VOLATILE ORGANICS GAS CHROMATOGRAPHY / MASS SPECTROMETRY

Note: Make enough copies of Pages 1-12 to assess each test method in use at the laboratory, one method at a time

CHEMISTRY TEST METHOD EVALUATED: _____________________________

___ 5.5.4.1.2(a) Does the laboratory have an in-house methods manual for each accredited analyte or method
Note: This manual may consist of copies of published or referenced test methods

___ 5.5.4.1.2(b) Does the laboratory clearly indicate in its methods manual any modifications made to the referenced test method and describe any changes or clarifications where the referenced test method is ambiguous or provides insufficient detail

Does each test method in the in-house methods manual include or reference, where applicable:

___ 5.5.4.1.2(b)(1) Identification of the test method
___ 5.5.4.1.2(b)(2) Applicable matrix or matrices
___ 5.5.4.1.2(b)(3) Method Detection Limit
___ 5.5.4.1.2(b)(4) Scope & application, including components to be analyzed
___ 5.5.4.1.2(b)(5) Summary of the test method
___ 5.5.4.1.2(b)(6) Definitions
___ 5.5.4.1.2(b)(7) Interferences
___ 5.5.4.1.2(b)(8) Safety
___ 5.5.4.1.2(b)(9) Equipment & supplies
___ 5.5.4.1.2(b)(10) Reagents & standards
___ 5.5.4.1.2(b)(11) Sample collection, preservation, shipment, & storage
___ 5.5.4.1.2(b)(12) Quality control
___ 5.5.4.1.2(b)(13) Calibration & standardization
___ 5.5.4.1.2(b)(14) Procedure
___ 5.5.4.1.2(b)(15) Calculations
___ 5.5.4.1.2(b)(16) Method performance
___ 5.5.4.1.2(b)(17) Pollution prevention
___ 5.5.4.1.2(b)(18) Data assessment & acceptance criteria for quality control measures
___ 5.5.4.1.2(b)(19) Corrective actions for out-of-control data
___ 5.5.4.1.2(b)(20) Contingencies for handling out-of-control or unacceptable data
___ 5.5.4.1.2(b)(21) Waste management
___ 5.5.4.1.2(b)(22) References
___ 5.5.4.1.2(b)(23) Tables, diagrams, flowcharts, validation data

___ D Does the laboratory ensure that the essential standards outlined in Appendix D are incorporated into the method manuals and/or Quality Manual

COMMENTS:
CHEMISTRY TEST METHOD EVALUATED: ________________________________

5.5.2.2 Do the laboratory's initial & continuing instrument calibration verifications meet the requirements in mandated test methods & regulations (see pages 13-14 for acceptance criteria and the number of standards required)

Note: If it is not apparent which standard is more stringent, then the requirements of the regulation or the mandated test method are to be followed.

5.5.2.1(a) Does the laboratory's test method SOP include or reference details of the initial instrument calibration procedures

Note: This includes calculations, integrations, & associated statistics

Note: If the test method is referenced for initial instrument calibration procedures, the laboratory must have this method & make it available for review.

5.5.2.1(b) Does the laboratory retain sufficient raw data records to permit reconstruction of the initial instrument calibration

Note: Examples of such data records include calibration date, test method, instrument, analysis date, each analyte name, analyst initials or signature, concentration & response, calibration curve or response factor, and unique equation or coefficient used to reduce instrument responses to concentration.

5.5.2.1(c) Does the laboratory quantitate sample results only from the initial instrument calibration and not from any continuing instrument calibration verifications, unless required by regulation, method, or program

5.5.2.1(d) Does the laboratory verify all initial instrument calibrations with a standard obtained from a second manufacturer or lot if the lot can be demonstrated from the manufacturer as prepared independently from other lots

Note: When commercially available, traceability shall be to a national standard.

5.5.2.1(e) Has the laboratory established criteria for the acceptance of an initial instrument calibration

Note: Examples include linear regression correlation coefficient & response factor %RSD

Note: The acceptance criteria must be appropriate to the calibration technique employed.

5.5.2.1(f) For purposes of establishing the working calibration range, is the lowest calibration standard concentration the lower limit of quantitation

5.5.2.1(f) Is all data reported below the lower limit of quantitation reported using defined qualifiers or flags or explained in the case narrative.

