer. The level 2 review is documented on the review checklist initiated at the level 1 review. See SOP DV-QA-0020 and DV-WI-0025.

10.9.3 Initial calibrations are subject to a separate two-level review that is documented on a checklist, GC/MS TALS Initial Calibration Review Checklist. See SOP DV-QA-0020.

10.10 Troubleshooting Guide

10.10.1 Daily Instrument Maintenance

In addition to the checks listed in Appendix C, the following daily maintenance should be performed.

- Clip column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Install new or cleaned gold seal and washer as necessary.
- Perform mass calibration as necessary.

10.10.2 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the repeller, cleaning the source, replacing the multiplier, and replacing the "top board" or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

11.0 Calibration

11.1 Summary

The instrument is tuned for DFTPP, calibrated initially with a minimum of a five levels, and verified each 12-hour shift with one or more continuing calibration standard(s). The number of continuing calibration standards depends upon the target compounds of interest in the analytical batch. Recommended instrument conditions for both the standard analysis and LVI are listed in Table 4.

11.2 All standards and extracts are allowed to warm to room temperature before injecting.

11.3 Instrument Tuning

11.3.1 At the beginning of every twelve-hour shift when analyses are to be performed, the GC/MS system must be checked to see if the acceptance criteria are achieved for DFTPP (decafluorotriphenylphosphine). See Table 5.

11.3.2 Inject 25 ng of the GC/MS tuning standard (Section 7.8) into the GC/MS system. Generate the automatically background-corrected mass spectra of DFTPP from Chrom or other quantitation software and confirm that all the key m/z criteria in Table 5 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

11.3.3 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. The acceptance criteria for the peak tailing factor for benzidine is <3.0 and pentachlorophenol is <5.0. DDT breakdown must be <20%. Refer to Section 12 for the appropriate calculations.

11.4 Initial Calibration

11.4.1 Detailed information regarding calibration models and calculations can be found

in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and under the public folder, Arizona Calibration Training.

11.4.2 Internal Standard (IS) Calibration Procedure: Internal standards are listed in Table 6. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

11.4.3 Compounds are typically assigned to the IS with the closest retention time.

11.4.4 A minimum of seven calibration standards are typically prepared, however the number of calibration points may vary. A minimum of five concentration levels are required for each parameter of interest when average response factors or linear regression curve fits are used. At a minimum, six standards must be used for a quadratic least-squares calibration. It may also be useful to analyze six or seven calibration levels and use the lower five or six for most analytes and the upper five or six for analytes that have poor response.

11.4.5 Rejection of Calibration Points

11.4.5.1 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

11.4.5.2 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious misinjection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

* The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and
* The lowest remaining calibration point is still at or below the project reporting limit; and
* The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and
* The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

11.4.6 Internal Standards

Add the internal standard mixture to each calibration standard, resulting in a 40 ng/µL final concentration. (For example, if the volume of the calibration standard used is 0.5 mL, add 50 µL of the 400 µg/mL internal standard).

11.4.7 The concentrations of all analytes in the calibration standards are listed in Tables 10, 11 and 12 for the standard analysis and Tables LVI-1, LVI-2 and LVI-3 for LVI. For the Best Practice method, see Table B-5 in Appendix B.

11.4.8 Analyze each calibration standard and tabulate the area of the primary

characteristic m/z against the concentration for each compound and internal standard. Calculate the response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in Section 12. Verify that the CCC and SPCC criteria, which are specified in Sections 11.4.10 and 11.4.9, are met. No sample analysis may be performed unless these criteria are met.

11.4.9 Calibration Check Compounds (CCCs)

The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this check.

11.4.9.1 If none of the CCCs are required analytes, then project-specific calibration specifications (which may include the use of the CCCs listed in Section 11.4.9.2) must be implemented with concurrence from the client.

11.4.9.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-Nitrosodiphenylamine (as Diphenylamine)
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

11.4.10 System Performance Check Compounds (SPCCs)

The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

11.4.10.1 SPCC Compounds:

N-Nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

11.4.11 If the RSD of any target analyte is 15% or less, then average relative response factor may be used for quantitation of that analyte.

11.4.11.1 If the RSD in the initial calibration is > 15%, then calibration using a curve fit must be used. Linear or quadratic curve fits may be used. If

it appears that substantially better accuracy would be obtained using quantitation from a curve fit, then the appropriate curve should be used for quantitation. Use of 1/Concentration or 1/(Concentration)^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

If a linear regression equation is used, the correlation coefficient r must be greater than 0.990. The Laboratory information management system, TALS, always controls the correlation for curve fits in terms of r^2 which is defined as the mathematical square of the correlation coefficient, r for a linear regression, in which case $r^2>(0.990)^2 = 0.9801$. For a quadratic equation, r^2 and COD are identical and the requirement is that the value will be >0.990. Use of second-order regression equations may be used on rare occasions. In these cases, the intercept and degree of curvature should be examined to be sure that results will be reliable throughout the working range, and the coefficient of determination (r^2) must be greater than 0.990.

Note: South Carolina does not allow use of second order calibration models.

11.4.11.2 An initial calibration verification (ICV) containing all components from a second source (an alternate vendor, a unique lot from the same vendor, or the same source but prepared by an alternate analyst) must be analyzed after calibration and prior to analyzing other QC or samples. Acceptance criteria for ICV percent recovery (%R) are dependent on the requirements of the client or project. For example, DoD QSM 4.2 requires 80-120% ICV recovery; but non-DoD projects may only require 65-135% for routinely linear compounds (e.g., 625/8270C HSL components); and clients generally accept laboratory recoveries of 45-155% for poorly performing compounds (analytes where the CCC RSD exceeds 15%, e.g., 8270C AP9, Custom, Refinery, Di-n-butyl phthalate, benzaldehyde).

Note: Verify that the CCC and SPCC criteria, which are specified in Sections 11.4.10 and 11.4.9, are met. No sample analysis may be performed unless these criteria are met.

11.4.12 Weighting of Calibration Data Points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentration or 1/(Concentration)^2 weighting (often called 1/x or $1/x^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

11.4.13 If time remains in the 12-hour period initiated by the DFTPP injection immediately preceding the initial calibration, samples may be analyzed following the initial calibration. Otherwise, proceed to continuing calibration, Section 11.5.

NOTE: Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

11.5 Continuing Calibration Verification (CCV)

11.5.1 At the start of each 12-hour analysis period, the GC/MS tuning standard must be analyzed. A 25-ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 5.

11.5.2 Following a successful DFTPP analysis, the continuing calibration verification (CCV) standard(s) are analyzed. The standard(s) must contain all semivolatile analytes, including all required surrogates. A mid-level calibration standard is used for the CCV. The standard concentrations used for the CCV are:

Standard ID	Concentration (µg/mL)
Primary (HSL) Standard	80
Appendix 9 Standard (AP9)	80
Refinery (REF)	80
Custom (CUST)	80
Phenyl Compounds (PHE)	80
Dinitroxylenes (DNX)	80
LVI HSL	20
LVI AP9	20
LVI REF	20

11.5.3 The following criteria must be met for the CCV to be acceptable:

11.5.3.1 The SPCC compounds must have a response factor ≥ 0.050.

11.5.3.2 The percent difference or drift (%D) of the concentration of the CCC compounds must be ≤ 20%. (See Section 12.8 for calculation.)

11.5.3.3 For compounds of interest, reliably performing compounds (see Table 14, List 1 Reliably Performing Compounds) should have a %D ≤ 35%. Poorly performing compounds (see Table 15, List 2 Poorly Performing Compounds) should have a %D ≤ 50%. Up to 6 target analytes from the entire set of target analytes are allowed to have %D values greater than the applicable limit. Any compound of interest that does not meet the applicable criteria must be documented in a NCM.

11.5.3.4 The internal standard (IS) response of the CCV must be within 50 - 200% of the response in the ICIS of the initial calibration.

11.5.3.5 If any internal standard (IS) retention time in the CCV changes by more than 30 seconds from that of the same level of the corresponding initial calibration, the chromatographic system must be inspected for malfunctions and corrections made as required.

11.5.3.6 If none of the CCCs are required analytes, project-specific calibration requirements (which may include the use of the CCCs listed in Section 11.4.9.2) must be implemented with concurrence from the client.

11.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample injected less than or equal to 12 hours after the DFTPP is acceptable.)

NOTE: Some states (like Arizona and South Carolina) have special requirements. Please refer to the posted QAS.

12.0 Calculations / Data Reduction

12.1 Detailed calibration equations can be found in the corporate SOP CA-Q-S-005 "Calibration Curves" and under the folder, Arizona Calibration Training.

12.2 Qualitative Identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. See Tables 6 and 7 for characteristic ions and approximate retention time order in the primary (HSL) and Appendix 9 standards. Table 8 provides characteristic ions for non-routine compounds.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

12.2.1 The retention time for the component in the sample must compare to within ± 30 seconds of the retention time of the component in the standard. For reference, the standard must be run within the same twelve hours as the sample.

12.2.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.2.3 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.2.4 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.2.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.3 Tentative Identification

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches

shall the mass spectral interpretation specialist assign a tentative identification. Following are guidelines for making tentative identification:

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

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

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

12.3.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or the presence of co-eluting compounds.

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

12.3.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.4 Isomers with identical mass spectra and close elution times pose problems for definitive identification. The following compounds, listed in elution order, fall into this category.

Aniline and bis(2-chloroethyl) ether
1,3-Dichlorobenzene, 1,4-dichlorobenzene, and 1,2-dichlorobenzene
2-Methylphenol, 3-methylphenol, and 4-methylphenol
(Note: 3-methylphenol and 4-methylphenol co-elute)
2,4,6-Trichlorophenol and 2,4,5-trichlorophenol
Phenanthrene and anthracene
Fluoranthene and pyrene
Benzo(a)anthracene and chrysene
Benzo(b) and (k)fluoranthene

Identification of these compounds requires both experience and extra precautions on the part of the analyst. Specifically, the analyst must more closely scrutinize the comparison of retention times between the unknown and the calibration standard. The analyst must also check that peak resolution is sufficient (valley height of less than 25% of the sum of the two peak heights).

12.5 A second category of problem compounds consist of the poor responders or compounds that chromatograph poorly. The integrations for these types of compounds should be checked manually. The following compounds are included in this category:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Atrazine
Famphur
Benzidine

12.6 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x	=	Area of the characteristic ion for the compound being measured
A_{is}	=	Area of the characteristic ion for the specific internal standard
C_x	=	Concentration of the compound being measured (µg/L)
C_{is}	=	Concentration of the specific internal standard (µg/L)

12.7 Calculating the Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\%$$

Where:

$\overline{RF}$	=	Mean of RFs from the initial calibration for a compound
SD	=	Standard deviation for the mean RF from the initial calibration for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

RF_i	=	RF for each of the calibration levels
n	=	Number of RF values

12.8 Calculating the Continuing Calibration Percent Drift

$$\%Drift = \frac{C_{found} - C_{actual}}{C_{actual}} \times 100\%$$

Where:

C_{actual}	=	Known concentration in standard
C_{found}	=	Measured concentration using selected quantitation method

12.9 Calculating the Concentration in the Extract

The concentration of each identified analyte and surrogate in the extract is calculated from the average RF, or linear or quadratic curve fitted to the initial calibration points. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until the calibration acceptance criteria are met.

