TESTAMERICA KNOXVILE
STANDARD OPERATING PROCEDURE
TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS
BASED ON METHOD 8260B
(SUPERSEDES: KNOX-MS-0015, Revision 17)

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of volatile organic compounds in waters, wastewater, organic waste, soils, sludges, and other solid matrices. Standard analytes are listed in Tables 1 and 2.

1.2. This SOP is based on SW-846 Method 8260B, 5030B,5035 and 5035A.

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 400 µg/L for 5 mL waters, 1 to 80 µg/L for 25 mL purge waters, 5 to 400 µg/kg for low-level soils, and 250 to 20,000 µg/kg for high-level soils, and 1250 to 100,000 µg/Kg for waste. Reporting limits are listed in Tables 1, and 2.

1.4.1. Analysis in Selected Ion Monitoring Mode (SIM) for select analytes will have a lower working range. Analytes and reporting limits are listed in Table 1A

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2. SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If low detection limits are required, soil samples may be preserved with sodium bisulfate and purged directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40^{o}C (40^{o}C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic

column. The gas chromatographic column is then heated to elute the components that are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

2.4.1. Qualitative identification in SIM mode may provide a lesser degree of confidence in the compound identification.

3. DEFINITIONS

3.1. 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 24 hour time period. Using this method, each BFB analysis will normally start a new batch. Batches for high level soils, waste or other extracts, are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together. See section 11.3.2.

3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. When there is insufficient sample to analyze an MS/MSD, an LCS/LCSD is analyzed. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Refer to the QC Program document (QA-003) for further details of the batch definition.

3.2. Method Blank: A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data.

3.3. Laboratory Control Sample (LCS): Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. An LCS from a source other than that of the calibration standards may also be used as the calibration verification (CCV) as long as the acceptance criteria for both the LCS and CCV are met.

3.4. Surrogates: Surrogates are 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. Each sample, blank, LCS, and MS/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.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample that is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC): CCCs are a representative group of compounds that are used to evaluate initial calibrations and calibration verifications. Relative percent difference for the initial calibration and % drift (or % difference) for the calibration verification response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC): SPCCs are compounds that are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the calibration verification is calculated for the SPCC compounds and compared to the specified method criteria.

3.8. Internal Standards (IS): Internal Standards are compounds added to every standard, QC sample, client sample or extract at a known concentration prior to analysis for the purpose of quantitation. For example, internal standards are used as the basis for quantitation of target compounds by GC/MS.

3.9. Additional definitions can be found in the TestAmerica Knoxville Quality Assurance Manual (QAM), current revision.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. 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. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. The samples may be diluted to preserve instrument integrity.

5. SAFETY

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

5.2. Specific Safety Concerns or Requirements

5.2.1. 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.

5.2.2. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

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

Material	Hazards	Exposure Limit (2)	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.
1– Exposure limit refers to the OSHA regulatory exposure limit.			

5.3.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include acrylonitrile, benzene, carbon tetrachloride, chloroform, 1,2-dibromo-3-chloropropane, 1,4-dichlorobenzene, and vinyl chloride.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger, 0.006 inch ID needle.

6.2. Syringe: 5 or 25 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance capable of weighing 0.01 g.

6.4. Glassware:

6.4.1. Vials, with screw caps and Teflon liners: 5 ml, 20 ml and 40 ml.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipets: Pasteur.

6.7. pH paper: Wide range.

6.8. Stir bars.

6.9. Gases:

6.9.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.9.2. Compressed air: Used for instrument pneumatics.

6.9.3. Liquid nitrogen: Used for cryogenic cooling if necessary.

6.10. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, the desorber and the transfer line to the GC.

6.10.1. Sample Purger: The recommended purging chamber is designed to accept 25 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.10.2. Trap: A variety of traps may be used, depending on the target analytes required. For most purposes the Vocarb 3000 trap is suitable. Other traps, such as Vocarb 4000, or Tenax/Silica gel/Charcoal may be used if the quality control criteria are met.

6.10.3. Desorber: The desorber should be capable of rapidly heating the trap to 250°C. Many such devices are commercially available.

6.10.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low level soil analysis.

6.11. Gas Chromatograph/Mass Spectrometer System:

6.11.1. Gas Chromatograph/Mass Spectrometer (GC/MS) System: An HP5973 analytical system complete with a temperature-programmable gas chromatograph. The GC capillary column is directly coupled to the MS source.

6.11.2. Column: 20 m x 0.18 mm I.D. 1-µm film thickness silicon-coated fused-silica capillary column (J&W DB-624 or equivalent).

6.11.3. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.11.4. 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 allows searching any GC/MS data file for ions of a specific mass and 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. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.11.5. Autosampler: Varian Archon, Tekmar Solatek, Tekmar Aquatek, OI Analytical Sample Processor or equivalent.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap Grade, High Purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.4) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.1.3. Sodium bisulfate

7.1.4. Reagent sand (e.g., Ottawa sand), Fisher Scientific , Cat# S801561 or equivalent. No expiration date applied to sand.

7.2. Standards

7.2.1. Calibration Standards

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10°to -20°C.

NOTE: Unopened stock solutions expire according to the manufacturer's expiration date. Once opened, the stock solution expires on the manufacturer's expiration date or in 6 months, whichever is shorter. Stock standards prepared from pure standard materials expire 6 months from date of preparation. Stock standards for gases expire one week after opening.

7.2.1.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20% then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem, then a new initial calibration must be performed.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

NOTE: The following expiration criteria apply to working standards from above and the standards described in 7.2.2 – 7.2.6. Working standards made from stock solutions shall be replaced after one week. Stock standards for gases shall be replaced after one week. When using premixed certified standards, the unopened standard expiration is according to manufacturer's expiration.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 3 (or Table 3A for SIM) for internal standard components.

7.2.3. Surrogate Standards: Refer to Table 4 (or Table 4A for SIM) for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Tables 1 and 2 for LCS components. Full analyte spikes are typically used (Tables 1 and 2).

7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Tables 1,and 2.

7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/µL of 4-Bromo-fluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

7.2.6.1. Note: A tuning standard is not required for SIM analysis

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sampling is not performed for this method by TestAmerica Knoxville. For information regarding sample shipping, refer to SOP KNOX-SC-0003, Sample Receipt and Log In, current revision.

8.2. Holding times for all volatile analysis are 14 days from sample collection. The holding time for surface or ground water samples that are known to be not preserved prior to receipt is 7 days from collection.

8.3. The maximum holding time is 14 days from sampling until the sample is analyzed. Samples must be either preserved in the field or delivered in EnCore™ samplers for laboratory preservation. Lack of preservation must be addressed in the case narrative. Maximum holding time for the EnCore™ sampler (before the sample is added to methanol or sodium bisulfate or water) is 48 hours.

8.4. Aqueous samples are stored in 40ml glass vials with Teflon lined septa not frozen at $\leq$6°C, with minimum headspace.

8.5. Methanol solid extracts are allocated into 2 - 20 mL glass vials with Teflon lined caps and stored at $\leq$6°C.

8.6. Water samples are normally preserved at pH $\leq$ 2 with hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.7. Soil samples are typically taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Solid samples may be field preserved with sodium bisulfate solution for low level analysis, or with methanol for high level analysis.

For low level soil samples with carbonaceous material present, reagent water may be used.

8.8. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take a 5 g EnCore™ sample. Following shipment back to the lab, the soil is preserved in methanol. This is the high level procedure. If very low detection limits are needed (< 200 µg/kg), then it will be necessary to use two additional 5 g EnCore™ samplers or to use field preservation.

8.9. Sample collection for high level analysis using EnCore™ samplers.

8.9.1. Ship one 5 g EnCore™ sampler per field sample position.

8.9.2. An additional bottle must be shipped for percent moisture determination.

8.9.3. When the samples are returned to the lab, extrude the (nominal) 5g sample into a tared VOA vial containing 5 mL methanol. Obtain the weight of the soil added to the vial and note on the label.

8.9.4. Prepare a method blank and an LCS for each batch by adding 5 mL methanol to 5 g sand.

8.9.5. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.9.6. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at ≤6°C until analysis.

8.10. Sample collection for high level analysis using field methanol preservation

8.10.1. Prepare a 20 ml sample container by adding 5.0 mL purge and trap grade methanol.

8.10.2. Seal the bottle and attach a label.

8.10.3. Weigh the bottle to the nearest 0.1g and note the weight on the label.

8.10.4. Ship with appropriate sampling instructions.

8.10.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.10.6. At client request, the methanol addition and weighing may also be performed in the field.

8.10.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.10.8. Prepare a method blank and an LCS for each batch by adding 5 mL methanol to 5 g sand.

8.10.9. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.10.10. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at ≤6°C until analysis.

8.11. Low level procedure

8.11.1. If low detection limits are required (typically < 200 µg/kg), sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the high level procedure (field methanol preserved or using the EnCore™ sampler), in case the concentration of analytes in the soil is above the calibration range of the low level procedure.

8.11.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method (Varian Archon).

8.11.3. The soil sample is taken using a 5g EnCore™ sampling device and returned to the lab. It is recommended that two EnCore™ samplers be used for each field sample position to allow for any reruns that may be necessary. A separate sample for % moisture determination is also necessary.

8.11.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.11.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.11.6. Weigh the vial to the nearest 0.1g and note the weight in the TALS prep batch.

8.11.7. Extrude the soil sample from the EnCore™ sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note in the TALS prep batch.

NOTE**:** Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case, add 5 mL of water instead, and freeze at <-10°C until analysis. The holding time for the frozen sample is 14 days from sampling. If one sample from a lot (site) effervesces, all soil samples from the site will be preserved in water to protect the integrity of the samples, unless otherwise specified by the client.

8.11.8. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the high level procedure. Depending on the type of soil, it may also be necessary to ship vials with no or extra preservative.

8.11.9. A preservation blank is prepared using the same reagents (i.e., sodium bisulfate/water solution) as the samples. A stir bar and 5 grams sand is added to the vial. The preservation blank is analyzed and evaluated using the same criteria as the method blank.

8.12. Unpreserved soils

8.12.1. At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030. It is no longer included and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.13. Holding Blank

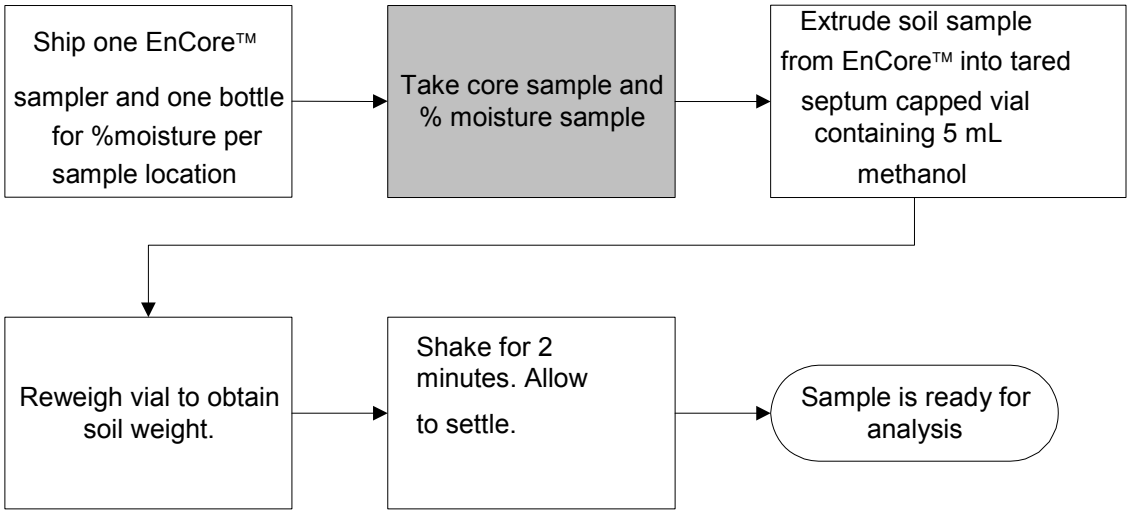
8.13.1. Sample receiving prepares holding blanks using reagent water obtained from the Metals laboratory and places the vials in the refrigerators used to store samples for volatiles analysis. These are logged into TALS once each week and are removed for analysis after two weeks.