5.5.2.1(g) Is the highest calibration standard the highest concentration for which quantitative data are to be reported

5.5.2.1(g) Is all data reported above the highest calibration standard reported using defined qualifiers or flags or explained in the case narrative

5.5.2.1(h) Does the laboratory report measured concentrations outside the working calibration range as having less certainty & using defined qualifiers or flags or explained in the case narrative

5.5.2.1(h) Is the lowest calibration standard above the limit of detection for each analyte
Note: For instrument technologies (e.g., ICP, ICP/MS) with validated techniques from manufacturers or methods employing standardization with a zero point & a single-point calibration std., the following must occur:

5.5.5.2.2.1(h)(1) Prior to the analysis of samples, are the zero point & single point calibration analyzed, and the linear range of the instrument established by analyzing a series of standards, one of which must be at the lowest quantitation level.

Note: Sample results within the established linear range will not require data qualifier flags.

5.5.5.2.2.1(h)(2) Are the zero point & single point calibration standard analyzed with each analytical batch.

5.5.5.2.2.1(h)(3) Is a standard corresponding to the limit of quantitation analyzed with each analytical batch & meet established acceptance criteria.

5.5.5.2.2.1(h)(4) Is the linearity verified at a frequency established by the test method and/or the manufacturer.

5.5.5.2.2.1(i) Does the laboratory perform corrective actions & reanalyze all associated samples if the initial instrument calibration results are outside established acceptance criteria.

5.5.5.2.2.1(i) When reanalysis is not possible, does the laboratory report sample data associated with unacceptable initial instrument calibrations with appropriate data qualifiers.

Note: NELAC Standards 5.5.5.2.2.1(h) & (i) may need to be assessed in conjunction with the Quality Systems data audit.

5.5.5.2.2.1(j) Does the laboratory have a standard operating procedure for determining the number of points for establishing the initial instrument calibration.

5.5.5.2.2.1(j) Does the laboratory use a minimum of two calibration standards (not including blanks or a zero standard) for performing an initial instrument calibration.

Note: This Standard applies if a reference or mandated method does not specify the number of calibration standards.

Note: One of the standards must be at the limit of quantitation.

Note: This Standard does not apply to instrument technologies for which it has been established by methodologies & procedures that a zero & a single point standard are appropriate for calibrations (see Section 5.5.5.2.2.1(h)).

COMMENTS:

5.5.5.10 Does the laboratory verify the validity of the initial calibration by a continuing instrument calibration verification with each analytical batch, prior to sample analyses, whenever an initial instrument calibration is not performed on the day of analysis.

5.5.5.10(a) Are the details of the continuing instrument calibration verification procedure, calculations, & associated statistics included or referenced in the test method SOP.

5.5.5.10(b) Is calibration verified for each compound, element, or other discrete chemical species.

Note: For multi-component analytes such as Aroclors, Total Petroleum Hydrocarbons, or Toxaphene, a representative chemical related substance or mixture can be used.
5.5.5.10(c)(1) Is the instrument calibration verification performed at the beginning & end of each analytical batch  
Note: Only one verification needs to be performed at the beginning of the analytical batch if an internal standard is used

5.5.5.10(c)(2) Is the instrument calibration verification performed whenever it is expected that the analytical system may be out of calibration or might not meet the verification acceptance criteria

5.5.5.10(c)(3) Is the instrument calibration verification performed if the time period for calibration or the most previous calibration verification has expired

5.5.5.10(c)(4) Is the instrument calibration verification performed for analytical systems that contain a calibration verification requirement

5.5.5.10(d) Does the laboratory retain sufficient raw data records to permit reconstruction of the continuing instrument calibration verification  
Note: Such records include test method, instrument, analysis date, name of each analyte, concentration & response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations

5.5.5.10(d) Does the laboratory’s continuing calibration verification records explicitly connect the continuing verification data to the initial instrument calibration

5.5.5.10(e) Has the laboratory established criteria for the acceptance of a continuing instrument calibration verification (e.g. relative percent difference)

5.5.5.10(e) Does the laboratory perform corrective actions if the continuing instrument calibration verification results are outside established acceptance criteria

5.5.5.10(e) Does the laboratory perform a new initial instrument calibration if the routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria  
Note: Alternatively, the laboratory can demonstrate acceptable performance after correction with 2 consecutive calibration verifications

5.5.5.10(e) If the laboratory has not verified calibration, do sample analyses not occur until the analytical system is calibrated or calibration verified  
Note: For sample data associated with an unacceptable calibration verification, the results must be flagged but the data may be usable under the following special conditions:  
- Non-detects for analytes in associated samples where the acceptance criteria for the continuing calibration verifications are exceeded high  
- Any test result for an analyte that indicates exceedence of a maximum regulatory limit or decision level, when the acceptance criteria for the continuing calibration verification for that analyte is exceeded low  
Any samples with test results that do not meet either of the above criteria must be re-analyzed after a new initial instrument calibration has been established, evaluated, & accepted
CHEMISTRY TEST METHOD EVALUATED: ______________________________

5.5.4.2.2(a) C.1

Has the laboratory performed a **satisfactory demonstration of method capability** prior to the acceptance & institution of this test method.