12.9.1 Average Response Factor Calibration

If the RSD of the response factors for each compound of interest in the initial calibration is ≤15%, the average response factor from the initial calibration may be used for quantitation. Average response factor is calculated as follows:

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

Where:

C_{ex}	=	Concentration in the extract, µg/mL
R_x	=	Response for the analyte
R_{is}	=	Response for the internal standard
C_{is}	=	Concentration of the internal standard
$\overline{RF}$	=	Average response factor

12.9.2 Linear Fit Calibration

If the RSD of the response factors for each compound of interest in the initial calibration is >15%, the linear fit calibration may be used for quantitation. To calculate the concentration in an unknown sample extract, the regression equation is solved for:

$$C_{ex} = \frac{\left[\frac{R_x C_{is}}{R_{is}} - b\right]}{m_1}$$

Where:

C_{ex}	=	Extract analyte concentration, µg/L
R_x	=	Response for analyte
R_{is}	=	Response for internal standard
C_{is}	=	Concentration of internal standard
b	=	y – Intercept
m_1	=	Slope

12.9.3 Quadratic Fit Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. To calculate the concentration in an unknown sample extract, the roots of the quadratic equation are solved for:

$$C_{ex} = \frac{-m_1 \pm \sqrt{(m_1)^2 - 4(m_2)\left(b - \frac{R_x C_{is}}{R_{is}}\right)}}{2m_2}$$

Where:

C_{ex}	=	Extract analyte concentration, µg/L
R_x	=	Response for analyte
R_{is}	=	Response for internal standard

C_{is} = Concentration of internal standard
m_2 = Curvature
m_1 = Slope
b = y - Intercept

12.10 Calculating the Concentration in the Sample

12.10.1 Calculation for Aqueous Samples

$$\text{Concentration, } \mu\text{g / L} = \frac{C_{ex}V_t}{V_o}$$

Where:

C_{ex} = Concentration in the extract
V_t = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then V_t = 2,000 µL.)
V_o = Volume of the sample that was extracted (mL)

12.10.2 Calculation for Sediment, Soil, Sludge, and Waste Samples

Results for sediments, sludges, and soils are usually calculated on a dry-weight basis, and for waste, on a wet-weight basis.

$$\text{Concentration, } \mu\text{g/kg} = \frac{C_{ex}V_t}{W_sD}$$

Where:

C_{ex} = Concentration in the extract
V_t = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then V_t = 2,000 µL.)
W_s = Weight of sample extracted or diluted in grams
D = (100 - % moisture in sample)/100, for a dry-weight basis or 1 for a wet-weight basis

12.11 MS/MSD Percent Recovery Calculation

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

S_{SR} = Spike sample result
S_R = Sample result
S_A = Spike added

12.12 Calculating the Relative Percent Difference (RPD) MS/MSD Pair

$$RPD = \frac{\left|MS_R - MSD_R\right|}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD = Relative percent difference
MS_R = Matrix spike result
MSD_R = Matrix spike duplicate result

12.13 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the RF calculation (12.6) with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured
A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference
RF = 1

12.14 Calculating Percent DDT Breakdown

$$\%\ \text{DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDDarea}}$$

The areas for the m/z 235 ion are used for this calculation.

12.15 Calculating the Peak Tailing Factor

$$\text{Tailing Factor} = \frac{BC}{AB}$$

Where:

Peak width (AC) is measured at 10% peak height, and divided into two line segments at the peak centroid, so that

AC = AB + BC, with
AB = left-hand segment
BC = right-hand segment

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

13.1.1 An initial MDL study must be performed on each instrument before samples can be analyzed in accordance with Policy DV-QA-005P. An MDL study is performed or verified annually or once a year to satisfy state accreditation requirements. For DoD, AFCEE, DOE and Texas TRRP projects, MDL verification is performed quarterly. MDLs are stored in the LIMS.

13.1.2 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. 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.

13.1.3 Non-standard Analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration should include the analysis of an extracted standard at the reporting limit and a single point calibration.

13.2 Demonstration of Capabilities

13.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.

13.2.2 IDOCs and on-going proficiency demonstrations are conducted as follows. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample is typically the LCS spike level. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

13.3 Training Requirements

13.3.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

14.0 Pollution Control

14.1 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.2 The LVI method utilized smaller sample sizes and extraction volumes resulting in a decrease in the amount of waste generated for the analysis of water samples.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, “Waste Management and Pollution Prevention”, of the Environmental Health and Safety Manual, and DV-HS-001P, “Waste Management Program.”

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

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Methylene Chloride – B

15.2.3 Flammable Solvent – Waste Stream C

15.2.4 Used vials – Waste Stream A

NOTE: Radioactive, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.1.1 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Capillary Column Technique, Update III, December 1996.

16.1.2 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

16.1.3 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

16.1.4 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

16.1.5 Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.

16.1.6 Method 3540C, Soxhlet Extraction, Revision 3, December 1996.

16.1.7 Method 3546, Microwave Extraction, Revision 0, February 2007.

16.1.8 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

16.1.9 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

16.1.10 Method 3580, Waste Dilution, Revision 1, July 1992.

16.1.11 Method 3620C, Florisil Cleanup, Revision 3, February 2007.

16.1.12 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

16.1.13 Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996.

16.2 40 CFR, part 136, Appendix A, “Base/Neutrals and Acids”, Method 625.

17.0 Method Modifications:

17.1 Modifications from Reference Method

17.1.1 This SOP evaluates the hardware tune based on injection of 25 ng DFTPP compared to the 50 ng DFTPP stated in Method 8270C and Method 625.

17.1.2 This procedure does not use the relative retention time units specified in the reference method since some data systems do not have this capability. Instead, retention time windows of $\pm$ 0.5 minute from the preceding continuing calibration are used for all internal standards. A comparison of retention time shifts within these windows shows that they are well within the required relative retention time window of 0.06 specified in method 8270C.

17.1.3 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.4 This procedure includes the option for weighted linear regression curves using $1/(\text{concentration})^2$ weighting factors. Section 7.5.2 of Method 8000B discusses the use of weighted least square regression based on $1/\text{standard deviation}^2$ weighting factors, which would require multiple analyses of each standard to

determine the standard deviation. IAETL has presented information to the EPA Office of Solid Waste demonstrating that the variance [(standard deviation)^2] is proportional to the standard concentration. EPA accepted this argument and issued a letter in July 1998, which authorizes the use of 1/(concentration)^2 weighting factors.

17.1.5 This procedure uses specific criteria to quantifiably evaluate the tailing factors of benzidine and pentachlorophenol (for calculations see section 12.15). Method 8270C only specifies that no peak tailing should be visible. To avoid subjectivity, no visible tailing is defined as a tailing factor of <3.0 for benzidine and <5.0 for pentachlorophenol.

17.1.6 This procedure uses final surrogate concentrations of 100 ng/µL for the acid surrogates and 100 ng/µL for base/neutral surrogates. Method 8270C specifies that 1-2 uL injections contain 200 ng of acid surrogates and 100 ng of the base/neutral surrogates (assuming 100% recovery). Based on a 0.5 uL injection, this procedure uses concentrations approximately 0.5 to 3 times those mentioned in Method 8270C.

17.1.7 The Large Volume Injection technique utilizes modifications in the source methods regarding both sample size and injection volume. These modifications are explicitly allowed by EPA. SW-846, Chapter Two lists as acceptable variations adjustment to both the sizes of sample prepared and injection volumes. See TestAmerica White Paper No. CA-Q-W-010, "Large Volume Injection (LVI) Technique for Organic Preparation and Analysis Methods" for detailed discussions. Reporting limits remain the same for this technique.

18.0 Attachments

Table 1. TestAmerica Primary Standard and Standard Reporting Limits
Table 2. TestAmerica Appendix IX Standard Reporting Limits
Table 3. TestAmerica Non-Standard Compound Reporting Limits
Table 4. Suggested Instrument Conditions
Table 5. DFTPP Key Ions and Ion Abundance Criteria
Table 6. Characteristic Ions, Primary Standard (in approximate retention time order)
Table 7. Characteristic Ions, Appendix IX Standard (in approximate retention time order)
Table 8. Characteristic Ions, Non- Standard Compounds
Table 9. 8270C Surrogate Compounds
Table 10. Calibration Levels, Primary (HSL) Standard, µg/mL
Table 11. Calibration Levels, Appendix IX Standard, µg/mL
Table 12. Calibration Levels, Non-routine Compounds, µg/mL
Table 13. Initial Demonstration Recovery and Precision Limits
Table 14. List 1 – Reliably Performing Compounds
Table 15. List 2 – Poorly Performing Compounds
Table LVI-1 LVI Calibration Levels, Primary (HSL) Standard, µg/mL
Table LVI-2 LVI Calibration Levels, Appendix IX Standard, µg/mL
Table LVI-3 LVI Calibration Levels, Non-routine Compounds, µg/mL

APPENDIX A. Modifications Required for Analysis of Wastewater Following Method 625

Table A-1. TestAmerica Method 625 Standard Reporting List and Reporting Limits

APPENDIX B. Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)

Table B-1. TestAmerica Method 8270BP Standard Reporting Limits
Table B-2. Method 8270BP Calibration Levels
Table B-3. Method 8270BP LCS Spike Concentrations
Table B-4. 8270BP Surrogate Compounds
Table B-5. 8270BP Internal Standard Compounds
Table B-6. Suggested Instrument Conditions for 8270BP

APPENDIX C. Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

19.0 Revision History

- Revision 9, dated 31 January 2014
    - Updated calibration standards and their corresponding sublists throughout the SOP (including text, tables and appendices).
        - 1,1-Biphenyl, Acetophenone, 3-Methylphenol, 1,2,4,5-Tetrachlorobenzene, 1,3-Dinitrobenzene and 2,3,4,6-Tetrachlorophenol moved into the HSL sublist
        - 4,4'-Methylene bis(2-chloroaniline), 6-Methylchrysene, Acrylamide, Dibenz(a,h)acridine, and Quinoline moved into the AP9 sublist.
    - Modified the large volume injection (LVI) injection volume to 2.5 µL and internal standard concentration to 40 µg/mL.
    - Formatting and editorial changes throughout.
- Revision 8, dated 31 January 2013
    - Added information to analyze water extracts using Large Volume Injection throughout the SOP
    - Identified surrogate and internal standard associations and standard name for non-standard compounds in Table 8
    - Added surrogate concentrations for LVI extracts in Table 9
    - Added data for calibration standards identified as Refinery, Custom, PHE, and Benzaldehyde in Table 12
    - Added Tables LVI-1, LVI-2 and LVI-3 (LVI calibration standards)
    - Revised sections 9.1, 10.1 and 10.2 to reflect current practice
    - Removed AFCEE calibration curve (no longer used)
    - Formatting and editorial changes throughout
    - Added section 9.8 for 2012 MUR QC requirements
- Revision 7, dated 28 September 2012
    - Added Table 3 TestAmerica Non-Standard Compound Reporting Limits
    - Added Table 8 Characteristic Ions, Non- Standard Compounds
    - Updated references to tables
- Revision 6, dated 9 January 2012