8.13.2. The holding blanks are analyzed according to this SOP. The holding blank must be less than ½ the standard 25-ml purge reporting limit (RL) (<RL for common laboratory contaminants). If the holding blank is greater than ½ RL but less than the RL (for analytes that are not common lab

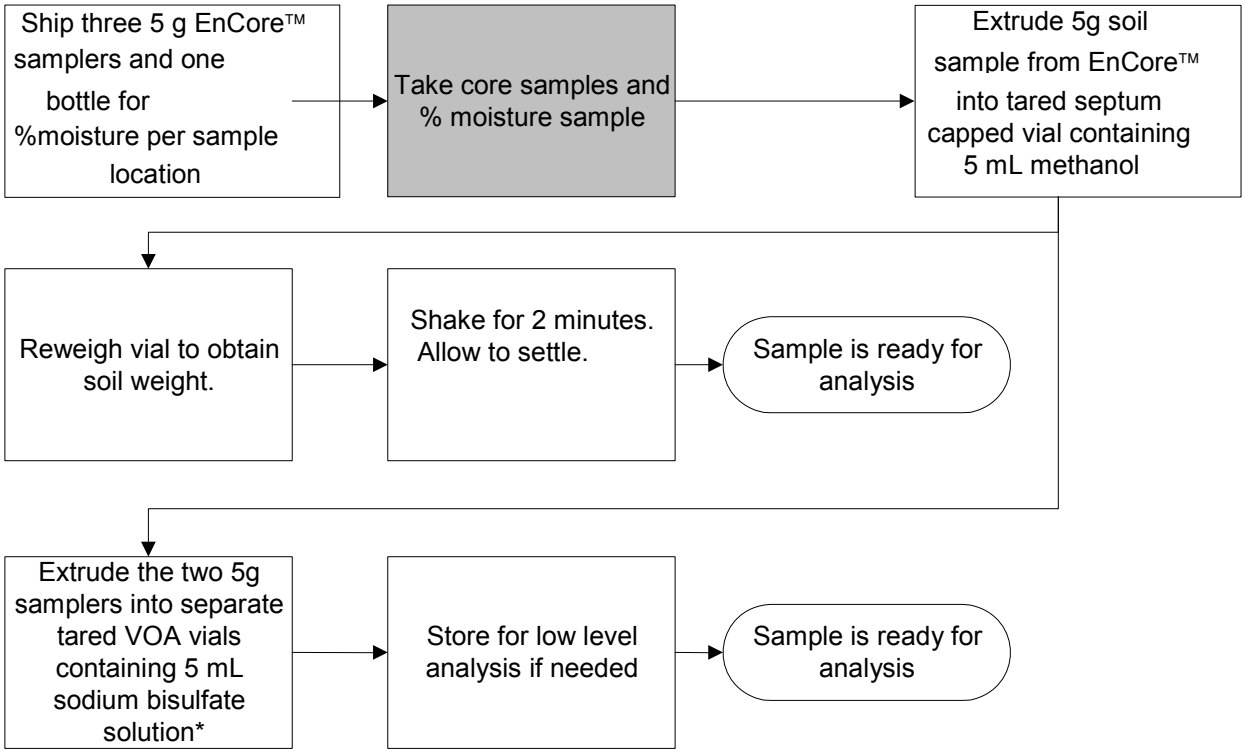
contaminants) document this on the quantitation report for the holding blank. If the holding blank is greater than the RL for any analyte, document this on the quantitation report and in a nonconformance memo. As corrective action, review associated method blanks and samples for the presence of any analyte greater than the RL that may be due to laboratory contamination that was observed in the associated holding blank. If laboratory contamination is observed in client samples, ensure that the data is appropriately flagged (B qualifier). Also discuss the nonconformance in the project narrative.

8.14. The methanol extracts are stored prior to analysis at ≤6°C. When long term storage after the analysis is requested by the client, the methanol extracts are also stored at ≤6°C.

EnCore™ procedure when low level is not required (field steps in gray)

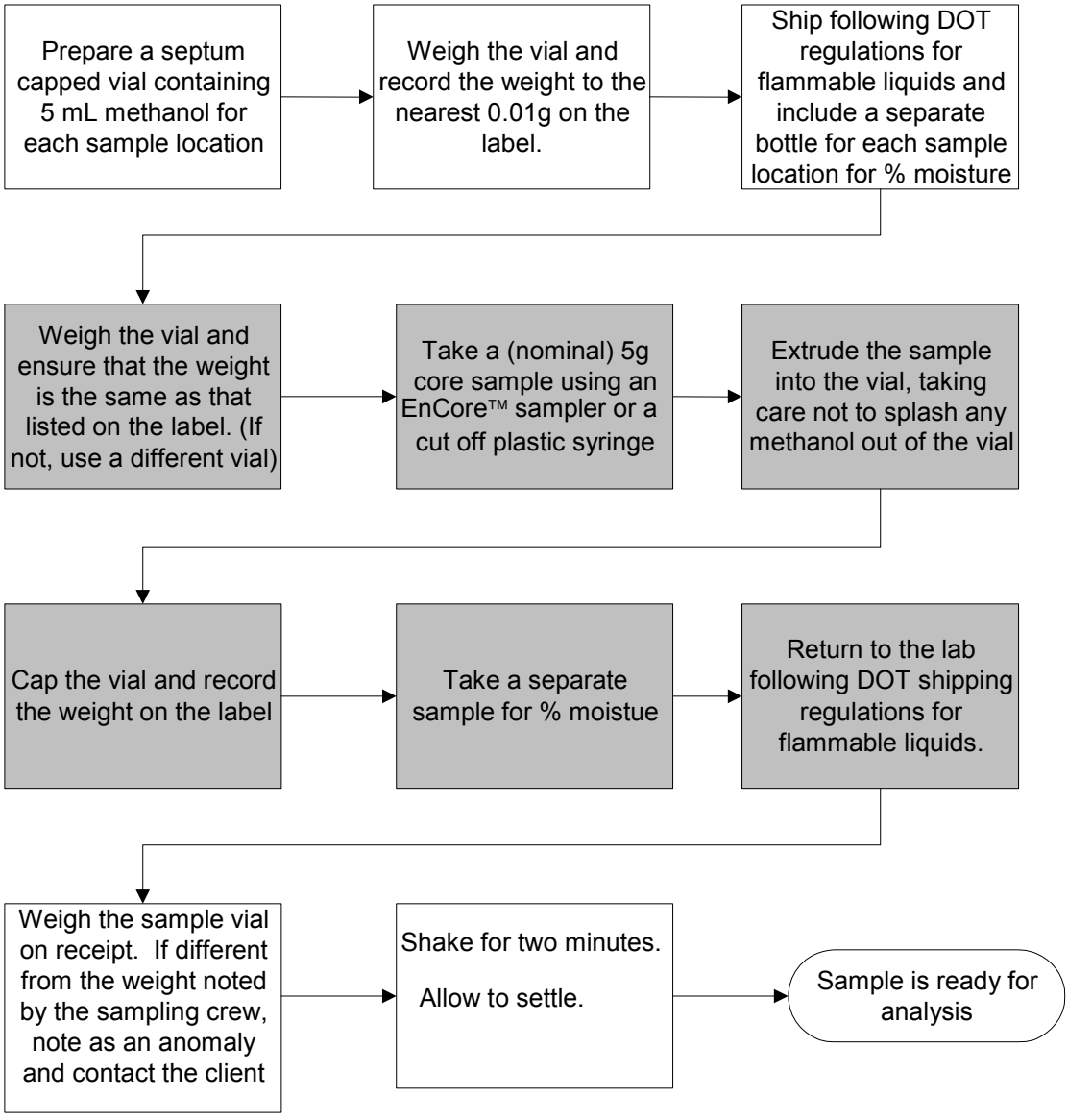


EnCore™ procedure when low level is required

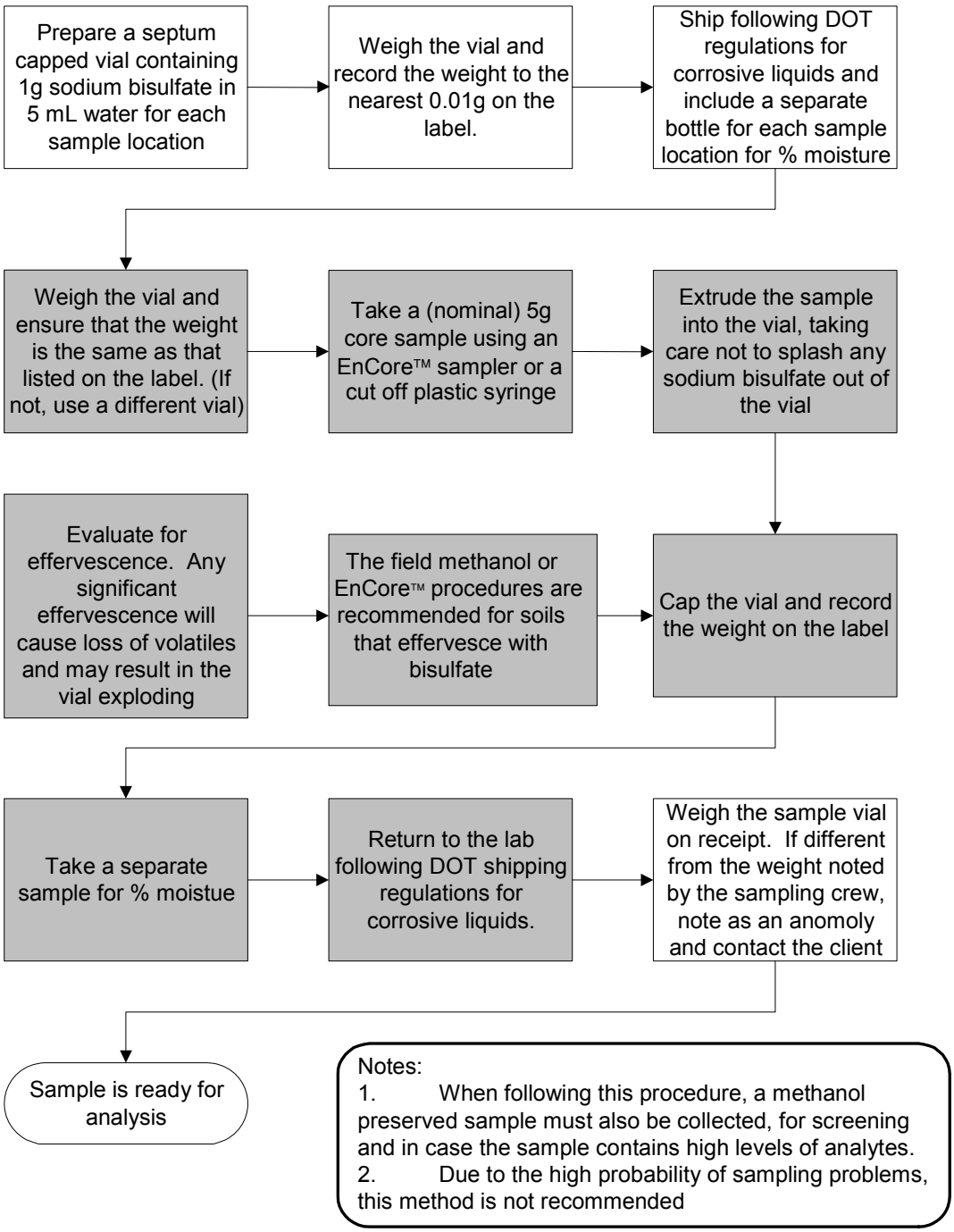


* Or 5 ml reagent water if samples from the lot or site effervesce.

Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



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9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. The initial demonstration described in section 13 and method detection limit (MDL) studies must be acceptable before analysis of samples may begin. MDLs must be analyzed for soils and aqueous samples. See section 13 for acceptance criteria.

9.2. Control Limits: In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits are determined annually. The historical control limits are set to the mean recovery +/- 3 standard deviations for surrogates, matrix spikes and LCS as described in SOP KNOX-QA-0004, current revision.

9.2.1. All surrogate, LCS, and MS recoveries (except when surrogates are diluted greater than 5X) must be entered into TALS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3. Surrogates: Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 4 (or Table 4A for SIM). Reanalyze samples with failing surrogates if sufficient sample material is available and matrix effects have not already been confirmed. The client may be contacted for input if the reanalysis is expected to take place after the sample holding time has been exceeded. If any surrogates are outside limits, the following corrective actions must take place (except when surrogates are diluted greater than 5X):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Reprepare and reanalyze the sample or flag the data as “Estimated Concentration” if neither of the above resolves the problem.
- The surrogate dibromofluoromethane has been determined to degrade in aqueous samples with pH ≥10, as well as soils where the preserved sample solution has a resulting pH ≥10. In these cases, the samples do not need to be reanalyzed. This should be noted in the narrative.

9.3.1. 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.

9.3.2. If the surrogates are out of control for the sample, matrix spike and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary as long as the LCS and method blank are acceptable. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.3.3. Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.4. Method Blanks: For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. The method blank contains the same reagents as the samples (e.g. 1 gram of sodium bisulfate per 5 ml reagent water for low level soils). For low-level waters, the method blank consists of reagent water. For high-level volatiles, the method blank consists of 5.0 mL of methanol and 5 g reagent sand. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone and toluene) the data may be reported with qualifiers when the concentration of the analyte is less than five times the reporting limit.

- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.4.1. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.4.2. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.4.3. Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.5. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. The LCS contains the standard set of target analytes (See Tables 1, and 2). The analytes are spiked at the same concentration as the calibration verification standard.

9.5.1. All target analytes requested that are listed in Tables 1 and 2 are considered control analytes in the LCS. Historical LCS limits are based on the mean recovery +/- 3 standard deviations as described in SOP KNOX-QA-0004, current revision. Marginal exceedances are allowed depending on the number of target analytes requested. The control limits for marginal exceedences are set to mean +/- 4 standard deviations. See the following table for the number of allowed marginal exceedances.

Number of target analytes in LCS	Allowable # of marginal exceedances of LCS control limits
>90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

9.5.2. An LCS is considered to be “out of control” if any target analyte is outside marginal exceedance limits, or if the total number of marginal exceedances is more than the allowed number. A nonconformance memo must be issued and corrective action must occur.

9.5.3. If marginal exceedances are observed, the analyst must review the previous LCS (e.g., review the control chart) for each analyte marginally exceeding the control limits to determine if the marginal exceedance is a consecutive occurrence. If there are two consecutive marginal

exceedances for the same analyte, the LCS is considered "out of control" and an NCM must be generated and corrective action taken.

9.5.3.1. When evaluating the control chart, the analyst should also check whether there was more than one out of the last three consecutive LCSs outside control limits. If more than one out of the last three LCSs was outside the LCS control limits but within the marginal exceedance limits, then the analyst should evaluate the system for non-random systematic trends.

9.5.4. Samples in the same batch as an LCS determined to be "out of control" shall be considered suspect and the samples re-analyzed or the data reported with appropriate data qualifying codes.

9.5.4.1. If the LCS recovery for a target analyte is biased high outside acceptance limits and that target analyte is not detected in any of the associated samples above the reporting limit, the sample data may be reported with qualification in the project narrative. Analytes that are biased high in the LCS and not detected in the associated samples are counted in the total number of allowable marginal exceedances.

9.5.4.2. If the batch is not reanalyzed, the reasons for reporting the batch must be clearly presented in the project narrative (e.g. due to limited sample volume)

9.5.5. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision .

9.5.6. Refer to the QC Program document (QA-003) for further details of the corrective action.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same analytes as the LCS. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific, historically generated limits; limits are based on the mean recovery +/- 3 standard deviations

9.6.1. If any individual spiked target analyte recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the LCS. If the LCS meets the acceptance limits stated in Section 9.5, then the laboratory

operation is in control and analysis may proceed; spike recovery or spike RPD failure is attributed to matrix effects.

9.6.2. If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to historical limits.

9.6.3. The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. For internal standard recovery acceptance criteria, refer to section 11.9.1.

9.8. Nonconformance and Corrective Action: Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager or designee.

9.9. Quality Assurance Summaries: Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to CA-Q-S-002, Manual Integration Practices, current revision for information on manual integration practices and documentation requirements.

10.2. Summary

10.2.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB electronic settings), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.2.1.1. Note: A BFB tune is not required for SIM analysis. The mass spectrometer is tuned as needed using perfluorotributylamine (PFTBA) and the instrument data system autotune program. Select the BFB tune optimization profile for the autotune program.

10.3. Recommended Instrument Conditions

10.3.1. General (full scan mode)

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 2 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: *30* mL/minute
Injector Condition:	1/35 split

10.3.2. SIM mode

Recommended GC & GC/MS Conditions are the same as 10.3.1 except the mass range is determined by the quantitation and secondary/tertiary ions monitored for each analyte, internal standard, and surrogate. See table 8 for the primary ion for quantitation, and use at least one of the secondary and/or tertiary ions as a monitor ion. Use the retention times from the full-scan analysis to set the MID switchpoints so that each analyte's elution time falls within their respective MID groups. See Figure 1 for example of GC & GC/MS conditions and settings.

10.3.3. Gas chromatograph suggested temperature program

10.3.3.1. BFB Analysis: 150°C for 1 minute, then 20°C/minute until 200°C.

10.3.3.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	3 minutes
Temperature Program:	11°C/minute
Final Temperature:	195°C
Second Temperature	Program: 25°C/minute
Final Temperature:	220°C
Final Hold Time:	2.0 minutes

10.4. Instrument Tuning

10.4.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 5 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.4.1.1. Note: A BFB tune is not required for SIM analysis. The 12-hour period begins at the moment of injection of the first acceptable ICAL point.

10.4.2. Inject 50 ng of the GC/MS tuning standard (1 uL of the 50 ug/ml solution) into the GC/MS system. Obtain a mass spectra of BFB and confirm that all the key m/z criteria in Table 5 are achieved. The typical approach is to use the average of the peak apex, the scan immediately before the apex, and the scan immediately after the apex, with background subtraction of a single scan. This single scan must be prior to and within 20 scans of the start of the BFB elution but must not be part of the BFB peak. Alternatively the peak apex may be used. Background subtraction is required. If all the criteria in Table 5 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.

10.5. Initial Calibration

10.5.1. A series of five or more initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Typical calibration levels are given in Tables 9 and 10 (or Table 9A for SIM). Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.5.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.

10.5.3. Internal standard calibration is used. The internal standards are listed in Table 3 (or Table 3A for SIM). Target compounds are typically referenced to the nearest internal standard (see Table 8). Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See section 12.4.1 for calculation of response factor.

10.5.4. The high point calibration standard is checked for saturation. If a quantitation ion saturates the mass spectrometer, the analyte will be removed from the calibration series, and the next highest concentration is checked for saturation as well. Saturation is present when an ion peak in Chrom reaches a Y axis maximum of 8.4×10^6.

10.5.5. The % RSD of the calibration check compounds (CCC) must be less than or equal to 30%. Refer to Table 7 for the CCCs.

10.5.6. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 6 for the SPCC compounds and required minimum response factors.

10.5.6.1. If none of the SPCCs are required analytes, project specific calibration specifications must be agreed to with the client.

10.5.7. The analyst will evaluate analytes with %RSD > 15% for calibration on a curve.

10.5.8. A read-back of the low calibration point used in the calibration curve should have a read-back (%D) of no more than 50% for any calibration model. However, common laboratory contaminants (i.e. methylene chloride and acetone) and poor performing compounds (defined by QA and control charts) may have a read-back limit of no more than 80%.

10.5.9. Linear or quadratic curve fits may be used. Use of $1/\text{Concentration}^2$ weighting may be used to improve the accuracy of quantitation at the low end of the curve. The correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990.

10.5.9.1. Analyst may elect to drop points from the calibration to improve subsequent quantitation. <u>The rules for dropping points are:</u>

- May drop points below the RL as long as there is a point remaining at or below the RL.
- May drop high points, decreasing linear range.
- May NOT drop a point between points.

For more guidance see "CA-Q-P-003, Calibration Curves and the Selection of Calibration Points, current revision."

10.5.9.2. Rules for curve use:

- The r^2 value obtained from Chrom™ must be ≥ 0.990.

- At least 5 points must be used for average or linear curve.
- At least 6 points must be used for a quadratic curve.
- For quadratic curves, the tangent line to the slope of the curve must be continuous and have either only positive or negative slopes (i.e., no parabolas or breaks in the curve). Quadratic curves cannot be used to extend the calibration range.
- Forcing through zero is allowed. To activate "force through zero" in Chrom, pick a "Curve Select" model that uses a "Force" for "Origin". "Include" zero for "curve origin" must NOT be used.
- If "forced through zero" is not used, the X and Y-intercept must be below the RL.
- To evaluate the X- and Y-intercept, evaluate each analyte's Curve Details in Chrom to ensure that the Zero Intercept flag is not activated due to improper intercept.

10.5.9.3. See corporate policy, CA-Q-P-003, "Calibration Curves & Selection of Calibration Points" current revision, for definitions and calculations using the different curve models.

10.5.10. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to calibration verification.

10.5.11. A separate five point calibration must be prepared for analysis of low level soils. Low level soils analysis requires the use of a closed vial autosampler (Varian Archon). Each standard is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1 g sodium bisulfate. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Alternatively, add 5 ml of water to a 40 ml VOA vial for soil samples that effervesce or are received in jars.

10.5.12. Initial Calibration Verification Standard (2nd Source Standard): A mid-level standard from a second source is analyzed as an initial calibration verification (ICV). The ICV shall be analyzed with each initial calibration. The ICV must be within +/- 30% of its expected value. Poorer performing analytes may have an alternate acceptance criterion with QA approval (e.g., ketones < 35% and alcohols <40%). If the criteria are not met, the analyst must first verify the concentrations of the primary and secondary source standards and calculations. If no errors are found, repeat the ICV analysis.

10.6. Calibration verification: The initial calibration must be verified every twelve hours.

10.6.1. Calibration verification begins with analysis of BFB as described in Section 10.4. If the system tune is acceptable, the calibration verification standard(s) are analyzed. The level 3 calibration standard is suggested as the calibration verification.

10.6.1.1. Note: A BFB tune is not required for SIM analysis. The 12-hour period begins at the moment of injection of an acceptable CCV.

10.6.2. The RF data or concentrations from the standards are compared with the average RF or concentrations from the initial five-point calibration to determine the percent difference or drift of the CCC compounds. The calculation is given in Section 12.4.4.

10.6.3. The % drift or difference of the CCCs must be $\leqq$ 20% for the calibration verification to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 6. In addition, the % drift of all analytes must be $\leqq$ 50%.

10.6.3.1. If none of the CCCs are required analytes, then project specific calibration specifications should be negotiated with the client.

10.6.4. If the CCCs and or the SPCCs do not meet the criteria in Sections 10.6.3, the system must be evaluated and corrective action must be taken. The BFB tune and calibration verification must be acceptable before analysis begins. Extensive corrective action such as the installation of a new column will require a new initial calibration.

10.6.5. 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 calibration verification RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample desorbed less than or equal to 12 hours after the BFB is acceptable.)

10.6.5.1. Note: For SIM, the 12-hour clock is from the injection of an acceptable CCV or first point in the initial calibration series.

10.6.6. If the retention time for any internal standard in the calibration verification changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.6.7. If the internal standard response in the calibration verification is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.6.8. If the calibration verification does not meet acceptance criteria, corrective action is required before sample analysis.