**Note:** Demonstrations of capability are done in an applicable & available **clean quality system matrix sample** in a quality system matrix where **no target analytes or interferences present** at concentrations that impact the results of a specific test method.

**Note:** These following steps are **may not be applicable** for tests with which **spiking is not an option** and for which Quality Control samples are **not readily available**.

**Note:** Actual sample spike results, such as **4 consecutive matrix spikes** (or quality control samples of analytes that do not lend themselves to spiking), within the **last 12 months** may be used to show that a demonstration of capability is not required.

**Note:** A demonstration of capability is **not required** in cases where samples are analyzed with this test method in use by the laboratory **before July 1999** & where there have been **no significant changes** in instrument type, personnel, or test method, in which case the analyst’s documentation of continued proficiency is acceptable (the laboratory must have records on file to show that a demonstration of capability is not required).

**Note:** Continuing demonstration of method performance, per the QC requirements in App. D (e.g., laboratory control samples), is required thereafter.

C.1

Does the laboratory **document** in its Quality Manual **other adequate approaches to Demonstration of Capability** if the procedure below is **not required** by the mandated test method or regulation and if the laboratory **elects not to perform** this procedure.

C.1(a)

Is the **quality control sample** used for this Demonstration of Capability obtained from an **outside source**.

**Note:** If an outside source is not available, the laboratory may prepare this sample with stock standards that are **prepared independently** from those used in instrument calibration.

C.1(b)

Are the analytes diluted in a volume of **clean quality system matrix** sufficient to prepare **4 aliquots** at the **specified concentration** or to a concentration approximately **1-4 times** the **limit of quantitation**.

C.1(c)

Are **at least 4 such aliquots prepared & analyzed** according to the test method.

**Note:** These analyses may occur either concurrently or over a period of days.

C.1(d)

Does the laboratory **calculate the mean recovery** in the appropriate reporting units & the **standard deviation** of the population sample (n-1) in the same units for each parameter of interest using **all of the analysis results obtained**.

**Note:** When it is not possible to assess mean & standard deviation, such as for **presence-absence & logarithmic values**, the laboratory must assess performance against established & documented criteria.

C.1(e)

Are the mean and standard deviation for each parameter **compared** to the corresponding **acceptance criteria for precision & accuracy** in the test method (if applicable) or in laboratory-generated acceptance criteria (if the method or analyte is non-standard).

C.1(f)

Does the laboratory consider the performance unacceptable & **not analyze actual samples** for parameters that **fail the acceptance criteria**.

C.1(f)

When one or more parameters fail at least one of the **acceptance criteria**, does the analyst:

- **Locate & correct** the source of the problem, then **repeat the test** for all parameters of interest, **OR**
- **Repeat the test** for all parameters that failed to meet criteria.

**Note:** Repeated failure from employing the second option above indicates a general problem with the entire measurement system, and the analyst must then perform the first option above.
CHEMISTRY TEST METHOD ASSESSED: ________________________________

C.1 Is an initial evaluation performed for all analytes to be added to an existing accredited test method (for analytes not currently found on the laboratory’s list of accredited analytes)

5.5.2.6(c)(3) Does each Analyst have documentation of continued proficiency by at least one of the following once per year:

- Acceptable performance of a blind sample (single blind to the analyst)
- An initial measurement system evaluation or another demonstration of capability
- Successful performance of a blind performance sample on a similar test method using the same technology (acceptable limits must be determined prior to analysis)
- At least 4 consecutive laboratory control samples with acceptable levels of precision & accuracy (the acceptable limits must be determined prior to analysis)
- Analysis of authentic samples that have been analyzed by another trained analyst with statistically indistinguishable results

5.5.4.2.2(d) Does the laboratory use the NELAC-specified certification statement to document the completion of each Demonstration of Capability (initial & continuing)

C.2 Are copies of these certification statements retained in the personnel records of each employee performing the test method

5.5.4.2.2(d) Does the laboratory retain & make available all associated supporting data necessary to reproduce the analytical results summarized in the appropriate certification statement