- Source method review
- Clarified procedural details in section 10, 11 and 12 including expanding ICV and CCV acceptance criteria for various programs
- Revised Section 12
- Updated method modifications section
- Expanded Table 6 to include IS and Surrogate compound assignments for all target analytes
- Removed Tables 7 and 8 and referenced Organic Prep SOPs and TALS reagent module for composition and concentration of LCS solutions. Renumbered remaining tables
- Removed Table A-2 and inserted reference to organic prep SOPs and TALS reagent module for composition and concentration of LCS and MS solutions for Method 625

- Revision 5.3, dated 31 May 2011
  - Annual review
  - Reorganized and updated Section 9
  - Added Section 10.8 on data review
  - Updated sections for references to current LIMS system and current practices
  - Expanded reference section to include all prep methods and Method 8000
  - Updated Section 17
  - Formatting and grammatical changes throughout
- Revision 5.2, dated 04 May 2010
  - Annual Review
  - Added section 6.9
  - Added section 12.1 referencing corporate SOP CA-Q-S-005 “Calibration Curves”
- Revision 5.1, dated 17 April 2009
  - Updated Table 8 to contain a longer list of LCS compounds
  - Corrected several references to incorrect sections
  - Removed all references to the isotope dilution method
- Revision 5, dated 20 March 2008
  - Integration for TestAmerica and STL operations
  - Revised Tables 1 and 2 to reflect current reporting limits
  - Removed the use of average from the calibration section 11.4.10
- Changes from Previous Major Revision
  - Removed the modifications for 1,4-dioxane by isotope dilution, and included this compound in Appendix B, 8270 Best Practice

Table 1.
TestAmerica Primary Standard and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
Pyridine	110-86-1	20	660
N-Nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	4	330
1,4-Dichlorobenzene	106-46-7	4	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	4	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	4	330
2-Nitroaniline	88-74-4	10	1600
Dimethyl phthalate	131-11-3	4	330
1,3-Dinitrobenzene	99-65-0	10	330
Acenaphthylene	208-96-8	4	330
3-Nitroaniline	99-09-2	10	1600
Acenaphthene	83-32-9	4	330
2,4-Dinitrophenol	51-28-5	30	1600
4-Nitrophenol	100-02-7	10	1600
Dibenzofuran	132-64-9	4	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330

Table 1 (continued)
TestAmerica Primary Standard and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
Diethylphthalate	84-66-2	4	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	4	330
4-Nitroaniline	100-01-6	10	1600
4,6-Dinitro-2-methylphenol	534-52-1	20	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	4	330
Anthracene	120-12-7	4	330
Carbazole	86-74-8	4	330
Di-n-butyl phthalate	84-74-2	4	330
Fluoranthene	206-44-0	4	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	4	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	4	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
4,4-Methylenebis(2-chloroaniline)	101-14-4	100	330
Chrysene	218-01-9	4	330
Di-n-octylphthalate	117-84-0	4	330
Benzo(b)fluoranthene	205-99-2	4	330
Benzo(k)fluoranthene	207-08-9	4	330
Benzo(a)pyrene	50-32-8	4	330
Indeno(1,2,3-cd)pyrene	193-39-5	4	330
Diethyl phthalate	84-66-2	4	660
Dibenz(a,h)anthracene	53-70-3	4	330
Benzo(g,h,i)perylene	191-24-2	4	330
Acetophenone	98-86-2	10	330
3/4-Methylphenol	15831-10-4	10	330
1,4-Dioxane	54841-74-6	20	660

1. The TAL primary standard, or HSL standard is the standard normally used at TAL. Additional standards, such as the Appendix IX standard (AP9), the Refinery standard (REF), the Custom standard (CUST), the Dinitroxylenes standard (DNX), Benzaldehyde standard (BZHD) or the phenyl standard (PHE) may be necessary to include all target analytes required for some clients.
2. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether.

Table 2.
TestAmerica Appendix IX Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	10	660
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	1600
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	50	1600
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin	297-97-2	10	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp	3689-24-5	50	1000
Phorate	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate	2303-16-4	20	660
Dimethoate	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	10	660
Methyl Parathion	298-00-0	50	1600
1-chloronaphthalene	90-13-1	10	330
Biphenyl	92-51-3	10	330
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600

Table 2. (continued)
TestAmerica Appendix IX Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
Aramite	140-57-8	20	660
Isodrin	465-73-6	10	330
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	20	660
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	10	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660
Diphenylamine	122-39-4	10	330

1. The Appendix IX standard contains additional analytes required for the Appendix IX list. The TAL primary standard must also be analyzed to include all of the Appendix IX list.
2. May also be analyzed by method 8141, which can achieve lower reporting limits.
3. May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits.

Table 3.
TestAmerica Non-Standard Compound Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
1,1'-Biphenyl	92-52-4	10	330
1,2-Dimethyl-3,4-Dinitrobenzene	603-06-5	5.0	167
1,2-Dimethyl-3,5-Dinitrobenzene	STL01265	5.0	167
1,2-Dimethyl-3,6-Dinitrobenzene	STL01263	5.0	167
1,2-Dimethyl-4,5-Dinitrobenzene	3395-03-7	5.0	167
1,3-Dimethyl-2,4-Dinitrobenzene	610-23-1	5.0	167
1,3-Dimethyl-2,5-Dinitrobenzene	STL01264	5.0	167
1,3-Phenylenediamine	108-45-2	10	660
1,4-Dimethyl-2,3-Dinitrobenzene	711-41-1	5.0	167
1,4-Dimethyl-2,6-Dinitrobenzene	STL01266	5.0	167
1,5-Dimethyl-2,3-Dinitrobenzene	65151-56-6	5.0	167
1,5-Dimethyl-2,4-Dinitrobenzene	616-72-8	5.0	167
1-Methylnaphthalene	90-12-0	4.0	330
2,3-Dichlorobenzenamine	608-27-5	10	330
2,4,6-Tribromophenol	118-79-6	5	330
2,4-Xylidine	95-68-1	10	660
2-Ethoxyethanol	110-80-5	100	3300
3,5-Dimethylphenol	108-68-9	10	330
3-Methylphenol	108-39-4	10	330
4,4-Dichlorobenzil	3457-46-3	10	330
4-Chlorobenzenethiol	106-54-7	10	330
5-Methyl-o-Anisidine	120-71-8	10	660
6-Methylchrysene	1705-85-7	10	330
Acrylamide	79-06-1	200	1600
Alachlor	15972-60-8	20	330
Alpha Methyl Styrene	98-83-9	10	330
Alpha-Terpineol	98-55-5	5.0	330
Atrazine	1912-24-9	10	330
Benzaldehyde	100-52-7	10	330
Benzo(e)pyrene	192-97-2	4	330
Benzyl dichloride	98-87-3	50	2700
Bis(4-Chlorophenyl)disulfide	1142-19-4	10	330
Caprolactam	105-60-2	10	1600
Carbofuran Phenol	1563-38-8	50	2700
Dibenz(a,h)acridine	226-36-8	10	330
Dibenzo(a,e)pyrene	192-65-4	10	1600
Dimethylformamide	68-12-2	50	330
Diphenylsulfone	127-63-9	10	330
Famphur	52-85-7	100	660
Hexachlorophene	70-30-4	1000	33000
Hexadecane	544-76-3	10	330
Indene	95-13-6	10	330
Methyl 4-Chlorophenyl Sulfide	123-09-1	10	330

Table 3. (continued)
TestAmerica Non-Standard Compound Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
Methyl Styrene	25013-15-4	10	330
n-Decane	124-18-5	10	***
n-Hydroxymethlphthalimide	118-29-6	10	330
o-Anisidine	90-04-0	10	660
Octachlorostyrene	29082-74-4	10	330
p-Chlorophenyl Sulfone	80-07-9	10	330
Perylene	198-55-0	10	1600
Phenyl Disulfide	882-33-7	10	330
Phenyl Sulfide	139-66-2	10	330
Phenylmercaptan	108-98-5	100	3300
Phthalic Acid	88-99-3	400	2500
Phthalic Anhydride	85-44-9	400	2500
Quinoline	91-22-5	50	1600
Toluene Diamine (2,4- + 2,6 – Isomers)	STL00848	1000	1600
Tributyl Phosphate	126-73-8	50	1600
Triethyl Amine	121-44-8	100	2700
Triethyl Phosphate	78-40-0	100	1600
Tris (2,3-dibromopropyl)phosphate	126-72-7	100	6600

Table 4.
Suggested Instrument Conditions

Parameter	Settings	
	Standard Analysis	**Large Volume Injection**
Mass Range:	35 - 500 amu	35-500 amu
Scan Time:	≤ 1 second/scan	≤ 1 second/scan
Initial Column Temperature/Hold Time:	40 °C for 1 minute	55 °C for 1.5 minute
Column Temperature Program:	40 - 325 °C at 25 °C/min	55 - 190 °C at 25 °C/min 190 - 330 °C at 28 °C/min
Final Column Temperature/Hold Time:	325 °C (until at least one minute after benzo(g,h,i)perylene has eluted)	330 °C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature:	250 °C	250 °C
Transfer Line Temperature:	290 °C	300 °C
Source Temperature:	According to manufacturer's specifications	According to manufacturer's specifications
Injector:	Grob-type, split / splitless	Standard shell weldment inlet; Restek liner #23303.5 or equivalent
Syringe	5 or 10 µL	5 or 10 µL
Sample Volume:	0.5 µl	2.5 µL
Carrier Gas:	Helium at 3.4 mL/min	Helium at 3.4 mL/min

Table 5.
DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	40 - 100% of mass 198
443	17 - 23% of mass 442

Table 6.
Characteristic Ions, Primary Standard (in alpha-numeric order)