10.6.9. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications. If the laboratory has not demonstrated acceptable performance, sample analyses must not occur until a new initial calibration curve is established and verified.

11. PROCEDURE

11.1. Procedural Variations

11.1.1. 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 except those specified by project specific instructions shall be completely documented using a Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2. Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required

would use less than 1 µL of sample, then serial dilutions must be made in volumetric flasks.

11.2.2.1. The diluted concentration is to be estimated to be in the upper half of the calibration range. See section 11.10 for guidance.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the calibration verification standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a 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 24 hour time period. The batch also must contain a MS/MSD, a LCS, and a method blank. If insufficient sample is available to perform an MS/MSD pair, an LCS/LCSD pair will be prepared and analyzed.

11.3.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. For high level soils, waste, or other extracts, the batch is defined at the sample preparation stage.

11.3.2.2. Laboratory generated QC samples (blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.3.2.3. It is not always necessary to reanalyze batch QC with reanalyses of samples. For example, if the samples need to be analyzed at a different dilution, batch QC does not need to be reanalyzed. If samples need to be reanalyzed because the batch QC failed, then batch QC must be reanalyzed. Also, any reruns must be part of a valid batch

11.3.3. Weight/volume entries in the TALS Preparation Batch are not to be included in the Chrom Worklist, which would lead to “double-calculation” of the preparation step (e.g. initial/final weight/volumes & dilution factors).

11.4. Water Samples

11.4.1. All samples and standard solutions must be at ambient temperature before analysis.

11.4.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is ≧ 1 µL. Check and document the pH of the remaining sample.

11.4.3. Add each internal and surrogate standard at the concentrations stated in Tables 3 and 4 or Tables 3A and 4A for SIM. The internal standards and the surrogate standards may be mixed and added as one spiking solution via the autosampler. Inject the sample into the purging chamber.

11.4.4. The sample is purged for eleven minutes (the trap must be at or below 40°C).

11.4.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.4.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.5. Methanol Extract Soils

11.5.1. Add no more than 100 µL methanolic extract (from Section 8.9 or 8.10) to the 40 ml VOA vial containing 5 ml of organic free water. Add internal standards and surrogates through the 40 ml vial septum or allow the autosampler to add the appropriate amount. Add the appropriate matrix spike solution to the vial for the LCS/LCSD/MS/MSD as needed. Load the sample onto the autosampler/purge and trap device and analyze as for soil samples. If less than 1µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume no less than 1µL will be added to the water in the 40 ml VOA vial.

11.6. Liquid wastes that are soluble in methanol and insoluble in water.

11.6.1. Pipet 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

11.6.2. Quickly add methanol, and bring the final volume to 10 mL. Cap the vial and invert several times to mix thoroughly.

11.6.3. Add no more than 100 µL methanolic extract to the 40 ml VOA vial containing 5 ml of organic free water. Add internal standards and surrogate through the 40 ml vial septa or allow the autosampler to add the appropriate amount. Add the appropriate matrix spike solution to the vial for the LCS/LCSD/MS/MSD as needed. Load the sample onto the

autosampler/purge and trap device and analyze as for soil samples. If less than 1µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume no less than 1µL will be added to the water in the 40 ml VOA vial.

11.7. Aqueous and Low level Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.7.1. Units that sample from the VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.7.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils. If multiple vials are provided and all have sediment present, decant the liquid layer into another clean 40-mL VOA vial for analysis, leaving the sediment in the original vial (the sediment is not analyzed).

11.7.3. Soil samples must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate or water as the samples (1 g in 5 mL).

11.7.4. Soil sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.7.5. For aqueous samples, check and document in the TALS batch worksheet the pH of the sample remaining in the VOA vial after analysis is completed.

11.8. Low-Level Solids Analysis (from bulk container/jar)

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and internal standards, and matrix spiking standards when applicable. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40^{o}C**.**

11.8.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.8.2. Weigh out 5 g (or other appropriate aliquot) of sample into a 40 ml VOA vial. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5 g. If the sample is contaminated with analytes such that a purge amount less than *0.5* g is appropriate, use the high level method described in section 11.5.

11.8.3. Add 5 mL of organic free water, and add matrix spike solutions (if required), directly to the sample from section 11.8.2.

11.8.4. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.8.5. Analyze as described in Section 11.7.

11.9. Initial review and corrective actions

11.9.1. Any samples that do not meet the internal standard criteria for the calibration verification must be evaluated for validity. **Note:** The sample internal standard recovery is referenced against the calibration verification, not the initial calibration; see sections 10.6.6 and 10.6.7. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis is not necessary. The sample in question must be bracketed by acceptable runs. If the change in sensitivity is due to instrument problems, all affected samples must be reanalyzed after the problem is corrected.

11.9.2. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis is not necessary. Out of control surrogate standard response may be a matrix effect; obvious matrix effects (i.e., high level interfering peaks) that affect the surrogate quantitation do not need to be reanalyzed. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

11.10. Dilutions: If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract or sample is prepared 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 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.10.1. Guidance for Dilutions Due to Matrix

11.10.1.1. If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non-target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment.

11.10.1.2. If samples have reportable results greater than 2x the reporting limit or ¼ the internal standard height for non-target analytes for a methanol prep, a low level prep is not required, unless otherwise specified by the client.

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

11.11. Troubleshooting Guide (Refer to the manufacturer's manual for specific guidance)

11.11.1. Daily Instrument Maintenance: In addition to the checks listed in the instrument maintenance schedule in TestAmerica Knoxville QAM, the following daily maintenance should be performed as necessary.

11.11.1.1. Install new or cleaned injection port liner.

11.11.1.2. Install new septum.

11.11.1.3. Install new inlet seal.

11.11.1.4. Perform/adjust mass calibration (autotune/BFB tune).

11.11.1.5. Increase/decrease EM voltage to desired sensitivity based on internal standard response.

11.11.2. Major Maintenance: A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, repairing the source, changing electronics, replacing the multiplier or replacing trap in the purge and trap.

11.11.3. Minor Maintenance

11.11.3.1. Minor maintenance includes daily instrument maintenance described in 11.11.1, cleaning injector port, replacing filters, changing pump oil, autotuning, cleaning the source, replacing source insulators, replacing or switching filaments, replacing transfer line, change/refill IS/surrogate standard vial, changing seals and o-rings, ballasting pump, replacing fuses, or replacing roughing pumps.

11.11.3.2. Replace filters and change pump oil about every 6-12 months.

11.11.3.3. A multiplier gain check is performed if sensitivity is still poor and/or analyst suspects that the multiplier is going bad.

11.11.3.4. Autotuning is performed if the analyst notices mass misassignments or a drift in the response of analytes or internal standards.

11.11.3.5. If minor maintenance does not result in acceptable chromatography, it may be necessary to change the column or clean the source.

11.12. Refer to current revision of TestAmerica Knoxville SOP-KNOX-IT-0001, "Good Automated Laboratory Practices", for requirements for computer hardware and software.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to Appendix A for an example data review checklists used to perform and document the review of the data. Using the data review checklist, the analyst also creates a narrative which includes any qualifications of the sample data.

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 NIST 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. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.) The characteristic ions from the reference mass spectrum are defined as the three ions with greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions are present in the reference spectrum (i.e. characteristic ions have relative intensity $\geq$30%).

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

- All characteristic ions must maximize in the same scan or within one scan of each other.

- 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 percent.)

➢ Note: For SIM analysis, since not all of the ions in a compound may be monitored, comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum) cannot be performed.

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

12.3. Tentatively Identified Compounds (TICs): Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the TestAmerica Knoxville SOP KNOX-MS-0014, current revision, "Determination of Tentatively Identified Compounds (TICs)".

12.3.1. The laboratory defaults to a TIC quality match of 85%. It is project manager's responsibility to relay any special TIC client requirements via special instructions.

12.3.2. Note: A TIC search cannot be performed when data has been acquired in SIM mode.

12.4. Calculations.

12.4.1. Response factor (RF):

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

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.4.2. Standard deviation (SD):

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

X_i = Value of X at i through N

N = Number of points

X = Average value of X_i

12.4.3. Percent relative standard deviation (%RSD):

$$\%\ RSD = \frac{\text{Standard Deviation}}{\overline{RF}} \times 100$$

12.4.4. Calibration verification percent drift and difference from the initial calibration (note: Chrom/TALS uses "%D" in the report header for both % difference and % drift):

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

Where

$C_{expecte}$ = Known entratio in d

C_{found} = Measured oncentrat on using selected antitatio method

$$\%Difference = \frac{RF - RRF}{RF} \times 100$$

RRF = Average Analyte Response Factor from Initial Calibration

RF = Measured Analyte Response Factor from Calibration Verification

12.4.5. Target compound and surrogate concentrations: Concentrations in the sample may be determined from linear or second order (quadratic) curve

fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the RSD criteria listed in section 10.5.7 are met.

12.4.5.1. C_{pv} = Concentration in purge vessel, ug/L

On-column concentration of sample as determined by the initial calibration curve model (see 10.5.9.3).

12.4.5.2. Calculation of Concentration for Water Samples:

$$\text{Concentration, } \mu\text{g/L} = \frac{C_{pv} \cdot DF \cdot Uf \cdot V_t}{V_o \cdot 1000}$$

Where:

V_t = Total Volume Purged (ul)

V_o = Sample Volume used (ml)

DF = Dilution Factor (e.g. for a one to ten dilution D=10)

Uf = Unit correction factor (default =1 [ml/ul])

12.4.5.3. Calculation of Concentration for Methanol Extracted Soils:

$$\text{Concentration, } \mu\text{g/kg} = \frac{C_{pv} \cdot DF \cdot Wd \cdot V_t}{V_a \cdot Ws \cdot X}$$

Where:

V_t = Final Methanol Extract Volume, uL

V_a = Nominal Volume of extract analyzed, 100 µL

W_s = Weight of sample extracted, g

Wd = Default volume of water purged, ml (Default=5ml)

X = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis (moisture factor applied by LIMS)

DF = Dilution Factor (e.g., if 10 uL of methanol extract are analyzed, the dilution factor is 10)

12.4.5.4. Calculation of Concentration for Low Level Soils:

$$\text{Concentration, } \mu\text{g/kg} = \frac{C_{pv} \cdot DF \cdot V_t}{Ws \cdot X \cdot 1000}$$

Where:

$\mathbf{V_t}$ = Low Soil Sample Purge Volume, uL

$\mathbf{W_s}$ = Weight of sample extracted, g

X = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis (moisture factor applied by LIMS)

12.4.6. MS/MSD Recovery

$$\textbf{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result

SR = Sample result

SA = Spike added

12.4.7. Relative % Difference calculation for the MS/MSD

$$\textbf{RPD} = \frac{|\textbf{MSR - MSDR}|}{\frac{1}{2}\textbf{(MSR + MSDR)}} \times \textbf{100}$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. Method Detection Limit: Method Detection Limit (MDL) - An MDL must be determined for each analyte in each routine matrix prior to the analysis of any samples. Method Detection limits are determined and verified as specified in the current revision of SOP CA-Q-S-006 based on 40 CFR Part 136 Appendix B.

13.2. Initial Demonstration: Each analyst must perform an initial demonstration of capability (IDOC) for each target analyte prior to performing the analysis independently. The IDOC is determined by analyzing four replicate spikes (e.g., LCSs) as detailed in TestAmerica Knoxville SOP KNOX-QA-0009. This requires analysis of QC check samples containing all of the routine analytes for the method (Table 1 and 2). The QC check sample is made at 10 ug/L (0.1 ug/L for SIM) or at the current LCS spike level. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. The %RSD should be ≤ 15% for each analyte, and the % recovery should be within 70-130%.