5.5.4.2.2(e) Does the laboratory complete a demonstration of capability each time there is a change in instrument type, personnel, or test method

5.5.4.2.2(f) Does the laboratory fully document the achievement of demonstration of capability requirements for each specialized work cell

Note: A work cell is defined as a group of analysts with specifically defined tasks that together perform the test method

5.5.4.2.2(g) Does the laboratory demonstrate & document acceptable performance through acceptable continuing performance checks (e.g. laboratory control samples) each time that membership in a work cell changes

5.5.4.2.2(g) Do the new members of the work cell work with experienced analysts in the specialty area

5.5.4.2.2(g) Does the laboratory repeat a Demonstration of Capability with the new work cell if the first 4 continuing performance checks following the change in personnel produce a failure in any sample batch acceptance criteria

5.5.4.2.2(g) Does the laboratory repeat a Demonstration of Capability if the entire work cell is changed or replaced

5.5.4.2.2(h) Is the performance of the work cell as a group linked to the training records of the individual members of the work cell

5.1.1 Does the laboratory’s procedure for demonstrating its capability to perform the method, the analyst’s capability to perform the method, or the acceptance criteria for precision & accuracy comply with the requirements specified in the mandated test method

Note: See page 14 for such Demonstration of Capability procedural requirements & acceptance criteria
CHEMISTRY TEST METHOD ASSESSED: ________________________________

___ D Does the laboratory have procedures for developing acceptance/rejection criteria for each Chemistry test method (where no regulatory or method criteria exist)

___ D Does the laboratory assess & evaluate all quality control measures on an on-going basis

___ D Does the laboratory use quality control acceptance criteria to determine the validity of the data

___ 5.5.9.2(d) Does the laboratory's Chemistry data indicate that the quality control protocols in the test methods manual are being followed (by all analysts)

___ App. D 5.1.1 Does the laboratory's acceptance criteria for blanks, laboratory control samples, duplicates, & matrix spikes fulfill the requirements in mandated test methods

Note: See page 14 for acceptance criteria

___ 5.1.1 Does the laboratory fulfill additional requirements specified in the mandated test method or regulation

Note: See page 15 for the additional requirements stated in test methods

___ D.1.1.1(a) Does the laboratory process the method blank along with & under the same conditions as the associated samples to include all steps in the analytical procedure

___ D.1.1.1(a) Does the laboratory have procedures in place to determine if a method blank is contaminated

___ D.1.1.1(b) Does the laboratory analyze method blanks at a frequency of at least one per preparation batch or one per 20 environmental samples analyzed together with the same method & personnel using the same lots of reagents

___ D.1.1.1(c) Does the method blank consist of a quality system matrix similar to associated samples & known to be free of the analytes of interest

___ D.1.1.1(d) Does the laboratory critically evaluate each method blank as to the nature of any interferences & the effect on the analyses of each sample within the batch

___ D.1.1.1(d) Is the source of the contamination investigated & measures taken to minimize or eliminate the problem

___ D.1.1.1(d) Are all samples associated with a contaminated blank reprocessed for analysis or reported with appropriate data qualifying codes

Note: Such sample results can be reported with data qualifiers:
- If the analyte concentration in the blank is at or above the reporting limit AND is greater than 1/10 of the amount measured in any sample OR
- If the method blank contamination affects the sample results as per test method requirements or individual project data quality objectives

___ D.1.1.1(d) Does the laboratory document all corrective actions taken with respect to a contaminated blank
CHEMISTRY TEST METHOD ASSESSED: ______________________________

____ D.1.1.2(b) Does the laboratory analyze at least one laboratory control sample (LCS or QC Check Sample) per preparation batch or one per 20 environmental samples analyzed together with the same method & personnel using the same lots of reagents

Note: This Standard does not apply to analytes for which spiking solutions are not available (e.g., Total Suspended Solids, Total Dissolved Solids, Total Volatile Solids, Total Solids, pH, Color, Odor, Temperature, Dissolved Oxygen, or Turbidity)

Note: The matrix spike may be used in place of this control sample as long as the acceptance criteria are as stringent as for the laboratory control sample

Note: The LCS may consist of media containing known & verified concentrations of analytes or as a Certified Reference Material

____ D.1.1.2(c) Does the laboratory include all target analytes in the LCS spike mixture over a 2-year period

____ D.1.1.2(c) Are all analyte concentrations in the LCS within the calibration range of the test method

____ D.1.1.2(c) Are the components spiked into the LCS as specified by the mandated test method or other regulatory requirement or as requested by the client