Analyte	Primary	Secondary	Tertiary	IS	Surr
1,2,4,5-Tetrachlorobenzene	216	214	218	2	4
1,2,4-Trichlorobenzene	180	182	145	2	4
1,2-Dichlorobenzene	146	148	113	1	4
1,3-Dichlorobenzene	146	148	113	1	4
1,3-Dinitrobenzene	168	50	76	3	2
1,4-Dichlorobenzene	146	148	113	1	4
1,4-Dichlorobenzene-d_4 (Internal Standard)	152	150	115	1	-
1,4-Dioxane	88	58	-	1	3
2,2'-oxybis(1-chloropropane)[1]	45	77	79	1	4
2,3,4,6-Tetrachlorophenol	232	230	131	3	2
2,4,5-Trichlorophenol	196	198	200	3	1
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141	3	1
2,4,6-Trichlorophenol	196	198	200	3	1
2,4-Dichlorophenol	162	164	98	2	3
2,4-Dimethylphenol	107	121	122	2	3
2,4-Dinitrophenol	184	63	154	3	1
2,4-Dinitrotoluene	165	63	89	3	2
2,6-Dinitrotoluene	165	63	89	3	2
2-Chloronaphthalene	162	164	127	3	2
2-Chlorophenol	128	64	130	1	3
2-Fluorobiphenyl (Surrogate Standard)	172	171	170	3	2
2-Fluorophenol (Surrogate Standard)	112	64	63	1	3
2-Methylnaphthalene	142	141	115	2	2
2-Methylphenol	108	107	77	1	3
2-Nitroaniline	65	92	138	3	2
2-Nitrophenol	139	65	109	2	3
3,3'-Dichlorobenzidine	252	254	126	5	2
3/4-Methylphenol	108	107	77	1	3
3-Nitroaniline	138	108	92	3	2
4,6-Dinitro-2-methylphenol	198	105	51	4	1
4-Bromophenylphenylether	248	250	141	4	2
4-Chloro-3-methylphenol	107	144	142	2	1
4-Chloroaniline	127	129	65	2	4
4-Chlorophenylphenylether	204	206	141	3	2
4-Methylphenol	108	107	79	1	3
4-Nitroaniline	138	92	108	3	2
4-Nitrophenol	109	139	65	3	1
Acenaphthene	153	152	154	3	6
Acenaphthene-d_{10} (Internal Standard)	164	162	160	3	-
Acenaphthylene	152	151	153	3	6
Acetophenone	105	77	120	1	3
Aniline	93	66	-	1	3
Anthracene	178	179	176	4	6
Azobenzene	77	182	105	3	2
Benzidine	184	92	185	5	6

Table 6. (continued)
Characteristic Ions, Primary Standard (in alpha-numeric order)

Analyte	Primary	Secondary	Tertiary	IS	Surr
Benzo(a)anthracene	228	229	226	5	6
Benzo(a)pyrene	252	253	125	6	6
Benzo(b)fluoranthene	252	253	125	6	6
Benzo(g,h,i)perylene	276	138	277	6	6
Benzo(k)fluoranthene	252	253	125	6	6
Benzoic Acid	122	105	77	2	3
Benzyl Alcohol	108	79	77	1	4
Bis(2-chloroethoxy)methane	93	95	123	2	4
Bis(2-chloroethyl)ether	93	63	95	1	4
Bis(2-ethylhexyl)phthalate	149	167	279	5	2
Butylbenzylphthalate	149	91	206	5	2
Carbazole	167	166	139	4	1
Chrysene	228	226	229	5	6
Chrysene-d_{12} (Internal Standard)	240	120	236	5	-
Dibenz(a,h)anthracene	278	139	279	6	2
Dibenzofuran	168	139	84	3	2
Diethylphthalate	149	177	150	3	2
Dimethylphthalate	163	194	164	3	2
Di-n-butylphthalate	149	150	104	4	2
Di-n-octylphthalate	149	167	43	5	2
Famphur	218	93	125	5	6
Fluoranthene	202	101	100	4	6
Fluorene	166	165	167	3	6
Hexachlorobenzene	284	142	249	4	2
Hexachlorobutadiene	225	223	227	2	4
Hexachlorocyclopentadiene	237	235	271	3	2
Hexachloroethane	117	201	199	1	4
Indeno(1,2,3-cd)pyrene	276	138	277	5	6
Isophorone	82	95	138	2	4
Naphthalene	128	129	127	2	6
Naphthalene-d_8 (Internal Standard)	136	68	54	2	-
Nitrobenzene	77	123	65	2	4
Nitrobenzene-d_5 (Surrogate Standard)	82	128	54	2	4
N-Nitrosodimethylamine	74	42	-	1	3
N-Nitroso-di-n-propylamine	70	42	101,130	1	4
N-Nitrosodiphenylamine	169	168	167	4	2
Pentachlorophenol	266	264	268	4	1
Perylene-d_{12} (Internal Standard)	264	260	265	6	-
Phenanthrene	178	179	176	4	6
Phenanthrene-d_{10} (Internal Standard)	188	94	80	4	-
Phenol	94	65	66	1	5
Phenol-d_5 (Surrogate Standard)	99	42	71	1	5
Pyrene	202	101	100	5	6
Pyridine	79	52	-	1	3
Terphenyl-d_{14} (Surrogate Standard)	244	122	212	5	6

1. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether.

Table 7.
Characteristic Ions, Appendix IX Standard (in alpha-numeric order)

Analyte	Primary	Secondary	Tertiary	IS	SURR
1,3,5-Trinitrobenzene	213	75	120	4	--
1,4-Dinitrobenzene	168	75	122	3	2
1,4-Naphthoquinone	158	104	102	3	2
1-Naphthylamine	143	115	--	3	2
2,6-Dichlorophenol	162	164	63	2	4
2-Acetylaminofluorene	181	180	223	5	6
2-Naphthylamine	143	115	--	3	2
2-Picoline	93	66	92	1	3
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147	4	--
3,3'-Dimethylbenzidine	212	106	--	5	6
3-Methylcholanthrene	268	252	253	6	6
4-Aminobiphenyl	169	168	115	4	--
4-Nitroquinoline-1-oxide	190	128	160	4	--
5-Nitro-o-toluidine	152	77	106	3	2
7,12-Dimethylbenz(a)anthracene	256	241	120	6	6
a,a-Dimethyl-phenethylamine	58	91	--	2	4
Aramite 1	185	319	--	5	6
Aramite 2	185	319	--	5	6
Diallate	86	234	--	4	--
Dibenz(a,j)acridine	279	280	--	6	6
Dimethoate	87	93	125	4	--
Disulfoton	88	97	89	4	--
Ethyl methanesulfonate	79	109	97	1	3
Hexachloropropene	213	215	211	2	4
Isodrin	193	66	195	4	--
Isosafrole 1	162	104	131	3	2
Isosafrole 2	162	104	131	3	2
Methapyrilene	97	58	--	4	--
Methyl methanesulfonate	80	79	65	1	3
Methyl parathion	109	125	263	4	--
N-Nitrosodiethylamine	102	44	57	1	3
N-Nitrosodi-n-butylamine	84	57	41	2	4
N-Nitrosomethylethylamine	88	42	43	1	3
N-Nitrosomorpholine	116	56	86	1	3
N-Nitrosopiperidine	114	42	55	2	4
N-Nitrosopyrrolidine	100	41	42	1	3
O,O',O"-Triethyl-Phosphorothioate	198	121	93	2	4

Table 7. (continued)
Characteristic Ions, Appendix IX Standard (in alpha-numeric order)

Analyte	Primary	Secondary	Tertiary	IS	SURR
o-Toluidine	106	107	77	1	3
p-(Dimethylamino)azobenzene	120	225	77	5	--
Parathion	109	97	291	4	--
p-Chlorobenzilate	251	139	253	5	6
Pentachlorobenzene	250	248	252	3	2
Pentachloroethane	117	119	167	1	3
Pentachloronitrobenzene	237	142	214	4	--
Phenacetin	108	179	109	4	--
Phorate	75	97	121	4	--
p-Phenylenediamine	108	80	54	2	6
Pronamide	173	175	255	4	--
Safrole	162	104	77	2	4
Sulfotepp	97	322	202	4	--
Thionazin	97	96	143	3	2

Table 8.
Characteristic Ions, Non-Standard Compounds[1]

Analyte	Primary	Secondary	Tertiary	IS	SURR	Curve ID
1,1'-Biphenyl	154	153	76	3	2	HSL
1,2-Dimethyl-3,4-Dinitrobenzene	179	91	77	4	--	DNX
1,2-Dimethyl-3,5-Dinitrobenzene	179	77	103	4	--	DNX
1,2-Dimethyl-3,6-Dinitrobenzene	179	77	133	3	--	DNX
1,2-Dimethyl-4,5-Dinitrobenzene	91	196	77	4	--	DNX
1,3-Dimethyl-2,4-Dinitrobenzene	77	179	91	3	--	DNX
1,3-Dimethyl-2,5-Dinitrobenzene	179	77	104	3	--	DNX
1,3-Phenylenediamine	108	80	81	1	2	PHE
1,4-Dimethyl-2,3-Dinitrobenzene	77	91	179	3	--	DNX
1,4-Dimethyl-2,6-Dinitrobenzene	179	77	91	3	--	DNX
1,5-Dimethyl-2,3-Dinitrobenzene	179	91	77	4	--	DNX
1,5-Dimethyl-2,4-Dinitrobenzene	179	77	91	3	--	DNX
1-Chloronaphthalene	162	127	164	3	2	AP9
1-Methylnaphthalene	142	141	115	2	4	HSL
2,3-Dichlorobenzenamine	161	163	90	3	2	HSL
2,4,6-Tribromophenol	329.9	331.9	141	3	1	HSL
2,4-Xylidine	121	120	106	2	4	PHE
2-Ethoxyethanol	59	45	72	1	3	CUST
3,5-Dimethylphenol	107	122	121	2	--	HSL
3-Methylphenol	108	107	77	1	3	HSL
4,4-Dichlorobenzil	139	141	111	--	--	BRC
4,4-Methylene bis(2-Chloroaniline)	231	266	238	5	6	AP9
4-Chlorobenzenethiol	144	146	109	--	--	BRC
5-Methyl-o-Anisidine	122	137	94	1	3	PHE
6-Methylchrysene	242	241	239	5	6	AP9
Acrylamide	71	54.9	44	1	3	AP9
Alachlor	188	160	45	1	--	HSL
Alpha Methyl Styrene	118	117	103	1	3	HSL
Alpha-Terpineol	59	93	121	1	--	REF
Atrazine	200	58	43	4	--	HSL
Benzaldehyde	106	105	77	1	--	BZHD
Benzo(e)pyrene	252	250	253	6	--	HSL
Benzyl dichloride	124.9	159.9	89	2	3	CUST
Bis(4-Chlorophenyl)disulfide	143	145	108	--	--	BRC
Caprolactam	55	113	84	2	4	HSL
Carbofuran Phenol	164	121.9	123	2	4	CUST
Dibenz(a,h)acridine	279	280	278	6	6	AP9
Dibenzo(a,e)pyrene	302	150	151	6	6	REF
Dimethylformamide	73	44	42	1	3	CIST

[1] Some non-routine analytes are not contained in the standard mixes. Appropriate calibrations are performed when these analytes are requested, along with assignment of IS and surrogate compounds at time of analysis.