13.2.3. If any analyte does not meet the acceptance criteria, determine if historical data indicates that the analyte purges poorly. In this case, QA approval is required for the IDOC to be acceptable. If the recovery or precision is outside the 70-130% limits and the above criteria is not met, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification: The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. Refer to SOP KNOX-QA-0009 current revision for further requirements for performing and documenting initial and on-going demonstrations of capability.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. 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 method and the policies in section 13 of the TestAmerica Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

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

- Aqueous waste generated from analysis. This material may have a pH of less than 2. This waste will be placed in an acid satellite accumulation container.
- Solvent waste generated from analysis is placed in the flammable waste stream, contained in a steel satellite accumulation container type or flammable solvent container.
- VOA vials containing extracted soil samples, which will contain small amounts of methanol will be placed in the vial waste stream 55 gallon open top drum.
- Expired Standards are stored in metal closed-top containers.

16. REFERENCES

16.1. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996.

16.2. SW846, Method 5030B, "Purge and Trap for Aqueous Samples", Revision 2, December 1996.

16.3. SW846, Method 5035, "Closed-System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples", Revision 0, December 1996.

16.4. TestAmerica Knoxville Quality Assurance Manual (QAM), current revision.

16.5. CA-Q-S-002, Manual Integration, current revision.

16.6. Method 8000C "Determinative Chromatographic Separations", March 2003.

16.7. Method 5035A "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples", draft revision 1, July, 2002.

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.

17.1.2. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

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 SOP allows for the use of the NIST library in the qualitative identification of an analyte. Method 8260B allows for the use of the mass spectra for standard reference from the user's instrument.

17.1.5. Section 7.3.6 of method 5035 states that methanolic extracts are stored at 4°C. Section 11.3.6 of method 5035A states that extracts must be stored at 4°C. Section 6.1 of method 5030B states that samples are stored at 4°C or less. This SOP allows the storage of extracts to be $\leq$6°C.

17.1.6. 8260B indicates that the surrogates are calibrated using a minimum of five different concentrations. The concentration of the surrogates in this SOP is constant throughout the initial calibration event, as noted in tables 9 & 10. The EPA has recognized an option for allowing the autosampler to spike the initial calibration standards with surrogates in the same manner as the samples are spiked.

Table 1 - Standard Analytes and Reporting Limits

Compound	CAS Number	Water[1] µg/L	Low soil[2] µg/kg	Med. Soil[2] µg/kg
Acetone	67-64-1	10	20	1000
Benzene	71-43-2	1	5	250
Bromochloromethane	74-97-5	1	5	250
Bromodichloromethane	75-27-4	1	5	250
Bromoform	75-25-2	1	5	250
Bromomethane	74-83-9	2	10	500
2-Butanone (syn: methyl ethyl ketone, MEK)	78-93-3	5	20	1000
Carbon disulfide	75-15-0	1	5	250
Carbon tetrachloride	56-23-5	1	5	250
Chlorobenzene	108-90-7	1	5	250
Chlorodibromomethane	124-48-1	1	5	250
Chloroethane	75-00-3	2	10	500
Chloroform	67-66-3	1	5	250
Chloromethane	74-87-3	2	10	500
1,2-Dibromoethane (EDB)	106-93-4	1	5	250
Dibromomethane (syn: methylene bromide)	74-95-3	1	5	250
1,1-Dichloroethane	75-34-3	1	5	250
1,2-Dichloroethane	107-06-2	1	5	250
1,1-Dichloroethene	75-35-4	1	5	250
cis-1,2-Dichloroethene	156-59-2	1	5	250
trans-1,2-Dichloroethene	156-60-5	1	5	250
1,2-Dichloroethene (Total)	540-59-0	1	5	250
1,2-Dichloropropane	78-87-5	1	5	250
2,2-Dichloropropane	594-20-7	1	5	250
1,1-Dichloropropene	563-58-6	1	5	250
cis-1,3-Dichloropropene	10061-01-5	1	5	250
trans-1,3-Dichloropropene	10061-02-6	1	5	250
Ethylbenzene	100-41-4	1	5	250
2-Hexanone	591-78-6	5	20	1000
Methylene chloride	75-09-2	2	5	250
4-Methyl-2-pentanone (syn: methyl isobutyl ketone, MIBK)	108-10-1	5	20	1000
Styrene	100-42-5	1	5	250
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	1	5	250
Tetrachloroethene	127-18-4	1	5	250
Toluene	108-88-3	1	5	250
1,1,1-Trichloroethane	71-55-6	1	5	250
1,1,2-Trichloroethane	79-00-5	1	5	250
Trichloroethene	79-01-6	1	5	250
Vinyl chloride	75-01-4	2	10	500
o-xylene	95-47-6	1	5	250
m-Xylene and p-Xylene	136777-61-2	2	10	500
Xylenes (total)	1330-20-7	3	15	750

[1]Levels for 5 mL purge water samples are 5 times higher. This is achieved by analyzing 5 mL sample in a 25 mL final volume.

[2]Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table 1A – Reporting limit for SIM analytes

Compound	CAS Number	Water[1] µg/L
Chlorobenzene	75-05-8	0.01
Tetrachloroethene	107-13-1	0.01

Table 2 - Additional Analytes and Reporting Limits

Compound	CAS Number	Water[1] µg/L	Low soil[2] µg/kg	Med. Soil[2] µg/kg
Acetonitrile	75-05-8	20	100	5000
Acrylonitrile	107-13-1	20	100	5000
Bromobenzene	108-86-1	1	5	250
1,3-Butadiene	106-99-0	1	5	250
n-Butylbenzene	104-51-8	1	5	250
Sec-Butylbenzene	135-98-8	1	5	250
tert-Butylbenzene	98-06-6	1	5	250
2-Chloropropane	75-29-6	1	5	250
2-Chlorotoluene	95-49-8	1	5	250
4-Chlorotoluene	106-43-4	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	2	10	500
1,2-Dichlorobenzene	95-50-1	1	5	250
1,3-Dichlorobenzene	541-73-1	1	5	250
1,4-Dichlorobenzene	106-46-7	1	5	250
cis-1,4-dichloro-2-butene	1476-11-5	2	10	500
trans-1,4-dichloro-2-butene	110-57-6	2	10	500
1,4-dichloro-2-butene (Total)	164-41-0	4	20	1000
Dichlorodifluoromethane	75-71-8	2	10	500
1,3-Dichloropropane	142-28-9	1	5	250
Hexachlorobutadiene	87-68-3	2	10	500
Hexane	110-54-3	2	10	500
Iodomethane (syn: methyl iodide)	74-88-4	2	10	500
Isopropylbenzene (syn: Cumene)	98-82-8	1	5	250
Isopropyltoluene (syn: p-Cymene)	99-87-6	1	5	250
Methyl *tert*-butyl ether (MTBE)	1634-04-4	1	5	250
Methyl methacrylate	80-62-6	1	5	250
Napthalene	91-20-3	1	5	250
n-Propylbenzene	103-65-1	1	5	250
Tetrahydrofuran	109-99-9	4	20	1000
1,2,3-Trichlorobenzene	87-61-6	1	5	250
1,2,4-Trichlorobenzene	120-82-1	1	5	250
Trichlorofluoromethane	75-69-4	2	10	500
1,2,3-Trichloropropane	96-18-4	1	5	250
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	2	10	500
1,2,4-Trimethylbenzene	95-63-6	1	5	250
1,3,5-Trimethylbenzene	108-67-8	1	5	250
Vinyl acetate	108-05-4	2	10	500
Vinyl bromide	593-60-2	2	10	500
Methyl Acetate	79-20-9	1	5	250
Cyclohexane	110-82-7	1	5	250
Methyl Cyclohexane	108-87-2	1	5	250

2-Propanol[3]	67-63-0	50	-	-

[1]Levels for 5 mL purge water samples are 5 times higher. This is achieved by analyzing 5 mL sample in a 25 mL final volume.
[2]Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.
[3]Analyte exhibits poor precision and accuracy, not used as a control analyte in the LCS

Table 3 - Internal Standards

	Amount added to sample being purged, ng	Quantitation ion
Fluorobenzene	250	96
Chlorobenzene-d5	250	117 (119)
1,4-Dichlorobenzene-d4	250	152

Notes:
1) This results in a concentration of each internal standard in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge or 50 ug/kg for a 5 gram soil purge.
2) This is achieved by spiking 5 uL of a 50 ug/mL standard manually, or if an autosampler loop is used, the concentration of the solution is adjusted to the volume of the spiking loop.
3) Mass 119 is used for Chlorobenzene-d5 for 25ml analyses
4) Except for high level soils, the surrogate and internal standards may be combined in one solution.

Table 3A - Internal Standards (SIM)

	Amount added to sample being purged, ng	Quantitation ion	Secondary ion
Chlorobenzene-d5	2.5	117	82
1,4-Dichlorobenzene-d4	2.5	152	150

Note: This results in a concentration of each internal standard in the sample of 0.1 µg/L for a 25 mL purge

Table 4 - Surrogate Standards

Surrogate Compounds	Amount added to sample being purged, ng
1,2-Dichloroethane-d_4	150
Dibromofluoromethane	150
Toluene-d_8	150
4-Bromofluorobenzene	150

Notes:
1) This results in a concentration of each surrogate in the sample of 6 µg/L for a 25 mL purge or 30 ug/kg for a 5 gram soil purge.
2) This is achieved by spiking 5 uL of a 30 ug/mL standard manually, or if an autosampler loop is used, the concentration of the solution is adjusted to the volume of the spiking loop.
3) Except for high level soils and waste, the surrogate and internal standards may be combined in one solution.
4) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 4A - Surrogate Standards (SIM)

Surrogate Compounds	Amount added to sample being purged, ng
Toluene-d_8	2.5
4-Bromofluorobenzene	2.5

Note: This results in a concentration of each surrogate in the sample of 0.1 µg/L for a 25 mL purge

Table 5 - BFB Key Ion Abundance Criteria (not required for SIM)

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% - 120% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 6 - SPCC Compounds and Minimum Response Factors

Compound	8260B Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 7 - CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for Calibration Verification
Vinyl Chloride	≤30	≤20
1,1-Dichloroethene	≤30	≤20
Chloroform	≤30	≤20
1,2-Dichloropropane	≤30	≤20
Toluene	≤30	≤20
Ethylbenzene	≤30	≤20

Table 8 - Quantitation Reference and Quantitation Ions***

Compound	IS Group	Primary*	Secondary	Tertiary
Dichlorodifluoromethane	1	85	87	50
Chloromethane	1	50	52	49
Vinyl chloride	1	62	64	61
1,3-Butadiene	1	39		
Bromomethane	1	94	96	
Chloroethane	1	64	66	49
Vinyl bromide	1	106		
Trichlorofluoromethane	1	101	103	66
2-Chloropropane	1	43		
1,1-Dichloroethene	1	96	61	98
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	1	151	101	153
Iodomethane	1	142	127	141
Carbon disulfide	1	76	78	
Acetone	1	43	58	
Acetonitrile	1	41	40	39
Methylene chloride	1	84	49	51
Acrylonitrile	1	53	52	51
trans-1,2-Dichloroethene	1	96	61	98
Methyl tert butyl ether	1	73		
Hexane	1	57	43	
1,1-Dichloroethane	1	63	65	83
Vinyl acetate	1	86	43	
cis-1,2-Dichloroethene	1	96	61	98
2,2-Dichloropropane	1	77	97	
2-Butanone	1	43	72**	
Tetrahydrofuran	1	42		
Bromochloromethane	1	128	130	49
Chloroform	1	83	85	47
1,1,1-Trichloroethane	1	97	99	117
Carbon tetrachloride	1	117	119	121
1,1-Dichloropropene	1	75	77	110
1,2-Dichloroethane	1	62	64	98
Benzene	1	78	52	77
Trichloroethene	1	130	95	97
1,2-Dichloropropane	1	63	65	41
Dibromomethane	1	93	174	95
Methyl methacrylate	1	41	69	100
Bromodichloromethane	1	83	85	129
cis-1,3-Dichloropropene	1	75	77	39
4-Methyl-2-pentanone	1 (2 for soil)	43	58	57
Toluene	2	91	92	65
trans-1,3-Dichloropropene	2	75	77	39
1,1,2-Trichloroethane	2	97	83	85
Tetrachloroethene	2	164 (166-SIM)	166	131
1,3-Dichloropropane	2	76	78	
2-Hexanone	2	43	58	57
Chlorodibromomethane	2	129	127	