Note: In the absence of such requirements, the minimum number of analytes to spike are:
- For methods with 1-10 target analytes, spike all analytes
- For methods with 11-20 analytes, spike at least 10 analytes or 80%, whichever is greater
- For methods with more than 20 target analytes, spike at least 16 analytes

Note: The analytes selected for spiking must be representative of all analytes reported & must represent the chemistries and elution patterns of the components to be reported, when some components interfere with accurate assessment (e.g., simultaneously spiking technical Chlordane, Toxaphene, & PCB’s)

____ D.1.1.2(d) Does the laboratory document the calculations for percent recovery of the individual batch LCS

____ D.1.1.2(d) Are the individual analyte percent recoveries compared to the acceptance criteria published in the mandated test method or, where such criteria are not established, to client-specified acceptance criteria or to internal criteria determined at the laboratory

Note: The laboratory must document the method used to establish internal LCS recovery limits

____ D.1.1.2(d) Are all samples associated with an out-of-control LCS reprocessed for analysis or reported with appropriate data qualifying codes

____ D.1.1.2(e) For large number of analytes in the LCS, does the laboratory take corrective actions if acceptance criteria (3 standard deviations) are not achieved:
- for 2 analytes when the LCS contains 11-30 analytes
- for 3 analytes when the LCS contains 31-50 analytes
- for 4 analytes when the LCS contains 51-70 analytes
- for 5 analytes when the LCS contains 71-90 analytes
- for 6 analytes when the LCS contains over 90 analytes

____ D.1.1.2(e) Does the laboratory locate the source of error & take corrective action if the same analyte exceeds LCS control limits repeatedly

____ D.1.1.2(e) Does the laboratory have a written procedure to monitor the application of marginal exceedance allowances to LCS control limits to ensure random behavior

8
CHEMISTRY TEST METHOD ASSESSED: ________________________________

D.1.1.3  Does the laboratory document procedures for determining the effect of the sample matrix on test method performance

Note: These procedures relate to the analysis of quality system matrix specific QC samples & could be data quality indicators for a specific sample using a designated test method; these controls alone are not used to judge laboratory performance

D.1.1.3  Does the laboratory have procedures in place for tracking, managing, & handling matrix-specific QC criteria

Note: These procedures must include spiking appropriate components at appropriate concentrations, calculating recoveries & relative percent difference, and evaluating & reporting results based on performance of the QC samples

D.1.1.3.1(b)  Does the laboratory perform matrix spikes (MS) at a frequency specified by the test method

Note: This matrix spike analysis frequency is specified in pages xx-xx

Note: If the test method is not mandated, the laboratory must determine the frequency of matrix spike analysis as part of a systematic planning process (e.g., data quality objectives)

D.1.1.3.1(c)  Are the components spiked into the MS as specified by the mandated test method or other regulatory requirement or as requested by the client

Note: In the absence of such requirements, the minimum number of analytes to spike are:
- For methods with 1-10 target analytes, spike all analytes
- For methods with 11-20 analytes, spike at least 10 analytes or 80%, whichever is greater
- For methods with more than 20 target analytes, spike at least 16 analytes

Note: The analytes selected for spiking should represent the chemistries & elution patterns of components to be reported (e.g., simultaneously spiking Chlordane, Toxaphene, & PCB’s)

D.1.1.3.1(c)  Does the laboratory include all target analytes in the MS spike mixture over a 2-year period

D.1.1.3.1(d)  Does the laboratory document the calculations for percent recovery & relative percent difference in matrix spikes & matrix spike duplicates

D.1.1.3.1(d)  Are the individual analyte percent recoveries compared to the acceptance criteria published in the mandated test method

D.1.1.3.1(d)  If there is no established criteria, has the laboratory determined internal criteria & documented the method used to establish the limits

D.1.1.3.1(d)  Are all samples associated with matrix spike results outside established criteria documented with corrective actions or reported with appropriate data qualifying codes

COMMENTS:
CHEMISTRY TEST METHOD EVALUATED: ________________________________

___ D.1.1.3.2(b) Does the laboratory perform **matrix duplicates** at a frequency specified by the **required mandated test method**

**Note:** This matrix duplicate analysis frequency is specified in pages xx-xx

___ D.1.1.3.2(c) Are matrix duplicates performed on **replicate aliquots of actual samples**

___ D.1.1.3.2(d) Does the laboratory **document the calculations for relative percent difference** or other statistical treatments