Table 8. (continued)
Characteristic Ions, Non-Standard Compounds[1]

Analyte	Primar	Secondary	Tertiary	IS	SURR	Curve ID
Diphenylamine	169	168	167	3	2	AP9
Diphenylsulfone	125	77	51	--	--	BRC
Famphur	218	93	125	5	6	HSL
Hexachlorophene	196	198		5	6	HSL
Hexadecane	57	43	71	3	2	HSL
Indene	116	115	89	1	3	HSL
Isosafrole	162	131	104	3	2	AP9
Methyl 4-Chlorophenyl Sulfide	158	160	108	--	--	BRC
Methyl Styrene	118	117	--	--	--	--
n-Decane	43	57	71	1	2	HSL
n-Hydroxymethlphthalimide	147	104	76	--	--	HSL
o-Anisidine	80	108	123	1	3	PHE
Octachlorostyrene	308	378	343	--	--	BRC
p-Chlorophenyl Sulfone	159	161	111	--	--	BRC
Perylene	252	253	126	6	6	CUST
Phenyl Disulfide	109	218	154	--	--	BRC
Phenyl Sulfide	186	185	184	--	--	BRC
Phenylmercaptan	110	77	109	1	3	REF
Phthalic Acid	148	149	76	3	2	REF
Phthalic Anhydride	76	104	50	3	2	REF
Quinoline	129	102	128	2	4	AP9
Toluene Diamine (2,4- + 2,6 –Isomers)	122	121	94	3	2	CUST
Tributyl Phosphate	99	155	211	3	--	CUST
Triethyl Amine	85.9	57.9	101	1	3	CUST
Triethyl Phosphate	99	155	127	2	4	CUST
Tris (2,3-dibromopropyl)phosphate	119	121	219	6	6	CUST

[1] Some non-routine analytes are not contained in the standard mixes. Appropriate calibrations are performed when these analytes are requested, along with assignment of IS and surrogate compounds at time of analysis.

Table 9.
8270C Surrogate Compounds

Surrogate Compounds	Standard Analysis ng/µL in extract	LVI Analysis ng/µL in extract
Nitrobenzene-d_5	100	25
2-Fluorobiphenyl	100	25
Terphenyl-d_{14}	100	25
1,2-Dichlorobenzene-d_4[1]	100	25
Phenol-d_5	100	25
2-Fluorophenol	100	25
2,4,6-Tribromophenol	100	25
2-Chlorophenol-d_4[1]	100	25

[1] Included in standard mix, but not evaluated or reported.
Recovery limits for surrogates are generated from historical data and are maintained by the QA department and stored in the LIMS

Table 10.
Calibration Levels[1], Primary (HSL) Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Pyridine	-	10	20	50	80	120	160	200
1,4-Dioxane	4	10	20	50	80	120	160	200
N-nitrosodimethylamine	-	10	20	50	80	120	160	200
Aniline	-	10	20	50	80	120	160	200
Phenol	-	10	20	50	80	120	160	200
Bis(2-chloroethyl)ether	-	10	20	50	80	120	160	200
2-Chlorophenol	-	10	20	50	80	120	160	200
1,3-Dichlorobenzene	4	10	20	50	80	120	160	200
1,4-Dichlorobenzene	4	10	20	50	80	120	160	200
Benzyl alcohol	-	10	20	50	80	120	160	200
1,2-Dichlorobenzene	4	10	20	50	80	120	160	200
2-Methylphenol	-	10	20	50	80	120	160	200
2,2'-oxybis(1-chloropropane)[2]	-	10	20	50	80	120	160	200
4-Methylphenol	-	10	20	50	80	120	160	200
3-Methylphenol	-	10	20	50	80	120	160	200
Acetophenone	4	10	20	50	80	120	160	200
N-Nitroso-di-n-propylamine	-	10	20	50	80	120	160	200
Hexachloroethane	4	10	20	50	80	120	160	200
Nitrobenzene	-	10	20	50	80	120	160	200
Isophorone	-	10	20	50	80	120	160	200
2-Nitrophenol	-	10	20	50	80	120	160	200
2,4-Dimethylphenol	-	10	20	50	80	120	160	200
Benzoic acid	-	--	20	50	80	120	160	200
Bis(2-chloroethoxy)methane	-	10	20	50	80	120	160	200
2,4-Dichlorophenol	-	10	20	50	80	120	160	200
1,2,4-Trichlorobenzene	4	10	20	50	80	120	160	200
Naphthalene	4	10	20	50	80	120	160	200
4-Chloroaniline	-	10	20	50	80	120	160	200
Hexachlorobutadiene	-	10	20	50	80	120	160	200
1,2,4,5-Tetrachlorobenzene	-	10	20	50	80	120	160	200
4-Chloro-3-methylphenol	-	10	20	50	80	120	160	200
2-Methylnaphthalene	4	10	20	50	80	120	160	200
Hexachlorocyclopentadiene	-	--	20	50	80	120	160	200
2,4,6-Trichlorophenol	-	10	20	50	80	120	160	200
2,4,5-Trichlorophenol	-	10	20	50	80	120	160	200
2-Chloronaphthalene	4	10	20	50	80	120	160	200
2-Nitroaniline	-	--	20	50	80	120	160	200
Dimethyl phthalate	4	10	20	50	80	120	160	200
1,3-Dinitrobenzene	-	10	20	50	80	120	160	200
Acenaphthylene	4	10	20	50	80	120	160	200
3-Nitroaniline	-	--	20	50	80	120	160	200
Acenaphthene	4	10	20	50	80	120	160	200
2,4-Dinitrophenol	-	20	40	100	160	240	320	400
4-Nitrophenol	8	20	40	100	160	240	320	400
Dibenzofuran	4	10	20	50	80	120	160	200
2,3,4,6-Tetrachlorophenol	-	10	20	50	80	120	160	200
2,4-Dinitrotoluene	-	10	20	50	80	120	160	200
2,6-Dinitrotoluene	-	10	20	50	80	120	160	200

Table 10. (continued)
Calibration Levels, Primary (HSL) Standard, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8
Diethylphthalate	4	10	20	50	80	120	160	200
4-Chlorophenyl phenyl ether	-	10	20	50	80	120	160	200
Fluorene	4	10	20	50	80	120	160	200
4-Nitroaniline	-	--	20	50	80	120	160	200
4,6-Dinitro-2-methylphenol	-	20	40	100	160	240	320	400
N-Nitrosodiphenylamine	-	10	20	50	80	120	160	200
Azobenzene[3]	4	10	20	50	80	120	160	200
4-Bromophenyl phenyl ether	-	10	20	50	80	120	160	200
Hexachlorobenzene	-	10	20	50	80	120	160	200
Pentachlorophenol	-	20	40	100	160	240	320	400
Phenanthrene	4	10	20	50	80	120	160	200
Anthracene	4	10	20	50	80	120	160	200
Carbazole	4	10	20	50	80	120	160	200
Di-n-butyl phthalate	4	10	20	50	80	120	160	200
Fluoranthene	4	10	20	50	80	120	160	200
Benzidine	-	--	20	50	80	120	160	200
Pyrene	4	10	20	50	80	120	160	200
Butyl benzyl phthalate	4	10	20	50	80	120	160	200
3,3'-Dichlorobenzidine	-	--	20	50	80	120	160	200
Benzo(a)anthracene	4	10	20	50	80	120	160	200
Bis(2-ethylhexyl)phthalate	4	10	20	50	80	120	160	200
Chrysene	4	10	20	50	80	120	160	200
Di-n-octylphthalate	4	10	20	50	80	120	160	200
Benzo(b)fluoranthene	4	10	20	50	80	120	160	200
Benzo(k)fluoranthene	4	10	20	50	80	120	160	200
Benzo(a)pyrene	4	10	20	50	80	120	160	200
Indeno(1,2,3-cd)pyrene	4	10	20	50	80	120	160	200
Dibenz(a,h)anthracene	4	10	20	50	80	120	160	200
Benzo(g,h,i)perylene	4	10	20	50	80	120	160	200

1. All compounds are spiked in all standard levels (4, 10, 20, 50, 80, 120, 160 and 200 µg/mL). Levels listed in this table are the required spike levels needed in order to calibrate to at the reporting limit for each compound. Additional lower-level standards may be included in the calibrations as long as the ICAL criteria are met.
2. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
3. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 11.
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2-Picoline	10	20	50	80	120	160	200
N-Nitrosomethylethylamine	10	20	50	80	120	160	200
Methyl methanesulfonate	10	20	50	80	120	160	200
N-Nitrosodiethylamine	10	20	50	80	120	160	200
Ethyl methanesulfonate	10	20	50	80	120	160	200
Pentachloroethane	--	20	50	80	120	160	200
N-Nitrosopyrrolidine	10	20	50	80	120	160	200
N-Nitrosomorpholine	10	20	50	80	120	160	200
o-Toluidine	10	20	50	80	120	160	200
N-Nitrosopiperidine	10	20	50	80	120	160	200
O,O',O"-Triethyl-Phosphorothioate	--	20	50	80	120	160	200
a,a-Dimethyl-phenethylamine	--	20	50	80	120	160	200
2,6-Dichlorophenol	10	20	50	80	120	160	200
Hexachloropropene	--	20	50	80	120	160	200
p-Phenylenediamine	--	20	50	80	120	160	200
n-Nitrosodi-n-butylamine	10	20	50	80	120	160	200
Safrole	--	20	50	80	120	160	200
Isosafrole 1 + 2	10	20	50	80	120	160	200
1,4-Dinitrobenzene	10	20	50	80	120	160	200
1,4-Naphthoquinone	--	20	50	80	120	160	200
Pentachlorobenzene	10	20	50	80	120	160	200
1-Naphthylamine	10	20	50	80	120	160	200
2-Naphthylamine	10	20	50	80	120	160	200
N-Nitro-o-toluidine	10	20	50	80	120	160	200
Thionazin	10	20	50	80	120	160	200
1,3,5-Trinitrobenzene	--	20	50	80	120	160	200
Sulfotepp	--	20	50	80	120	160	200
Phorate	--	20	50	80	120	160	200
Phenacetin	10	20	50	80	120	160	200
Diallate 1 + 2	10	20	50	80	120	160	200
Dimethoate	10	20	50	80	120	160	200
4-Aminobiphenyl	--	20	50	80	120	160	200
Pentachloronitrobenzene	--	20	50	80	120	160	200
Pronamide	10	20	50	80	120	160	200
Disulfoton	--	20	50	80	120	160	200
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	20	50	80	120	160	200
Methyl parathion	--	20	50	80	120	160	200
4-Nitroquinoline-1-oxide	--	20	50	80	120	160	200
Parathion	--	20	50	80	120	160	200

Table 11. (continued)
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Isodrin	10	20	50	80	120	160	200
Methapyrilene	--	20	50	80	120	160	200
Aramite 1 and 2	10	20	50	80	120	160	200
p-(Dimethylamino) azobenzene	10	20	50	80	120	160	200
p-Chlorobenzilate	10	20	50	80	120	160	200
3,3'-Dimethylbenzidine	10	20	50	80	120	160	200
2-Acetylaminofluorene	--	20	50	80	120	160	200
Dibenz (a,j)acridine	10	20	50	80	120	160	200
7,12-Dimethylbenz(a) anthracene	10	20	50	80	120	160	200
3-Methylcholanthrene	10	20	50	80	120	160	200