Table 8 - Quantitation Reference and Quantitation Ions (continued)

Compound	IS Group	Primary*	Secondary	Tertiary
1,2-Dibromoethane	2	107	109	188
Chlorobenzene	2	112	114	77
1,1,1,2-Tetrachloroethane	2	131	133	119
Ethylbenzene	2	106	91	
m-Xylene and p-Xylen	2	106	91	
o-Xylene	2	106	91	
Styrene	2	104	103	78
Bromoform	2	173	171	175
Isopropylbenzene	2	105	120	
Cis-1,4-Dichloro-2-butene	2	53	88/89	
1,1,2,2-Tetrachloroethane	2	83	85	131
Bromobenzene	2	156	158	77
1,2,3-Trichloropropane	3	110	75	
Trans-1,4-Dichloro-2-butene	3	53	88/89	
n-Propylbenzene	3	91	120	
2-Chlorotoluene	3	91	126	
1,3,5-Trimethylbenzene	3	105	120	
4-Chlorotoluene	3	91	126	
tert-Butylbenzene	3	119	91	134
1,2,4-Trimethylbenzene	3	105	120	
sec-Butylbenzene	3	105	134	
1,3-Dichlorobenzene	3	146	148	111
p-isopropyltoluene	3	119	134	91
1,4-Dichlorobenzene	3	146	148	111
n- Butylbenzene	3	91	92	134
1,2-Dichlorobenzene	3	146	148	111
1,2-Dibromo-3-chloropropane	3	157	155	75
1,2,4-Trichlorobenzene	3	180	182	145
Hexachlorobutadiene	3	225	223	227
Naphthalene	3	128	129	127
1,2,3-Trichlorobenzene	3	180	182	145
Methyl Acetate	1	43	74	59
Cyclohexane	1	56	84	41
Methyl Cyclohexane	1	83	55	98
1,2-Dichloroethane-d_4 (Surrogate)	1	65	102	
Dibromofluoromethane (Surrogate)	1	113	111	
Toluene-d_8 (Surrogate)	2	98	70	100
4-Bromofluorobenzene (Surrogate)	3	95	174	176

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

***The Chrom software designates the nature of the ions as “Quant”, “Qual” or “Monitor”.

- Quant- i.e., quantitate; designates that ion for quantitation of the target analyte
- Qual – i.e., qualify; designates that ion that must be present in order to identify (or integrate) that analyte.
- Monitor- designates that ion as a monitor for additional qualitative analysis. The Chrom software does not use this mode to determine if a peak will be integrated.

Primary ions listed in this SOP shall be used in the “Quant” mode unless there are interferences present. In that case, a secondary ion is used. Secondary and tertiary ions can be designated as either “Qual” or “Monitor” mode.

Table 9 - Typical Water Calibration Levels (ug/L) - 25ml Purge

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Dichlorodifluoromethane	1	2	10	20	40	80
Chloromethane	1	2	10	20	40	80
Vinyl chloride	1	2	10	20	40	80
1,3-Butadiene	1	2	10	20	40	80
Bromomethane	1	2	10	20	40	80
Chloroethane	1	2	10	20	40	80
Vinyl bromide	1	2	10	20	40	80
Trichlorofluoromethane	1	2	10	20	40	80
2-Chloropropane	1	2	10	20	40	80
1,1-Dichloroethene	1	2	10	20	40	80
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	1	2	10	20	40	80
Iodomethane	1	2	10	20	40	80
Carbon disulfide	1	2	10	20	40	80
Acetone	4	8	40	80	160	320
Acetonitrile	10	20	100	200	400	800
Methylene chloride	1	2	10	20	40	80
Acrylonitrile	10	20	100	200	400	800
trans-1,2-Dichloroethene	1	2	10	20	40	80
Methyl tert butyl ether	1	2	10	20	40	80
Hexane	1	2	10	20	40	80
1,1-Dichloroethane	1	2	10	20	40	80
Vinyl acetate	2	4	20	40	80	160
cis-1,2-Dichloroethene	1	2	10	20	40	80
2,2-Dichloropropane	1	2	10	20	40	80
2-Butanone	4	8	40	80	160	320
Bromochloromethane	1	2	10	20	40	80
Tetrahydrofuran	2	4	20	40	80	160
Chloroform	1	2	10	20	40	80
1,1,1-Trichloroethane	1	2	10	20	40	80
Carbon tetrachloride	1	2	10	20	40	80
1,1-Dichloropropene	1	2	10	20	40	80
1,2-Dichloroethane	1	2	10	20	40	80
Benzene	1	2	10	20	40	80
Trichloroethene	1	2	10	20	40	80
1,2-Dichloropropane	1	2	10	20	40	80
Dibromomethane	1	2	10	20	40	80
Methyl methacrylate	1	2	10	20	40	80
Bromodichloromethane	1	2	10	20	40	80
cis-1,3-Dichloropropene	1	2	10	20	40	80
4-Methyl-2-pentanone	4	8	40	80	160	320
Toluene	1	2	10	20	40	80
trans-1,3-Dichloropropene	1	2	10	20	40	80
1,1,2-Trichloroethane	1	2	10	20	40	80
Tetrachloroethene	1	2	10	20	40	80
1,3-Dichloropropane	1	2	10	20	40	80
2-Hexanone	4	8	40	80	160	320
Chlorodibromomethane	1	2	10	20	40	80
1,2-Dibromoethane	1	2	10	20	40	80

Table 9 - Typical Water Calibration Levels (ug/L) - 25ml Purge (continued)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Methyl Acetate	5	10	50	100	200	400
Cyclohexane	1	2	10	20	40	80
Methyl Cyclohexane	1	2	10	20	40	80
Chlorobenzene	1	2	10	20	40	80
1,1,1,2-Tetrachloroethane	1	2	10	20	40	80
Ethylbenzene	1	2	10	20	40	80
m-Xylene and p-Xylene	1	2	10	20	40	80
o-Xylene	1	2	10	20	40	80
Styrene	1	2	10	20	40	80
Bromoform	1	2	10	20	40	80
Isopropylbenzene	1	2	10	20	40	80
cis-1,4-Dichloro-2-butene	1	2	10	20	40	80
1,1,2,2-Tetrachloroethane	1	2	10	20	40	80
Bromobenzene	1	2	10	20	40	80
1,2,3-Trichloropropane	1	2	10	20	40	80
trans-1,4-Dichloro-2-butene	1	2	10	20	40	80
n-Propylbenzene	1	2	10	20	40	80
2-Chlorotoluene	1	2	10	20	40	80
1,3,5-Trimethylbenzene	1	2	10	20	40	80
4-Chlorotoluene	1	2	10	20	40	80
tert-Butylbenzene	1	2	10	20	40	80
1,2,4-Trimethylbenzene	1	2	10	20	40	80
sec-Butylbenzene	1	2	10	20	40	80
1,3-Dichlorobenzene	1	2	10	20	40	80
p-isopropyltoluene	1	2	10	20	40	80
1,4-Dichlorobenzene	1	2	10	20	40	80
n- Butylbenzene	1	2	10	20	40	80
1,2-Dichlorobenzene	1	2	10	20	40	80
1,2-Dibromo-3-chloropropane	1	2	10	20	40	80
1,2,4-Trichlorobenzene	1	2	10	20	40	80
Hexachlorobutadiene	1	2	10	20	40	80
Naphthalene	1	2	10	20	40	80
1,2,3-Trichlorobenzene	1	2	10	20	40	80
1,2-Dichloroethane-d_4 (Surrogate)	6	6	6	6	6	6
Dibromofluoromethane (Surrogate)	6	6	6	6	6	6
Toluene-d_8 (Surrogate)	6	6	6	6	6	6
4-Bromofluorobenzene (Surrogate)	6	6	6	6	6	6

Table 9A - Typical Water Calibration Levels (ug/L) - 25ml Purge (SIM)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Tetrachloroethene	0.004	0.01	0.025	0.1	0.5	1.0
Chlorobenzene	0.004	0.01	0.025	0.1	0.5	1.0
Toluene-d8 (Surrogate)	0.1	0.1	0.1	0.1	0.1	0.1
4-Bromofluorobenzene (Surrogate)	0.1	0.1	0.1	0.1	0.1	0.1

Table 10 - Typical Soil Calibration Levels (ug/kg) – Low Level

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Dichlorodifluoromethane	5	10	50	100	250	400
Chloromethane	5	10	50	100	250	400
Vinyl chloride	5	10	50	100	250	400
1,3-Butadiene	5	10	50	100	250	400
Bromomethane	5	10	50	100	250	400
Chloroethane	5	10	50	100	250	400
Vinyl bromide	5	10	50	100	250	400
Trichlorofluoromethane	5	10	50	100	250	400
2-Chloropropane	5	10	50	100	250	400
1,1-Dichloroethene	5	10	50	100	250	400
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	5	10	50	100	250	400
Iodomethane	5	10	50	100	250	400
Carbon disulfide	5	10	50	100	250	400
Acetone	20	40	200	400	1000	1600
Acetonitrile	50	100	500	1000	2500	4000
Methylene chloride	5	10	50	100	250	400
Acrylonitrile	50	100	500	1000	2500	4000
trans-1,2-Dichloroethene	5	10	50	100	250	400
Methyl tert butyl ether	5	10	50	100	250	400
Hexane	5	10	50	100	250	400
1,1-Dichloroethane	5	10	50	100	250	400
Vinyl acetate	10	20	100	200	500	800
cis-1,2-Dichloroethene	5	10	50	100	250	400
2,2-Dichloropropane	5	10	50	100	250	400
2-Butanone	20	40	200	400	1000	1600
Bromochloromethane	5	10	50	100	250	400
Tetrahydrofuran	10	20	100	200	500	800
Chloroform	5	10	50	100	250	400
1,1,1-Trichloroethane	5	10	50	100	250	400
Carbon tetrachloride	5	10	50	100	250	400
1,1-Dichloropropene	5	10	50	100	250	400
1,2-Dichloroethane	5	10	50	100	250	400
Benzene	5	10	50	100	250	400
Trichloroethene	5	10	50	100	250	400
1,2-Dichloropropane	5	10	50	100	250	400
Dibromomethane	5	10	50	100	250	400
Methyl methacrylate	5	10	50	100	250	400
Bromodichloromethane	5	10	50	100	250	400
cis-1,3-Dichloropropene	5	10	50	100	250	400
4-Methyl-2-pentanone	20	40	200	400	1000	1600
Toluene	5	10	50	100	250	400
trans-1,3-Dichloropropene	5	10	50	100	250	400
1,1,2-Trichloroethane	5	10	50	100	250	400
Tetrachloroethene	5	10	50	100	250	400
1,3-Dichloropropane	5	10	50	100	250	400
2-Hexanone	20	40	200	400	1000	1600
Chlorodibromomethane	5	10	50	100	250	400
1,2-Dibromoethane	5	10	50	100	250	400
Chlorobenzene	5	10	50	100	250	400