___ D.1.1.3.2(d) Are the individual analyte duplicate precisions **compared to the acceptance criteria** published in the mandated test method

___ D.1.1.3.2(d) If there is no established criteria, has the laboratory **determined internal criteria & documented the method** used to establish the limits

___ D.1.1.3.2(d) Are **all samples** associated with duplicate precisions **outside established criteria** documented with corrective actions or **reported** with appropriate **data qualifying codes**

___ D.1.1.3.3(b) Does the laboratory add **surrogate compounds** to all **samples, standards, & blanks** for all **appropriate test methods**

**Note:** This Standard does not apply if the sample matrix precludes the use of surrogates or when a surrogate is not commercially available

___ D.1.1.3.3(c) Do the surrogates **represent the various chemistries** of the method’s target analytes & deliberately chosen for **being unlikely to occur** as an environmental contaminant

___ D.1.1.3.3(d) Are the surrogate recoveries **compared to the acceptance criteria** in the mandated test method

___ D.1.1.3.3(d) Does the laboratory evaluate surrogate recoveries outside acceptance limits for **the effect indicated** for the individual sample results

___ D.1.5(a) Has the laboratory **evaluated selectivity** by following the checks established within the method

**Note:** These evaluations may include mass spectral tuning, second-column confirmation, chromatography retention time windows, ICP inter-element interference checks, sample blanks, spectrochemical absorption or fluorescence profiles, co-precipitation evaluations, & electrode response factors.

___ D.1.5(b) Does the laboratory perform confirmations to **verify compound identification** when positive results are detected on a **sample from a location** that has **not been previously tested** by the laboratory

**Note:** These confirmations are performed on pesticides, herbicides, acid extractables, or other organic tests, or when recommended by the analytical test method

**Note:** Confirmation is not required when the analysis involves the use of a mass spectrometer

**Note:** Confirmation is required unless stipulated in writing by the client

___ D.1.5(b) Does the laboratory **document all confirmations** of compound identity

___ D.1.5(c) If a mass spectrometer is used, has the laboratory documented **acceptance criteria for mass spectral tuning**
CHEMISTRY TEST METHOD EVALUATED: ________________________________

___ D.1.2 Does the laboratory document all procedures & retain all supporting data in determining & verifying limits of detection & limits of quantitation

___ D.1.2.1 Does this test method provide limits of detection (LOD’s) that are appropriate & relevant for the intended use of the data

___ D.1.2.1 Has the laboratory determined the limit(s) of detection by the protocol in the mandated test method or applicable regulation

Note: If the protocol for determining LOD’s is not specified, the laboratory must still determine the LOD’s but according to a procedure that reflects instrument limitations & intended application of the test method

Note: In the absence of regulatory or client requirements, an LOD is not required when test results are not reported outside of the calibration range

___ D.1.2.1(a) Has the laboratory initially determined the detection limits for the compounds of interest in this test method in a quality system matrix in which there are no target analytes or interferences at a concentration that would impact the results

Note: If this is not possible, the laboratory must determine these detection limits in the quality system matrix of interest

___ D.1.2.1(b) Does the laboratory determine LOD’s each time there is a change in the test method that affects how the test is performed or when a change in instrumentation occurs that affects the sensitivity of the analysis

___ D.1.2.1(c) Does the laboratory have established procedures to relate LOD’s with Limits of Quantitation (LOQ’s)

___ D.1.2.1(d) Has the laboratory verified the LOD annually for each quality system matrix, test method, & analyte

Note: All sample processing steps of the analytical method must be included in the determination of the LOD

Note: Validity of the LOD is confirmed by qualitative identification of the analyte(s) in a quality control sample in each quality system matrix containing the analyte at no more than 2-3x the LOD for single-analyte tests and 1-4x the LOD for multiple analyte tests

Note: LOD verification must be performed on every instrument that is to be used for analysis of samples & reporting of data

Note: A LOD study is not required for any component for which spiking solutions or quality control samples are not available (e.g., Temperature), or when test results are not to be reported to the LOD (versus the Limit of Quantitation or working range of instrument calibration according to Appendices D.1.2, D.4.5, D.5.4, and D.6.6 to NELAC Chapter 5).
CHEMISTRY TEST METHOD EVALUATED: ________________________________

___ D.1.2.2(a)  Are all established LOQ’s above the LOD’s for each analyte

___ D.1.2.2(b)  Has the laboratory verified the LOQ annually for each quality system matrix, test method, & analyte

Note: The LOQ study is not required for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate (e.g., pH).