Table 12.
Calibration Levels, Nonroutine compounds, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8	CURVE ID
1,1'-Biphenyl	--	10	20	50	80	120	160	200	HSL
1,2-Dimethyl-3,4-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,2-Dimethyl-3,5-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,2-Dimethyl-3,6-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,2-Dimethyl-4,5-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,3-Dimethyl-2,4-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,3-Dimethyl-2,5-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,3-Phenylenediamine	--	10	20	50	80	120	160	200	PHE
1,4-Dimethyl-2,3-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,4-Dimethyl-2,6-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,5-Dimethyl-2,3-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,5-Dimethyl-2,4-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1-Chloronaphthalene	--	10	20	50	80	120	160	200	AP9
1-Methylnaphthalene	4	10	20	50	80	120	160	200	HSL
2,3-Dichlorobenzenamine	4	10	20	50	80	120	160	200	HSL
2,4,6-Tribromophenol	4	10	20	50	80	120	160	200	HSL
2,4-Xylidine	--	10	20	50	80	120	160	200	PHE
2-Ethoxyethanol	--	10	20	50	80	120	160	200	CUST
3,5-Dimethylphenol	4	10	20	50	80	120	160	200	HSL
3-Methylphenol	4	10	20	50	80	120	160	200	HSL
4,4-Dichlorobenzil	--	10	20	50	80	120	160	200	BRC
4,4-Methylene bis(2-Chloroaniline)	--	10	20	50	80	120	160	200	AP9
4-Chlorobenzenethiol	--	10	20	50	80	120	160	200	BRC
5-Methyl-o-Anisidine	--	10	20	50	80	120	160	200	PHE

Table 12. (continued)
Calibration Levels, Nonroutine compounds, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8	CURVE ID
6-Methylchrysene	--	10	20	50	80	120	160	200	AP9
Acrylamide	--	10	20	50	80	120	160	200	AP9
Alachlor	4	10	20	50	80	120	160	200	HSL
Alpha Methyl Styrene	4	10	20	50	80	120	160	200	HSL
Alpha-Terpineol	--	10	20	50	80	120	160	200	REF
Atrazine	4	10	20	50	80	120	160	200	HSL
Benzaldehyde	--	10	20	50	80	120	160	200	BZHD
Benzo(e)pyrene	4	10	20	50	80	120	160	200	HSL
Benzyl dichloride	--	10	20	50	80	120	160	200	CUST
Bis(4-Chlorophenyl)disulfide	--	10	20	50	80	120	160	200	BRC
Caprolactam	4	10	20	50	80	120	160	200	HSL
Carbofuran Phenol	--	10	20	50	80	120	160	200	CUST
Dibenz(a,h)acridine	--	10	20	50	80	120	160	200	AP9
Dibenzo(a,e)pyrene	--	10	20	50	80	120	160	200	REF
Dimethylformamide	--	10	20	50	80	120	160	200	CUST
Diphenylamine	--	10	20	50	80	120	160	200	AP9
Diphenylsulfone	--	10	20	50	80	120	160	200	BRC
Famphur	4	10	20	50	80	120	160	200	HSL
Hexachlorophene	4	10	20	50	80	120	160	200	HSL
Hexadecane	4	10	20	50	80	120	160	200	HSL
Indene	4	10	20	50	80	120	160	200	HSL
Isosafrole	--	10	20	50	80	120	160	200	AP9
Methyl 4-Chlorophenyl Sulfide	--	10	20	50	80	120	160	200	BRC
n-Decane	4	10	20	50	80	120	160	200	HSL
n-Hydroxymethlphthalimide	4	10	20	50	80	120	160	200	HSL
o-Anisidine	--	10	20	50	80	120	160	200	PHE
Octachlorostyrene	--	10	20	50	80	120	160	200	BRC
p-Chlorophenyl Sulfone	--	10	20	50	80	120	160	200	BRC
Perylene	--	10	20	50	80	120	160	200	CUST
Phenyl Disulfide	--	10	20	50	80	120	160	200	BRC
Phenyl Sulfide	--	10	20	50	80	120	160	200	BRC
Phenylmercaptan	--	10	20	50	80	120	160	200	REF
Phthalic Acid	--	10	20	50	80	120	160	200	REF
Phthalic Anhydride	--	10	20	50	80	120	160	200	REF
Quinoline	--	10	20	50	80	120	160	200	AP9
Toluene Diamine (2,4- + 2,6 –	--	10	20	50	80	120	160	200	CUST
Tributyl Phosphate	--	10	20	50	80	120	160	200	CUST
Triethyl Amine	--	10	20	50	80	120	160	200	CUST
Triethyl Phosphate	--	10	20	50	80	120	160	200	CUST
Tris (2,3-dibromopropyl)phosphate	--	10	20	50	80	120	160	200	CUST

Table 13.
Initial Demonstration Recovery and Precision Limits

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9

Table 13. (continued)
Initial Demonstration Recovery and Precision Limits

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

1. Organochlorine pesticides and PCBs project DQOs generally require better sensitivity than is provided by 8270C, so methods 8081 and 8082 are used instead. These compounds will not be included in the initial demonstration of capability for method 8270C.

Table 14.
List 1 – Reliably Performing Compounds

Acenaphthene	Dibenzofuran	1H-Indene
Acenaphthylene	1,4-Dioxane	Indeno(1,2,3-cd)pyrene
Acetophenone	n-Dodecane	Isophorone
Alachlor	n-Docosane	1-Methylnaphthalene
Aniline	1,2-Dichlorobenzene	2-Methylnaphthalene
Anthracene	1,3-Dichlorobenzene	2-Methylphenol
Atrazine	1,4-Dichlorobenzene	4-Methylphenol
Benzo(a)anthracene	2,3-Dichlorobenzeneamine	Methylstyrene
Benzo(a)pyrene	3,3'-Dichlorobenzidine	Naphthalene
Benzo(b)fluoranthene	2,4-Dichlorophenol	2-Nitroaniline
Benzo(k)fluoranthene	Diethyl phthalate	3-Nitroaniline
Benzo(g,h,i)perylene	2,4-Dimethylphenol	4-Nitroaniline
Benzoic acid	Dimethyl phthalate	Nitrobenzene
Benzyl alcohol	Di-n-butyl phthalate	2-Nitrophenol
Bis(2-chloroethoxy)methane	4,6-Dinitro-2-methylphenol	4-Nitrophenol
Bis(2-chloroethyl)ether	2,4-Dinitrophenol	N-Nitrosodimethylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	N-Nitrosodiphenylamine
Butyl benzyl phthalate	1,2-Diphenylhydrazine (as Azobenzene)	2,2'-Oxybis(1-chloropropane) aka "bis(2-chloroisopropyl) ether"
Caprolactam	Di-n-octyl phthalate	n-Octadecane
Carbazole	n-Eicosane	Pentachlorophenol
4-Chloroaniline	Famphur	Phenanthrene
4-Chloro-3-methylphenol	Fluoranthene	Phenol
2-Chloronaphthalene	Fluorene	Pyrene
2-Chlorophenol	Hexachlorobenzene	Pyridine
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	n-Tetradecane
Chrysene	Hexachlorobutadiene	1,2,4-Trichlorobenzene
n-Decane	Hexachloroethane	2,4,5-Trichlorophenol
Dibenz(a,h)anthracene	n-Hexadecane	2,4,6-Trichlorophenol

Table 15.
List 2 – Poorly Performing Compounds

2-Acetylaminofluorene	Diphenylamine	N-Nitrosopyrrolidine
Acrylamide	Disulfoton	Parathion
4-Aminobiphenyl	2-Ethoxyethanol	Pentachlorobenzene
Aramite (#1)	Ethyl methanesulfonate	Pentachloroethane
Aramite (#2)	Hexachlorophene	Pentachloronitrobenzene
Benzenethiol	Hexachloropropene	Perylene
Benzidine	Isosafrole (#1)	Phenacetin
Benzyl chloride	Isosafrole (#2)	p-Phenylenediamine
Biphenyl	Isodrin	Phorate
Carbofuran phenol	Methapyrilene	Phthalic anhydride
Chlorobenzilate	Methomyl	2-Picoline
Diallate (#1)	3-Methylcholanthrene	Pronamide
Diallate (#2)	6-Methylchrysene	Quinoline
Dibenz(a,h)acridine	4,4''-Methylenebis(2-chloroaniline)	Safrole
Dibenz(a,j)acridine	Methyl methanesulfonate	2-secbutyl-4,6-dinitrophenol (Dinoseb)
Dibenzo(a,e)pyrene	Methyl Parathion	Sulfotepp
Tris(2,3-Dibromopropyl) phosphate	1-Naphthylamine	1,2,4,5-Tetrachlorobenzene
2,6-Dichlorophenol	2-Naphthylamine	2,3,4,6-Tetrachlorophenol
Dimethoate	1,4-Naphthoquinone	Thionazin
p-(Dimethylamino)azobenzene	5-Nitro-o-toluidine	o-Toluidine
7,12-Dimethylbenz(a)anthracene	4-Nitroquinoline-1-oxide	2,4- and 2,6-Toluenediamine
3,3'-Dimethylbenzidine	N-Nitrosodiethylamine	Triethylamine
N,N-Dimtheylformamide	n-Nitrosodi-n-butylamine	Triethylphosphate
a,a-Dimethyl-phenethylamine	N-Nitrosomethylethylamine	o,o,o-Triethylphosphorothioate
1,3-Dinitrobenzene	N-Nitrosomorpholine	1,3,5-Trinitrobenzene
1,4-Dinitrobenzene	N-Nitrosopiperidine	

Table LVI-1.
LVI Calibration Levels, Primary (HSL) Standard, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8
Pyridine	1	2.5	5	12.5	20	30	40	50
N-nitrosodimethylamine	1	2.5	5	12.5	20	30	40	50
Aniline	1	2.5	5	12.5	20	30	40	50
Phenol	1	2.5	5	12.5	20	30	40	50
Bis(2-chloroethyl)ether	1	2.5	5	12.5	20	30	40	50
2-Chlorophenol	1	2.5	5	12.5	20	30	40	50
1,3-Dichlorobenzene	1	2.5	5	12.5	20	30	40	50
1,4-Dichlorobenzene	1	2.5	5	12.5	20	30	40	50
Benzyl alcohol	1	2.5	5	12.5	20	30	40	50
1,2-Dichlorobenzene	1	2.5	5	12.5	20	30	40	50
2-Methylphenol	1	2.5	5	12.5	20	30	40	50
2,2'-oxybis(1-chloropropane)[1]	1	2.5	5	12.5	20	30	40	50
4-Methylphenol	1	2.5	5	12.5	20	30	40	50
3-Methylphenol	1	2.5	5	12.5	20	30	40	50
Acetophenone	1	2.5	5	12.5	20	30	40	50
N-Nitroso-di-n-propylamine	1	2.5	5	12.5	20	30	40	50
Hexachloroethane	1	2.5	5	12.5	20	30	40	50
Nitrobenzene	1	2.5	5	12.5	20	30	40	50
Isophorone	1	2.5	5	12.5	20	30	40	50
2-Nitrophenol	1	2.5	5	12.5	20	30	40	50
2,4-Dimethylphenol	1	2.5	5	12.5	20	30	40	50
Benzoic acid	2	5	10	25	40	60	80	100
Bis(2-chloroethoxy)methane	1	2.5	5	12.5	20	30	40	50
2,4-Dichlorophenol	1	2.5	5	12.5	20	30	40	50
1,2,4-Trichlorobenzene	1	2.5	5	12.5	20	30	40	50
Naphthalene	1	2.5	5	12.5	20	30	40	50
4-Chloroaniline	1	2.5	5	12.5	20	30	40	50
Hexachlorobutadiene	1	2.5	5	12.5	20	30	40	50
4-Chloro-3-methylphenol	1	2.5	5	12.5	20	30	40	50
2-Methylnaphthalene	1	2.5	5	12.5	20	30	40	50
Hexachlorocyclopentadiene	1	2.5	5	12.5	20	30	40	50
1,2,4,5-Tetrachlorobenzene	1	2.5	5	12.5	20	30	40	50
2,4,6-Trichlorophenol	1	2.5	5	12.5	20	30	40	50
2,4,5-Trichlorophenol	1	2.5	5	12.5	20	30	40	50
2-Chloronaphthalene	1	2.5	5	12.5	20	30	40	50
2-Nitroaniline	1	2.5	5	12.5	20	30	40	50
Dimethyl phthalate	1	2.5	5	12.5	20	30	40	50
1,3-Dinitrobenzene	1	2.5	5	12.5	20	30	40	50
Acenaphthylene	1	2.5	5	12.5	20	30	40	50
3-Nitroaniline	1	2.5	5	12.5	20	30	40	50
Acenaphthene	1	2.5	5	12.5	20	30	40	50
2,4-Dinitrophenol	2	5	10	25	40	60	80	100
4-Nitrophenol	2	5	10	25	40	60	80	100
Dibenzofuran	1	2.5	5	12.5	20	30	40	50
2,3,4,6-Tetrachlorophenol	1	2.5	5	12.5	20	30	40	50
2,4-Dinitrotoluene	1	2.5	5	12.5	20	30	40	50
2,6-Dinitrotoluene	1	2.5	5	12.5	20	30	40	50