Table 10 - Typical Soil Calibration Levels (ug/kg) – Low Level (continued)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
1,1,1,2-Tetrachloroethane	5	10	50	100	250	400
Ethylbenzene	5	10	50	100	250	400
m-Xylene and p-Xylene	5	10	50	100	250	400
o-Xylene	5	10	50	100	250	400
Styrene	5	10	50	100	250	400
Bromoform	5	10	50	100	250	400
Isopropylbenzene	5	10	50	100	250	400
cis-1,4-Dichloro-2-butene	5	10	50	100	250	400
1,1,2,2-Tetrachloroethane	5	10	50	100	250	400
Bromobenzene	5	10	50	100	250	400
1,2,3-Trichloropropane	5	10	50	100	250	400
trans-1,4-Dichloro-2-butene	5	10	50	100	250	400
n-Propylbenzene	5	10	50	100	250	400
2-Chlorotoluene	5	10	50	100	250	400
1,3,5-Trimethylbenzene	5	10	50	100	250	400
4-Chlorotoluene	5	10	50	100	250	400
tert-Butylbenzene	5	10	50	100	250	400
1,2,4-Trimethylbenzene	5	10	50	100	250	400
sec-Butylbenzene	5	10	50	100	250	400
1,3-Dichlorobenzene	5	10	50	100	250	400
p-isopropyltoluene	5	10	50	100	250	400
1,4-Dichlorobenzene	5	10	50	100	250	400
n- Butylbenzene	5	10	50	100	250	400
1,2-Dichlorobenzene	5	10	50	100	250	400
1,2-Dibromo-3-chloropropane	5	10	50	100	250	400
1,2,4-Trichlorobenzene	5	10	50	100	250	400
Hexachlorobutadiene	5	10	50	100	250	400
Naphthalene	5	10	50	100	250	400
1,2,3-Trichlorobenzene	5	10	50	100	250	400
1,2-Dichloroethane-d_4 (Surrogate)	30	30	30	30	30	30
Dibromofluoromethane (Surrogate)	30	30	30	30	30	30
Toluene-d_8 (Surrogate)	30	30	30	30	30	30
4-Bromofluorobenzene (Surrogate)	30	30	30	30	30	30

Appendix A: Example Data Review Checklist

TestAmerica Knoxville GC/MS Initial Calibration Review/Narrative Checklist
Methods: 8260B--KNOX-MS-0015 Rev 18 and VOST—KNOX-MS-0011 Rev 13

Instrument:			TALS Batch / Event #	8260B LL	8260B	Scanned □
Analysis Date:			List 1:	/	/	
Chrom WL #			List 2:	/	/	
			Other:	/	/	

Chrom Worklist/Peak Review	1st	*Comments*	2nd
1. Re-read each limit group [method editor-limit groups]			
2. Verify LOD [method editor -> edit -> MDL]			
3. Are the reagents & init./final vol. correct [Sample & Run Reagents]			
4. First levels "unlock/clear" or "unlock/clear by sublist" as appropriate?			
5. Are the Cal Levels & groups correct in WL?			
6. Did the BFB meet the tune criteria (NA for SIM) [F8]			
7. Were all standards injected within 12 hr of BFB (or 1st injection for SIM? [F7]			
8. Was the high point std checked for saturation [flags + visible inspection: $8.4x10^6$]			
9. If manual integrations were performed, are they appropriate with proper reason given?			
10. Were all peaks identified automatically? If not, list analytes: _____		*Modify method for detection must be attempted and all points reprocessed. Any non-detected peaks must be verified in each affected sample.*	
11. Elution order checked on isomeric pairs?			
• chlorobenzene-d5 and 1,1,1,2-tetrachloroethane			
• trichlorofluoromethane and Freon 113			
• hexane and vinyl acetate			
• cis- and trans- isomers			
• 1,1,1-TCA and CCL4			
• ethyl benzene / m/p-xylene / o-xylene			
• n-propylbenzene / 2-chlorotoluene / 4-chlorotoluene			
• 1,3,5-trimethylbenzene / 1,2,4-trimethylbenzene / secbutylbenzene			
• 1,3- , 1,4- , and 1,2-dichlorobenzene			
• 1,2,4 and 1,2,3 -trichlorobenzene			
12. ICAL start/end date/time correct on summary? [F6]			
13. Are ≥ 5 levels of each compound/surrogate active? [F6]			
14. Is low level standard at or below RL & points consecutive? [F6]			
15. Is %RSD ≤30% for all CCC's? [F6]			
16. Do the average RFs for SPCC's meet min. RF? [F6]			
17. Was a linear or quadratic fit used for analytes > 15% RSD? [F6]			
18. If curves were used, is correlation coefficient ≥0.990? [F6]			
19. At least 6 consecutive points used for quadratic curves? [F6]			
20. For quadratic: is a tangent's slope to the curve entirely positive or negative and continuous? [Cntrl-C, details]			
21. Is the intercept < RL for each curve? [Cntrl-C, details]			
22. Is the readback for each point within criteria? [F6-Drift] (<40% for all points, except low point < 50 %)			
23. Was the ICV± 30% recovery? [F8-icv]		ICV out, smp ND (NCM#_____)	
1st level reviewer:	**Date:**	**2nd level reviewer:**	**Date:**

TALS MLG Review	1st	Comments	2nd
24. Upload ICAL & confirm graphics uploaded [Sample List Tab]			
25. All points are in the most recent active calibration event #? [Calibration ID # in the sample results tab & Calibration Events] [Calibration Events - 'Fix ICAL Linkage' if needed]			
26. Runs linked to BFB? [QC Links]			
27. Run Data Review Checker and acknowledge findings.			
28. If criteria not met, was a NCM generated?			
29. After review in TALS, approve the calibrations in TALS			
30. After verifying TALS is correct, lock method in Chrom <resolve errors>			
31. Checklist scanned & attached properly?			
1st level reviewer:	**Date:**	**2nd level reviewer:**	**Date:**
Comments:			

Appendix A: Example Data Review Checklist, continued

TestAmerica Knoxville GC/MS CCAL/Batch Review/Narrative Checklist
Methods: 8260B--Knox-MS-0015 Rev 18 and VOST—KNOX-MS-0011 Rev 13

8260BLL ☐ 8260B ☐

Instrument			ICAL Chrom WL #	ICAL TALS Batch / Event #	Scanned ☐
Analysis Date:		List 1:			
CCAL Chrom WL #		List 2:			
CCAL TALS Batch #		Other:			
Prep batches:					

CCV Chrom/Worklist Review	1st	*Comments/NCM #*	2nd
1. Are the reagents & init/final volumes correct? (Verify reagents & amt. injected) [WL Sample Reagent Tab]			
2. Are all required calibration standards in worklist?			
3. Did the BFB meet tune criteria? (NA for SIM) [F8]			
4. Was the CCAL compared to the most recent & correct ICAL for each CCV? (verify ICAL batch #, start/end Cal date & time) [F8]			
5. Elution order checked on isomeric pairs/coeluters?			
• chlorobenzene-d5 and 1,1,1,2-tetrachloroethane			
• trichlorofluoromethane and Freon 113			
• hexane and vinyl acetate			
• cis- and trans- isomers			
• 1,1,1-TCA and CCL4			
• ethyl benzene / m/p-xylene / o-xylene			
• n-propylbenzene / 2-chlorotoluene / 4-chlorotoluene			
• 1,3,5-trimethylbenzene / 1,2,4-trimethylbenzene / secbutylbenzene			
• 1,3- , 1,4- , and 1,2-dichlorobenzene			
• 1,2,4 and 1,2,3 -trichlorobenzene			
6. Manual integrations properly performed, correctly ID'd, baseline clearly identified, and correct reason given?		*Note: manual selections should be updated and all data reprocessed*	
7. Were all peaks identified automatically? *If not, list analytes:*		*Note: any non-detected peaks must be verified in each affected sample.*	
8. Is %RSD ≤ 20% for all CCC's? *[F8] or TALS sample tab*		☐ CCV high, sample ND (NCM#_____)	
9. Do the RFs for SPCC's meet min. RF? [F8]			
10. Is the %D or drift ≤50% for all other compounds? [F8]		☐ CCV high, sample ND (NCM#_____)	
11. Are the internal standard retention times within limits for each CCV? (±30 seconds of the mid-level ICAL standard) [F8-istd]			
12. Are the internal standard responses within limits for each CCV? (50-200% of the mid-level ICAL standard) [F8-istd]			
13. Has the retention time been updated to the method?			

Sample Worklist/Chrom review	*1st*	*Comments/NCM #*	*2nd*
13.For solid and methanol extractions, were the prep batches dates/times/weights/volumes correct and 2nd level reviewed?			
14.Verify initial weights/volumes in Worklist			
15.Were all samples injected within 12 hrs. of first BFB? [F7]			
16.Have the sample ID's and dilution factors been confirmed (check sequence, autosampler positions, etc.)?			
17.Are all analytes in the method blank/instrument blank < RL?		☐ MB CLC <5x RL (NCM#_____) ☐ MB Rpt ND (NCM#_____) ☐ MB-Rpt. 10 x (NCM#_____) ☐ MB-insuff samp (NCM#_____) ☐ MB-insuff samp - CONSUMED (NCM#_____) ☐ MB RX/HT out (NCM#_____)	
18.Method blank Surrogate recoveries within QC limits? [F8] or [Batch Results SUR Tab]		☐ Surr-MB (1) high (NCM#_____) + (2) smp OK (NCM#_____) or (3) Insuff. sample (NCM#_____) or (4) CONSUMED (NCM#_____)	
19.MS/MSD done per batch? *('NA' for air samples)*		☐ MS/MSD/DUP- insufficient vol (2) (NCM#_____)	

* Such action must be taken in consultation with client.

Appendix A: Example Data Review Checklist, continued

TestAmerica Knoxville GC/MS CCAL/Batch Review/Narrative Checklist
Methods: 8260B--Knox-MS-0015 Rev 18 and VOST—KNOX-MS-0011 Rev 13

20.LCS criteria met for IDA and natives? [Chrom-F8] [TALS-Sample Results Tab] Number of target analytes in LCS / # marginal exceedances of LCS control limits allowed >90: 5 71 - 90: 4 51 - 70: 3 31 - 50: 2 11 - 30: 1 < 11: 0		□ LCS/D-Insuff smp (NCM#_____) □ LCS/D-Insuff smp - CONSUMED (NCM#_____) □ LCS/D %R High < RL in smp (1+2-5) (NCM#_____) □ LCS/D out-RX HT out (NCM#_____) □ LCS/D-%RPD (%R OK) (NCM#_____) □ Marg. Exceed. w/in ME limits & Random (NCM#_____)	
21.All runs - peaks ID'd correctly and false positives removed? Sample / Reason / Sample / Reason		□ Interf-Chrom: (NCM#_____) □ "cn" (see narrative): (NCM#_____)	
22.Manual integrations properly performed, correctly ID'd, baseline clearly identified, and correct reason given?		□ "cn" (see narrative): (NCM#_____)	
23.Are surrogate recoveries within QC limits? [Batch Results SUR Tab] If no, list samples, reason and NCM # Sample / Reason / Sample / Reason		□ Surr-High-ND (1,8) (NCM#_____) □ Surr-Incorrect Spike amt (NCM#_____) □ Surr-Insuff Smp (1) insuff smp (NCM#_____) or (4) CONSUMED (NCM#_____) + (2) low bias (NCM#_____) or (3) high bias (NCM#_____) □ Surr-Insuff Smp - CONSUMED (NCM#_____) □ Surr-Matrix (1-4) (NCM#_____) □ Surr-Rpt per Client (released w/o further investigation)* (NCM#_____) □ Surr-RX concur (NCM#_____) □ Surr-RX pass (RX outside HT, but was within limits) (NCM#_____) □ Surr-Spike Omitted Insuff Smp (NCM#_____) □ NCM#: **140-6061**: Ph >10 surr DBFM out	
24.Are internal standards within QC limits? [Batch Results IS Tab] If no, list samples, reason and NCM # Sample / Reason / Sample / Reason		□ ISTD- Matrix (NCM#_____) □ ISTD- DL (NCM#_____) □ ISTD-No RX/RR [insuff sample] (NCM#_____) □ ISTD-No RX/RR [insuff sample- CONSUMED] (NCM#_____) □ ISTD- non-targets (NCM#_____) □ ISTD- RX/RA concur (NCM#_____) □ NCM#: **140-5756**; Samples bracketed by acceptable runs*.	
TALS Review	***1st***	***Comments/NCM #***	***2nd***
25.Graphics uploaded?			
26.Sample special instructions verified?			
27.Is the calibration event # correct for each CCV/samples? [TALS Sample Results tab]			
28.For initial analysis with elevated RL's, explain: *List diluted samples and reason:* Sample / Reason / Sample / Reason		□ RL – Dil Screen (NCM#_____) □ RL – Dil Non-targets (NCM#_____) □ RL – Dil Targets (NCM#_____) □ RL – Final Vol (NCM#_____) □ RL – Init Vol (matrix) (NCM#_____) □ RL – Insuff. Smp (NCM#_____) □ RL – VOA,Dil,Foaming (NCM#_____)	
29.Was pH ≤ 2 for all water samples? [Noted on run log - transcribed to TALS worksheet tab]		□ HT-Preserv-VOA pH>2 (>7 days) (NCM#_____) □ Preserv-VOA insuff; correct bottle (5) (≤7 days) (NCM#_____)	
30.Suffixes assigned properly (DL/RE)? [Sample List Tab]			
31.Each job has QC created (CCV,MB,LCS)? [Sample List Tab]			
32.Analytes over calibration range set to secondary & dilutions scheduled for analysis? [Conditions Review Tab] Sample / Reason / Sample / Reason		□ ICAL-Range Exceed; Min Dil 1 – client OK'd* (NCM#_____) 2 – dil for other cmpds (NCM#_____) 3 – sample consumed (NCM#_____) □ ICAL-Range Exceed; *VOST ONLY*: Saturation + sample consumed (NCM#_____)	
33.For dilutions, diluted analytes set to primary, others to "Acceptable"? [Sample Results Tab]			
34.Sample prep and analyses done within preparation and analytical holding time (HT)?			