Note: The validity of the LOQ is confirmed by successful analysis of a quality control sample, containing the analytes of concern in each quality system matrix at 1-2 times the claimed LOQ

Note: A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria or client data quality objectives for accuracy.

Note: This single analysis is not required if the bias & precision of the measurement system are evaluated at the LOQ

Note: The LOQ verification is not required if the LOD is re-evaluated or verified

___ 5.1.1  Do the laboratory’s limits of detection fulfill the requirements of mandated test methods or regulations

Note: US EPA’s Safe Drinking Water Act (SDWA) & Clean Water Act (CWA) regulations require determination of Method Detection Limits according to the procedures & criteria in 40 CFR Part 136, Appendix B

Note: See page 15 for SDWA Maximum Contaminant Levels & RCRA Toxicity Characteristics, which the LOD, LOQ, or the lowest-concentration calibration standard must be reliably & consistently below

Note: Other regulations (including state regulations) & permits may contain additional requirements for Reporting Limits, Minimum Levels, Lower Limits of Detection, & other criteria

COMMENTS: List analytes for which the above requirements for measurement sensitivity have not been fulfilled
REQUIRED REAGANTS & STANDARDS

EPA 524.2, 624, 1624, 1666, 8260; SM6210B (<=19th ed.) (packed-column);
SM6200B (20th ed.), SM6210D (<=19th ed.) (both capillary-column GC-MS)

- Internal Standards (isotopically labeled for EPA 1624 & 1666)
- Surrogate solutions
- Bromofluorobenzene (BFB) Tuning Solution for MS (daily)
- Tenax/Silica Gel/Charcoal sorbent trap, or synthetic polymer, conditioned per manufacturer's instructions
  (includes EPA 5030 prior to 8260)

*Note:* Direct sample injection or other sample introduction techniques available for EPA 1666

EPA 3585 with EPA 8021, 8031, 8033, 8260
- Waste Dilution Solvent: n-Hexadecane

EPA 5035 with EPA 8021, 8260
- Methanol (or other water-miscible solvent)
- Sodium Bisulfate, as solid preservative

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

- 14-Day Holding Time, glass vial w/ Teflon-lined septum, 4 C, 0.008% Na2S2O3
  Purgeable Halocarbons (NO HEADSPACE)

- 14-Day Holding Time, glass vial with Teflon-lined septum, 4 C, 0.008% Sodium Thiosulfate, HCl to pH<2 (NO HEADSPACE)
  Purgeable Aromatic Hydrocarbons (7-Day Holding Time if pH adjustment not made)

- 14-Day Holding Time, glass vial with Teflon-lined septum, 4 C, 0.008% Sodium Thiosulfate, pH 4-5
  Acrolein & Acrylonitrile (3-Day Holding Time for Acrolein & 7-Day Holding Time for Acrylonitrile if pH adjustment not made)

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank
- SM6020B, 1a & 1b, applies to all SM Organics mtds., int. std. response factor (if used) < 20% RSD
- EPA 524.2, 7.8.1 & 10.2, response factor (if used) < 20% RSD
- EPA 624, 7.3, response factor (if used) < 35% RSD

5 standards + blank
- EPA 1624, 7.4-7.5, relative response factor (if used) < 20% RSD relative to the isotope-labeled analog, < 35% RSD relative to sample matrix internal standard
- EPA 1666, 10.4, labeled cmpd. CF or native cmpd. RF (if used) < 20% RSD
- EPA 8260, 7.3, response factor (if used) < 15% RSD for each analyte, response factor < 30% RSD for Calibration Check Compounds
  Requires client notification of analytes quantitated from CF or RF when mtd. criteria not met (and mean RSD < 20%)
CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 70-130%

EPA 524.2, 10.3, absolute peak areas for Internal Standards & Surrogates must also be checked
PLUS (40 CFR 141.131(b)(2)(iv)) 50-150% recovery for MRL verification std. at 1.0 ug/L (THM’s)

Recovery 80-120%

EPA 8260, 7.4, for Calibration Check Compounds
SM6020B, 1b (applies to SM Organics methods)
40 CFR 141.40, App. A, 3, mid-range std. plus 60-140% recovery for std. at or below Minimum Reporting Level (applies to SDWA Unregulated Contaminants)