Table LVI-1. (continued)
LVI Calibration Levels, Primary Standard, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8
Diethylphthalate	1	2.5	5	12.5	20	30	40	50
4-Chlorophenyl phenyl ether	1	2.5	5	12.5	20	30	40	50
Fluorene	1	2.5	5	12.5	20	30	40	50
4-Nitroaniline	1	2.5	5	12.5	20	30	40	50
4,6-Dinitro-2-methylphenol	2	5	10	25	40	60	80	100
N-Nitrosodiphenylamine	1	2.5	5	12.5	20	30	40	50
Azobenzene[2]	1	2.5	5	12.5	20	30	40	50
4-Bromophenyl phenyl ether	1	2.5	5	12.5	20	30	40	50
Hexachlorobenzene	1	2.5	5	12.5	20	30	40	50
Pentachlorophenol	2	5	10	25	40	60	80	100
Phenanthrene	1	2.5	5	12.5	20	30	40	50
Anthracene	1	2.5	5	12.5	20	30	40	50
Carbazole	1	2.5	5	12.5	20	30	40	50
Di-n-butyl phthalate	1	2.5	5	12.5	20	30	40	50
Fluoranthene	1	2.5	5	12.5	20	30	40	50
Benzidine	1	2.5	5	12.5	20	30	40	50
Pyrene	1	2.5	5	12.5	20	30	40	50
Butyl benzyl phthalate	1	2.5	5	12.5	20	30	40	50
3,3'-Dichlorobenzidine	1	2.5	5	12.5	20	30	40	50
Benzo(a)anthracene	1	2.5	5	12.5	20	30	40	50
Bis(2-ethylhexyl)phthalate	1	2.5	5	12.5	20	30	40	50
Chrysene	1	2.5	5	12.5	20	30	40	50
Di-n-octylphthalate	1	2.5	5	12.5	20	30	40	50
Benzo(b)fluoranthene	1	2.5	5	12.5	20	30	40	50
Benzo(k)fluoranthene	1	2.5	5	12.5	20	30	40	50
Benzo(a)pyrene	1	2.5	5	12.5	20	30	40	50
Indeno(1,2,3-cd)pyrene	1	2.5	5	12.5	20	30	40	50
Dibenz(a,h)anthracene	1	2.5	5	12.5	20	30	40	50
Benzo(g,h,i)perylene	1	2.5	5	12.5	20	30	40	50

1. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
2. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table LVI-2.
LVI Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2-Picoline	2.5	5	12.5	20	30	40	50
N-Nitrosomethylethylamine	2.5	5	12.5	20	30	40	50
Methyl methanesulfonate	2.5	5	12.5	20	30	40	50
N-Nitrosodiethylamine	2.5	5	12.5	20	30	40	50
Ethyl methanesulfonate	2.5	5	12.5	20	30	40	50
Pentachloroethane	2.5	5	12.5	20	30	40	50
N-Nitrosopyrrolidine	2.5	5	12.5	20	30	40	50
N-Nitrosomorpholine	2.5	5	12.5	20	30	40	50
o-Toluidine	2.5	5	12.5	20	30	40	50
N-Nitrosopiperidine	2.5	5	12.5	20	30	40	50
O,O',O"-Triethyl-Phosphorothioate	2.5	5	12.5	20	30	40	50
a,a-Dimethyl-phenethylamine	2.5	5	12.5	20	30	40	50
2,6-Dichlorophenol	2.5	5	12.5	20	30	40	50
Hexachloropropene	2.5	5	12.5	20	30	40	50
p-Phenylenediamine	2.5	5	12.5	20	30	40	50
n-Nitrosodi-n-butylamine	2.5	5	12.5	20	30	40	50
Safrole	2.5	5	12.5	20	30	40	50
Isosafrole 1 + 2	2.5	5	12.5	20	30	40	50
1,4-Dinitrobenzene	2.5	5	12.5	20	30	40	50
1,4-Naphthoquinone	2.5	5	12.5	20	30	40	50
Pentachlorobenzene	2.5	5	12.5	20	30	40	50
1-Naphthylamine	2.5	5	12.5	20	30	40	50
2-Naphthylamine	2.5	5	12.5	20	30	40	50
N-Nitro-o-toluidine	2.5	5	12.5	20	30	40	50
Thionazin	2.5	5	12.5	20	30	40	50
1,3,5-Trinitrobenzene	2.5	5	12.5	20	30	40	50
Sulfotepp	2.5	5	12.5	20	30	40	50
Phorate	2.5	5	12.5	20	30	40	50
Phenacetin	2.5	5	12.5	20	30	40	50
Diallate 1 + 2	2.5	5	12.5	20	30	40	50
Dimethoate	2.5	5	12.5	20	30	40	50
4-Aminobiphenyl	2.5	5	12.5	20	30	40	50
Pentachloronitrobenzene	2.5	5	12.5	20	30	40	50
Pronamide	2.5	5	12.5	20	30	40	50
Disulfoton	2.5	5	12.5	20	30	40	50
2-secbutyl-4,6-dinitrophenol (Dinoseb)	2.5	5	12.5	20	30	40	50
Methyl parathion	2.5	5	12.5	20	30	40	50
4-Nitroquinoline-1-oxide	2.5	5	12.5	20	30	40	50
Parathion	2.5	5	12.5	20	30	40	50

Table LVI-2. (continued)
LVI Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Isodrin	2.5	5	12.5	20	30	40	50
Methapyrilene	2.5	5	12.5	20	30	40	50
Aramite 1 and 2	2.5	5	12.5	20	30	40	50
p-(Dimethylamino) azobenzene	2.5	5	12.5	20	30	40	50
p-Chlorobenzilate	2.5	5	12.5	20	30	40	50
3,3'-Dimethylbenzidine	2.5	5	12.5	20	30	40	50
2-Acetylaminofluorene	2.5	5	12.5	20	30	40	50
Dibenz (a,j)acridine	2.5	5	12.5	20	30	40	50
7,12-Dimethylbenz(a) anthracene	2.5	5	12.5	20	30	40	50
3-Methylcholanthrene	2.5	5	12.5	20	30	40	50

Table LVI-3.
LVI Calibration Levels, Nonroutine compounds, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8	CURVE ID
1,1'-Biphenyl	--	2.5	5	12.5	20	30	40	50	HSL
1-Chloronaphthalene	--	2.5	5	12.5	20	30	40	50	AP9
1-Methylnaphthalene	1	2.5	5	12.5	20	30	40	50	HSL
2,3-Dichlorobenzenamine	1	2.5	5	12.5	20	30	40	50	HSL
2,4,6-Tribromophenol	1	2.5	5	12.5	20	30	40	50	HSL
3,5-Dimethylphenol	1	2.5	5	12.5	20	30	40	50	HSL
3-Methylphenol	1	2.5	5	12.5	20	30	40	50	HSL
4,4-Dichlorobenzil	1	2.5	5	12.5	20	30	40	50	BRC
4,4'-Methylene bis(2-chloroaniline)	1	2.5	5	12.5	20	30	40	50	AP9
4-Chlorobenzenethiol	1	2.5	5	12.5	20	30	40	50	BRC
6-Methylchrysene	1	2.5	5	12.5	20	30	40	50	AP9
Acrylamide	1	2.5	5	12.5	20	30	40	50	AP9
Alachlor	1	2.5	5	12.5	20	30	40	50	HSL
Alpha Methyl Styrene	1	2.5	5	12.5	20	30	40	50	HSL
Alpha-Terpineol	1	2.5	5	12.5	20	30	40	50	REF
Atrazine	1	2.5	5	12.5	20	30	40	50	HSL
Benzaldehyde	1	2.5	5	12.5	20	30	40	50	BZHD
Benzo(e)pyrene	1	2.5	5	12.5	20	30	40	50	HSL
Bis(4-Chlorophenyl)disulfide	1	2.5	5	12.5	20	30	40	50	BRC
Caprolactam	1	2.5	5	12.5	20	30	40	50	HSL
Dibenz(a,h)acridine	1	2.5	5	12.5	20	30	40	50	AP9
Dibenz(a,e)pyrene	1	2.5	5	12.5	20	30	40	50	REF
Diphenylamine	--	2.5	5	12.5	20	30	40	50	AP9
Diphenylsulfone	1	2.5	5	12.5	20	30	40	50	BRC
Famphur	1	2.5	5	12.5	20	30	40	50	HSL
Hexachlorophene	1	2.5	5	12.5	20	30	40	50	HSL
Hexadecane	1	2.5	5	12.5	20	30	40	50	HSL
Indene	1	2.5	5	12.5	20	30	40	50	HSL
Isosafrole	--	2.5	5	12.5	20	30	40	50	AP9
Methyl 4-Chlorophenyl Sulfide	1	2.5	5	12.5	20	30	40	50	BRC
n-Decane	1	2.5	5	12.5	20	30	40	50	HSL
n-Hydroxymethlphthalimide	1	2.5	5	12.5	20	30	40	50	HSL
Octachlorostyrene	1	2.5	5	12.5	20	30	40	50	BRC
p-Chlorophenyl Sulfone	1	2.5	5	12.5	20	30	40	50	BRC
Phenyl Disulfide	1	2.5	5	12.5	20	30	40	50	BRC
Phenyl Sulfide	1	2.5	5	12.5	20	30	40	50	BRC
Phenyl Mercaptan	1	2.5	5	12.5	20	30	40	50	REF
Phthalic Acid	1	2.5	5	12.5	20	30	40	50	REF
Phthalic Anhydride	1	2.5	5	12.5	20	30	40	50	REF
Quinoline	1	2.5	5	12.5	20	30	40	50	AP9

APPENDIX A

Modifications Required for Analysis of Wastewater Following Method 625

REQUIREMENTS FOR METHOD 625

- Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits or other CWA compliance situations. The standard analyte list and reporting limits are listed in Table A-1.
- This method can be applied to only aqueous matrices.
- The tune period for this method is defined as 24 hours.
- Initial calibration curve requirements are as follows:
  - The initial calibration curve for this method requires at least three points.
  - Target compounds must have RSD ≤ 35%.
  - If this requirement cannot be met, a regression curve must be constructed for the non-compliant compounds.
- Continuing calibration verification requirements are as follows:
  - All target compounds must have %D ≤ 20%.
- Matrix Spike and LCS requirements are as follows:
  - A full analyte spike is required for method 625. See sections 7.8 and 7.9.
- 2012 MUR Required QC Elements
  - The May 2012 EPA Method Update Rule (MUR) to 40 CFR Part 136 for compliance testing under the Clean Water Act (CWA) requires laboratories to include 12 QC elements when performing the published or approved methods. See Work Instruction WI-DV-0060, QC Requirements for Methods Designated in 40 CFR Part 136, for list of approved test procedures performed by TestAmerica-Denver and the required QC elements in each of these methods.