* Such action must be taken in consultation with client.

Appendix A: Example Data Review Checklist, continued

TestAmerica Knoxville GC/MS CCAL/Batch Review/Narrative Checklist
Methods: 8260B--Knox-MS-0015 Rev 18 and VOST—KNOX-MS-0011 Rev 13

35.If MS/MSD was done on this client's sample or for reported batch QC, were the MS/MSD recoveries and RPDs within laboratory generated QC limits?		□ MS/MSD - %R out, LCS OK □ MS/MSD - high targets □ MS/MSD – spk/surr diluted out □ MS/MSD/Dup - % RPD	
36.Verify reagents have not expired [Reagents Tab]			
37.Samples not reported set to "Acceptable" or "Rejected" [Sample Results Tab]			
38.Water samples received w/no headspace? Samples:		□ Headspace-Lim Vol (> 6 mm bubble) (NCM#_____)	
39.Encore/Terracore issues OK?		□ Encore-Effervesced 5035 (NCM#_____) □ Encore-not sealed (NCM#_____) □ See HT on job checklist	
40.BFB/CCV linked to all samples? [QC Links]			
41.Samples linked to correct MB & LCS/D & MS/D? [QC Links]			
42.Correct ICV's linked to all samples? [QC Links]			
43.Run Data Review Checker and acknowledge findings.			
44.Runs set to 1st level review?		Runs set to 2nd level review?	
45.QC checker run for batch and items addressed			
46.Checklist scanned & attached properly?			

1st level:	Date	2nd level:	Date
Comments:			
Example calculation:			

* Such action must be taken in consultation with client.

Appendix A: Example Data Review Checklist, continued

TestAmerica Knoxville GC/MS Job Review/Narrative Checklist
Methods: 8260B--Knox-MS-0015 Rev 18 and VOST—KNOX-MS-0011 Rev 13

Job #:_____

Method:	**8260B LL** □	**8260B** □

TALS Analytical Batch #'s:

ICAL Instrument:						
Batch #:						

CCAL batch #'s:						

Prep methods

Prep batch #'s:						

Job / Reports Review	*1st*	*Comments/NCM/Narrative notes*	*2nd*
1.QC checker run for job and items addressed			
2.Were all client special project requirements met?		□ Non-routine analyte-no MDL: (NCM_____)	
3.Sample prep and analyses done within analytical holding time? *If no, list samples and NCM #:* Sample Reason Sample Reason _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____		□ HT Init Analy: (NCM#_____) □ HT Insuff time: (NCM#_____) □ HT Re-Analysis: (NCM#_____) □ HT Receipt: (NCM#_____) □ HT RePrep: (NCM#_____) □ HT Rct;VOA Freeze (>48 hr): (NCM#_____)	
1.Appropriate NCM's on the right are assigned to the job?		□ NCM#: **140-6074**: VOST-0030 T & T/C □ NCM#: **140-6139:** Broken VOST tube □ NCM#: **140-6076**: Waste □ NCM#: **140-6081**: Micro-trap VOST □ NCM#: **140-6080**: Methanol VOST (20 mL) □ NCM#: **140-6066:** Impingers □ NCM#: **140-6116:** Refrigerants	
2.All NCM's are complete, free of grammatical/spelling errors, and correct references to batches/samples/analytes?		List all other NCM's with job:	
3.Run deliverable & assemble report.			
4.All "E" values have "DL" runs? (if no, NCM #)		If 'no', create NCM on analytical batch	
5.Data Summaries correct (L2 & L4)?			
6.Forms II-V & VIII correct (L4)?			
7.ICV present per ICAL (L4)?			
8.Checklists (ICAL & batch DRC) attached to Job (L4)?			

1st Level:	Date:	2nd Level:	Date:

* Such action must be taken in consultation with client.

FIGURE 1 – Example GC and GC/MS Conditions for SIM.

```
                              TOPLEVEL PARAMETERS
                              -------------------

Method Information For: C:\MSDCHEM\1\METHODS\SIM.M
Method Sections To Run:

  ( ) Save Copy of Method With Data
  ( ) MSTOP               Pre-Run Cmd/Macro  =
  ( ) Instrument Control  Pre-Run Cmd/Macro  =
  ( ) Data Analysis       Pre-Run Cmd/Macro  =
  (X) Data Acquisition
  ( ) Data Analysis
  ( ) MSTOP               Post-Run Cmd/Macro =
  ( ) Instrument Control  Post-Run Cmd/Macro =
  ( ) Data Analysis       Post-Run Cmd/Macro =
Method Comments:
  VOST/CONDENSATE SIM METHOD


                              END OF TOPLEVEL PARAMETERS
                              --------------------------




                              INSTRUMENT CONTROL PARAMETERS
                              -----------------------------

================================================================================
                               6890 GC METHOD
================================================================================

OVEN
   Initial temp:  35 'C (On)                Maximum temp:  230 'C
   Initial time:  2.00 min                  Equilibration time:  0.10 min
   Ramps:
      #  Rate  Final temp  Final time
      1 17.00      124        2.25
      2 21.00      200        1.00
      3   0.0(Off)
   Post temp:  0 'C
   Post time:  0.00 min
   Run time:  14.10 min

FRONT INLET (SPLIT/SPLITLESS)           BACK INLET (UNKNOWN)
   Mode:  Split
   Initial temp:  200 'C (On)
   Pressure:  21.66 psi (On)
   Split ratio:  30:1
   Split flow:  41.0 mL/min
   Total flow:  45.1 mL/min
   Gas saver:  On
   Saver flow:  15.0 mL/min
   Saver time:  10.00 min
   Gas type:  Helium

COLUMN 1                                COLUMN 2
   Capillary Column                        Capillary Column
   Model Number:  Agilent 121-1324         Model Number:  Agilent 121-1324
   DB-624, 0.18mm * 20m * 1um              DB-624, 0.18mm * 20m * 1um
   Max temperature:  260 'C                Max temperature:  260 'C
   Nominal length:  19.0 m                 Nominal length:  20.0 m
   Nominal diameter:  180.00 um            Nominal diameter:  180.00 um
   Nominal film thickness:  10.00 um       Nominal film thickness:  10.00 um
   Mode:  constant flow                    Mode:  (see column 1)
   Initial flow:  0.7 mL/min               Nominal initial flow:  0.8 mL/min
   Nominal init pressure:  22.64 psi       Nominal init pressure:  22.64 psi

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```

FIGURE 1 – Example GC and GC/MS Conditions for SIM. (Continued)

```
   Average velocity:  31 cm/sec              Average velocity:  39 cm/sec
   Inlet:  Front Inlet                       Inlet:  Front Inlet
   Outlet:  MSD                              Outlet:  MSD
   Outlet pressure:  ambient                 Outlet pressure:  vacuum

FRONT DETECTOR ()                         BACK DETECTOR ()

SIGNAL 1                                  SIGNAL 2
   Data rate:  20 Hz                         Data rate:  20 Hz
   Type:  test plot                          Type:  test plot
   Save Data:  Off                           Save Data:  Off
   Zero:  0.0 (Off)                          Zero:  0.0 (Off)
   Range:  0                                 Range:  0
   Fast Peaks:  Off                          Fast Peaks:  Off
   Attenuation:  0                           Attenuation:  0

COLUMN COMP 1                             COLUMN COMP 2
   (No Detectors Installed)                  (No Detectors Installed)

THERMAL AUX 1
   Use:  MSD Transfer Line Heater
   Description:
   Initial temp:  250 'C (On)
   Initial time:  0.00 min
      #  Rate  Final temp  Final time
      1   0.0(Off)

                                          POST RUN
                                             Post Time: 0.00 min

TIME TABLE
   Time       Specifier                      Parameter & Setpoint

                              7673 Injector


     Front Injector:
        Sample Washes                0
        Sample Pumps                 6
        Injection Volume           1.0 microliters
        Syringe Size              10.0 microliters
        Nanoliter Adapter          Off
        PostInj Solvent A Washes     0
        PostInj Solvent B Washes     0
        Viscosity Delay              0 seconds
        Plunger Speed             Fast

     Back Injector:
        Sample Washes                0
        Sample Pumps                 6
        Injection Volume           1.0 microliters
        Syringe Size              10.0 microliters
        Nanoliter Adapter          Off
        PostInj Solvent A Washes     0
        PostInj Solvent B Washes     0
        Viscosity Delay              0 seconds
        Plunger Speed             Fast

 Column 1 Inventory Number : DB-624
 Column 2 Inventory Number :

                               MS ACQUISITION PARAMETERS


General Information
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Tune File               : bfb.u
Acquistion Mode         : SIM

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```

FIGURE 1 – Example GC and GC/MS Conditions for SIM. (Continued)

```
MS Information
-- -----------

Solvent Delay            : 5.00 min

EM Absolute              : False
EM Offset                : 0
Resulting EM Voltage     : 1541.2

[Sim Parameters]

GROUP 1
Group ID                 : 3
Resolution               : Low
Plot 1 Ion               : 98.0
Ions/Dwell In Group      (  Mass,  Dwell) (  Mass,  Dwell)
                         (  98.0,     50) ( 100.0,     50)

GROUP 2
Group ID                 : B
Resolution               : Low
Group Start Time         : 5.80
Plot 1 Ion               : 131.0
Ions/Dwell In Group      (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,  Dwell)
                         ( 131.0,     40) ( 164.0,     40) ( 166.0,    200)

GROUP 3
Group ID                 : 4
Resolution               : Low
Group Start Time         : 6.40
Plot 1 Ion               : 112.0
Ions/Dwell In Group      (  Mass,  Dwell) (  Mass,  Dwell) (  Mass,  Dwell)
                         (  77.0,     50) (  82.0,     25) ( 112.0,    200)
                         ( 114.0,     50) ( 117.0,     25)

GROUP 4
Group ID                 : C
Resolution               : Low
Group Start Time         : 7.60
Plot 1 Ion               : 95.0
Ions/Dwell In Group      (  Mass,  Dwell) (  Mass,  Dwell)
                         (  95.0,     50) ( 174.0,    100)

GROUP 5
Group ID                 : 6
Resolution               : Low
Group Start Time         : 8.50
Plot 1 Ion               : 152.0
Ions/Dwell In Group      (  Mass,  Dwell) (  Mass,  Dwell)
                         ( 150.0,     50) ( 152.0,     50)

[MSZones]

MS Quad                  : 150 C   maximum 200 C
MS Source                : 230 C   maximum 250 C

                             END OF MS ACQUISITION PARAMETERS

PostRun InstCntl macro(s) exist: msacq2.mac


                           END OF INSTRUMENT CONTROL PARAMETERS
                           -----------------------------------


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```