Recovery within the Test Method QC Acceptance Criteria

EPA 624, 7.4
EPA 1624, 11.5
EPA 1666, 15.5

PRECISION & ACCURACY ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

INITIAL DEMONSTRATION OF CAPABILITY

Mean Accuracy 80-120% for each analyte; Precision RSD<20%
EPA 524.2, 9.3, concentrations 2.0-5.0 ug/L for each analyte, MDL also required

Average Recovery & Std. Dev. of Recovery compared to Acceptance Criteria in Table of Test Method
EPA 624, 1624, 8.2, concentration 20 ug/L or less for each target analyte
EPA 1666, 9.2
EPA 8000, 8.6, applicable to all EPA 8000-series methods

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample Recoveries within 70-130%
EPA 524.2, 9.6

QC Check Sample or Matrix Spike Recoveries within Test Method QC Acceptance Criteria for each Analyte
EPA 624, 8.4
EPA 8000, 8.8, applicable to EPA 8260
EPA 1666, 9.3

External QC Check Sample Analyzed Quarterly
EPA 524.2, 9.9

Internal Standards Responses
EPA 524.2, 10.3, 70-130% from last Calib. Verification & 50-150% from last Initial Calibration
EPA 1624, 8.3, serves as Matrix Spike since labeled stds. added to each sample, acceptance criteria in Table
EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

Each Regulated VOC (MDL requirement)  0.5  ug/L
Total Trihalomethanes  80  ug/L

RCRA TOXICITY CHARACTERISTICS

<table>
<thead>
<tr>
<th>Substance</th>
<th>Limit (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzene</td>
<td>0.5</td>
</tr>
<tr>
<td>Carbon Tetrachloride</td>
<td>0.5</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>100.0</td>
</tr>
<tr>
<td>Chloroform</td>
<td>6.0</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>7.5</td>
</tr>
<tr>
<td>1,2-Dichloroethane</td>
<td>0.5</td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>0.7</td>
</tr>
<tr>
<td>Methyl Ethyl Ketone</td>
<td>200.0</td>
</tr>
<tr>
<td>Tetrachloroethene</td>
<td>0.7</td>
</tr>
<tr>
<td>Trichloroethene</td>
<td>0.5</td>
</tr>
<tr>
<td>Vinyl Chloride</td>
<td>0.2</td>
</tr>
</tbody>
</table>

ADDITIONAL REQUIREMENTS

All samples spiked with Isotopically Labeled Compounds
- EPA 1624, 8.3
- EPA 1666, 9.3

Matrix Spikes analyzed every 10 samples
- SM6020B, 3c, or Monthly (whichever is more frequent) (applies to all SM Organics methods)

Matrix Spike & Matrix Spike Duplicate each batch of 20 samples or fewer
- 40 CFR 141.40, App. A, 6, must also alternate between mid- & low-level concentrations for spikes
  (applies to SDWA Unregulated Contaminants)
- EPA 8000, 8.5, applies to all 8000-series Organics mtds., may use sample dup. in place of MSD

Matrix Spike every 20 samples per site
- EPA 624, 8.3

GC Retention Time Windows established for each analyte
- EPA 1624, 11.3
- EPA 8000, 7.6

Chromatographic Resolution Checks
- EPA 1624, 11.4, Toluene & Toluene-d8

Response Factors > 0.3 for System Performance Check Compounds
- EPA 8260, 7.4, during Initial Instrument Calibrations & Calibration Verifications

Initial Instrument Calibration Concentration Range Must Encompass Minimum Reporting Level of 1.0 ug/L
- 40 CFR Part 141, applies to Trihalomethanes

GC/MS Tune Criteria with BFB (EPA 524.2, 624, 8260)

<table>
<thead>
<tr>
<th>mass ion (m/z)</th>
<th>abundance criteria</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>15-40% of m/z 95</td>
</tr>
<tr>
<td>75</td>
<td>30-60% of m/z 95 (30-80% for EPA 524.2)</td>
</tr>
<tr>
<td>95</td>
<td>100% (base peak)</td>
</tr>
<tr>
<td>96</td>
<td>5-9% of m/z 95</td>
</tr>
<tr>
<td>173</td>
<td>&lt;2% of m/z 174</td>
</tr>
<tr>
<td>174</td>
<td>&gt;50% of m/z 95</td>
</tr>
<tr>
<td>175</td>
<td>5-9% of m/z 174</td>
</tr>
<tr>
<td>176</td>
<td>95-101% of m/z 174</td>
</tr>
<tr>
<td>177</td>
<td>5-9% of m/z 176</td>
</tr>
</tbody>
</table>