Table A-1.
TestAmerica Method 625 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	20
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10

Table A-1. (continued)
TestAmerica Method 625 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10
N-Nitrosodimethylamine	62-75-9	10

APPENDIX B

Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)

REQUIREMENTS FOR METHOD 8270 BEST PRACTICE (8270BP)

- Method Best Practice is utilized to obtain lower reporting limits while still providing full scan data. The standard analyte list and reporting limits are listed in Table B-1.
- This method can be applied to only aqueous matrices.
- The extraction is the same with one exception. The final volume of the extract is 2 mL.
- The tune period for this method is defined as 12 hours.
- Initial calibration curve requirements are as follows:
  - Same as for 8270 detailed in Section 11.4 of this SOP.
  - The calibrations levels are shown in Table B-2.
- Continuing calibration verification requirements are as follows:
  - Same as for 8270 detailed in Section 11.5 of this SOP, except the level 7 calibration point (as defined in Table B-2) is used.
- Matrix Spike and LCS requirements are as follows:
  - The spike levels are listed in Table B-3.
- Internal Standards: The internal standard concentrations are listed in Table B-5.
- Surrogates: The surrogate concentrations are listed in Table B-4.
- Instrument Conditions are shown in Table B-6.

Table B-1.
TestAmerica Method 8270BP Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Pyridine	110-86-1	20
N-nitrosodimethylamine	62-75-9	5
Aniline	62-53-3	5
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	1
2-Chlorophenol	95-57-8	5
Benzyl alcohol	100-51-6	5
2-Methylphenol	95-48-7	5
2,2'-oxybis(1-chloropropane)[2]	108-60-1	5
4-Methylphenol	106-44-5	5
N-Nitroso-di-n-propylamine	621-64-7	5
Hexachloroethane	67-72-1	5
Nitrobenzene	98-95-3	5
Isophorone	78-59-1	5
2-Nitrophenol	88-75-5	5
Benzoic acid	65-85-0	10
Bis(2-chloroethoxy)methane	111-91-1	5
2,4-Dichlorophenol	120-83-2	5
1,2,4-Trichlorobenzene	120-82-1	5
Naphthalene	91-20-3	5
4-Chloroaniline	106-47-8	5
Hexachlorobutadiene	87-68-3	5
4-Chloro-3-methylphenol	59-50-7	5
2-Methylnaphthalene	91-57-6	5
Hexachlorocyclopentadiene	77-47-4	5
2,4,6-Trichlorophenol	88-06-2	5
2,4,5-Trichlorophenol	95-95-4	5
2-Chloronaphthalene	91-58-7	5
2-Nitroaniline	88-74-4	5
Dimethyl phthalate	131-11-3	5
Acenaphthylene	208-96-8	5
3-Nitroaniline	99-09-2	5
Acenaphthene	83-32-9	5
2,4-Dinitrophenol	51-28-5	5
4-Nitrophenol	100-02-7	5
Dibenzofuran	132-64-9	5
2,4-Dinitrotoluene	121-14-2	5
2,6-Dinitrotoluene	606-20-2	5
4-Chlorophenyl phenyl ether	7005-72-3	5
Fluorene	86-73-7	5
4-Nitroaniline	100-01-6	5
4,6-Dinitro-2-methylphenol	534-52-1	10
N-Nitrosodiphenylamine	86-30-6	5
Azobenzene	103-33-3	5
4-Bromophenyl phenyl ether	101-55-3	5

Table B-1. (continued)
TestAmerica Method 8270BP Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Hexachlorobenzene	118-74-1	1
Pentachlorophenol	87-86-5	10
Phenanthrene	85-01-8	1
Anthracene	120-12-7	5
Carbazole	86-74-8	5
Di-n-butyl phthalate	84-74-2	5
Fluoranthene	206-44-0	1
Benzidine	92-87-5	1
Pyrene	129-00-0	5
Butyl benzyl phthalate	85-68-7	5
3,3'-Dichlorobenzidine	91-94-1	5
Benzo(a)anthracene	56-55-3	1
Bis(2-ethylhexyl)phthalate	117-81-7	5
Chrysene	218-01-9	1
Di-n-octylphthalate	117-84-0	5
Benzo(b)fluoranthene	205-99-2	5
Benzo(k)fluoranthene	207-08-9	5
Benzo(a)pyrene	50-32-8	5
Indeno(1,2,3-cd)pyrene	193-39-5	5
Diethyl phthalate	84-66-2	5
Dibenz(a,h)anthracene	53-70-3	5
Benzo(g,h,i)perylene	191-24-2	5
1,4-Dioxane	123-91-2	1

Table B-2.
Method 8270BP Calibration Levels

Calibration Level	Calibration Concentration, µg/mL
1	0.25
2	0.40
3	1.00
4	2.50
5	5.00
6	7.50
7	10.
8	12.5
9	20.0
10	40.0
ICV	5.0

Table B-3.
Method 8270BP LCS Spike Concentrations

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	10
Bis(2-chloroethyl)ether	10
2-Chlorophenol	10
1,3-Dichlorobenzene	10
1,4-Dichlorobenzene	10
1,2-Dichlorobenzene	10
2,2'-oxybis(1-chloropropane)	10
N-Nitroso-di-n-propylamine	10
Hexachloroethane	10
Nitrobenzene	10
Isophorone	10
2-Nitrophenol	10
2,4-Dimethylphenol	10
Bis(2-chloroethoxy)methane	10
2,4-Dichlorophenol	10
1,2,4-Trichlorobenzene	10
Naphthalene	10
Hexachlorobutadiene	10
4-Chloro-3-methylphenol	10
Hexachlorocyclopentadiene	10
2,4,6-Trichlorophenol	10
2-Chloronaphthalene	10
Dimethyl phthalate	10
Acenaphthylene	10
Acenaphthene	10
2,4-Dinitrophenol	10
4-Nitrophenol	10
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
Diethylphthalate	10
4-Chlorophenyl phenyl ether	10
Fluorene	10
4,6-Dinitro-2-methylphenol	10
N-Nitrosodiphenylamine	10
4-Bromophenyl phenyl ether	10
Hexachlorobenzene	10
Pentachlorophenol	10
Phenanthrene	10
Anthracene	10
Di-n-butyl phthalate	10
Fluoranthene	10
Benzidine	10
Pyrene	10
Butyl benzyl phthalate	10
3,3'-Dichlorobenzidine	10
Benzo(a)anthracene	10

Table B-3. (continued)
Method 8270BP LCS Spike Concentrations

LCS Compounds	Spiking Level, ng/µL in extract[1]
Bis(2-ethylhexyl)phthalate	10
Chrysene	10
Di-n-octylphthalate	10
Benzo(b)fluoranthene	10
Benzo(k)fluoranthene	10
Benzo(a)pyrene	10
Indeno(1,2,3-cd)pyrene	10
Dibenz(a,h)anthracene	10
Benzo(g,h,i)perylene	10
N-Nitrosodimethylamine	10
1,4-Dioxane	10

Table B-4.
8270BP Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d_5	5
2-Fluorobiphenyl	5
Terphenyl-d_{14}	5
1,2-Dichlorobenzene-d_4[1]	5
Phenol-d_5	7.5
2-Fluorophenol	7.5
2,4,6-Tribromophenol	7.5
2-Chlorophenol-d_4[1]	7.5

[1] Included in standard mix, but not routinely evaluated for method 8270C

Recovery limits for surrogates are generated from historical data and are maintained by the QA department and stored in the LIMS

Table B-5.
8270BP Internal Standard Compounds

Internal Standard Compounds	Spiking Level, ng/µL in extract
1,4-Dichlorobenzene-d_4	40
Naphthalene-d_8	40
Acenaphthene-d_{10}	40
Phenanthrene-d_{10}	40
Terphenyl-d_{14}	40
Chrysene-d_{12}	40
Perylene-d_{12}	40

Table B-6.
Suggested Instrument Conditions for 8270BP

Mass Range:	35 - 500 amu
Scan Time:	≤1 second/scan
Initial Column Temperature/Hold Time:	50 ^{o}C for 1 minute
Column Temperature Program:	50 - 320 ^{o}C at 35^{o}C/min.
Final Column Temperature/Hold Time:	325 ^{o}C for 4 minute hold
Injector Temperature:	275 ^{o}C
Transfer Line Temperature:	290 ^{o}C
Source Temperature:	230 ^{o}C
Injector:	Single Taper Direct Connect Liner / Splitless injection
Injection Volume:	0.5 µL
Carrier Gas:	Helium at 1.0 mL/min.
Column:	DB-5 Capillary 20m x 0.18mm x 0.36 um film thickness

APPENDIX C

Instrument Maintenance Schedules
Mass Spectrometer & Gas Chromatograph

MASS SPECTROMETER Instrument Maintenance Schedule				
Daily	**Weekly**	**As Needed**	**Quarterly**	**Annually**
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43).	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Check vacuum, relays, gas pressures, and flows.	Replace the exhaust filters on the mechanical rough pump every 1 to 2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.		Change the oil in the mechanical rough pump.
Check inlets, septa.		Clean source, including all ceramics and lenses. Source cleaning is indicated by a variety of symptoms, including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.		Relubricate the turbomolecular pump-bearing wick.
Check baseline level.		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates the need for replacement.		

APPENDIX C (continued)

Instrument Maintenance Schedules
Mass Spectrometer & Gas Chromatograph

GAS CHROMATOGRAPH Instrument Maintenance Schedule (For GC/MS only.)	
Daily	***As Needed***
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Check inlets, septa. Clean injector port.	Replace septa.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Repair or replace flow controller if constant gas flow cannot be maintained.
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.