PESTICIDES GAS CHROMATOGRAPHY (GC)

DETECTORS: FLAME IONIZATION (FID), ELECTRON CAPTURE (ECD), PHOTOIONIZATION (PID), ELECTROLYTIC CONDUCTIVITY (ELCD), FLAME PHOTOMETRIC (FPD), NITROGEN-PHOSPHORUS (NPD), FOURIER TRANSFORM INFRARED (FTIR)

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: _____

____ **5.5.5.2.2** Do the laboratory's initial & continuing instrument calibration verifications meet the requirements in **mandated test methods & regulations** (see pages 17-18 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

____ **5.5.5.2.2.1(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

____ **5.5.5.2.2.1(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

____ **5.5.5.2.2.1(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

____ **5.5.5.2.2.1(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

____ **5.5.5.2.2.1(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

____ **5.5.5.2.2.1(f)** For purposes of establishing the **working calibration range**, is the lowest calibration standard concentration the **lower limit of quantitation**

____ **5.5.5.2.2.1(f)** Is all data reported **below the lower limit of quantitation** reported using **defined qualifiers** or flags or **explained in the case narrative**

____ **5.5.5.2.2.1(g)** Is the highest calibration standard the **highest concentration** for which **quantitative data are to be reported**

____ **5.5.5.2.2.1(g)** Is all data reported **above the highest calibration standard** reported using **defined qualifiers** or flags or **explained in the case narrative**

____ **5.5.5.2.2.1(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**

____ **5.5.5.2.2.1(h)** Is the **lowest calibration standard above the limit of detection** for each analyte

CHEMISTRY TEST METHOD EVALUATED: _____

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

CHEMISTRY TEST METHOD EVALUATED: _____

____ **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 useable 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

COMMENTS:

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 meet this Standard

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(e)** 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)** **C.2** 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)** **C.1** 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)** **C.1** 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 pages 18-19 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)** **App. D**	Does the laboratory's **Chemistry data** indicate that the **quality control protocols** in the test methods manual **are being followed** (by all analysts)
____	**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 pages 19-20 for acceptance criteria
____	**5.1.1**	Does the laboratory fulfill **additional requirements** specified in the **mandated test method** or **regulation** **Note:** See pages 21-22 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**

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 e**valuated at the LOQ**

Note: The LOQ verification is not required 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 pages 20-21 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

PESTICIDES GAS CHROMATOGRAPHY (GC)

DETECTORS: FLAME IONIZATION (FID), ELECTRON CAPTURE (ECD), PHOTOIONIZATION (PID), ELECTROLYTIC CONDUCTIVITY (ELCD), FLAME PHOTOMETRIC (FPD), NITROGEN-PHOSPHORUS (NPD), FOURIER TRANSFORM INFRARED (FTIR)

REQUIRED REAGANTS & STANDARDS

EPA 608, 608.1, 608.2, 627, 1656 (GC-ECD); 619, 622 (GC-NPD or FPD); 645 (GC-NPD); 1657 (GC-FPD); USGS O-3106-93 (GC-NPD); SM6630C (GC-ECD); EPA 3510, 3520, 3540, 3541 with 8081, 8082 (GC-ECD), and with 8141 (GC-NPD or FPD);

Methylene Chloride extraction solvent
Sodium Sulfate drying reagent
n-Hexane exchange solvent
Surrogate Standards (**EPA 1656, 1657, 8000's**)
Soxhlet extraction thimble (EPA 3540, 3541)
Note: Use Chloroform/Acetone to extract polar analytes in **EPA 1657**
Note: GC-ELCD may also be used for **EPA 1656**
Note: GC-MS may also be used for **EPA 608.1, 619, 622, 627**

EPA 508.1 (GC-ECD); 3M0222 (GC-ECD); EPA 3535 with EPA 8081, 8082 (GC-ECD)

Reverse-phase C-18 solid-phase disks or cartridges
Ethyl Acetate, Methylene Chloride, Methanol solid-phase conditioning reagents
Ethyl Acetate, then Methylene Chloride, as eluting solvents (**EPA 508.1**)
Methylene Chloride eluting solvent (**EPA 8081, 8082**)
Sodium Sulfate drying reagent
Endrin & DDT for GC degradation check (**EPA 508.1**)
Surrogate standards (required for **EPA 8000's**)
Note: Ethyl Acetate not required for **3M0222**

EPA 507 (GC-NPD), 508 (GC-ECD)

Methylene Chloride extraction solvent
Sodium Sulfate drying reagent
MTBE exchange solvent
Surrogate solutions

EPA 508A (GC-ECD)

Methylene Chloride extraction solvent
Sodium Sulfate & Sodium Bicarbonate drying agents
Antimony Pentachloride perchlorination reagent & Iron catalyst (converts PCB's to decachlorobiphenyl)
1:1 Hydrochloric Acid quenching reagent
n-Hexane final extraction solvent

EPA 515.1, 615, 1658, 8151 (GC-ECD); ASTM D5317-93 (GC-ECD)

Ethyl Ether extraction solvent (Methylene Chloride for **EPA 1658**, but continuous liq.-liq extraction required)
Potassium Hydroxide as phenoxyacid ester hydrolysis reagent
Sulfuric Acid to adjust aqueous-phase pH
Acidified Sodium Sulfate dehydrating agent
n-Hexane exchange solvent (MTBE exchange solvent for **EPA 515.1**)
Diazald to generate diazomethane derivatizing agent (if diazomethane solution made, usable within 48 hr)
Silicic Acid to quench excess diazomethane from Diazald or diazomethane solution
Trimethylsilyldiazomethane (TMSD) as derivatizing agent (available in **EPA 515.1** except for Dalapon)
2 M Acetic Acid in Methanol to quench excess TMSD
Pentafluorobenzyl Bromide as derivatizing agent (available in **EPA 8151**)
Surrogate Standards

EPA 515.2 (GC-ECD)

Polystyrenedivinylbenzene solid-phase extraction disks or cartridges
Sodium Sulfate to salt the aqueous phase & as drying agent
Sodium Hydroxide as phenoxyacid ester hydrolysis reagent
Sulfuric Acid to acidify aqueous phase
Methylene Chloride to extract out impurities
10% MTBE in Methanol, Methanol to condition solid phase (10% MTBE/Methanol is also eluting solvent)
MTBE eluting & exchange solvent
Diazald (to generate diazomethane) or TMSD as derivatizing agent
2 M Acetic Acid in Methanol as quenching agent if TMSD is used
Surrogate Standards

EPA 548.1 (GC-FID or MS); 552.1 (GC-ECD)

Anion Exchange solid-phase disks or cartridges
Methanol, water, 10% Sulfuric Acid/Methanol, water, 1 N NaOH, water to condition the solid phase
10% Sulfuric Acid/Methanol eluting solvent, derivatizes Endothall to its dimethyl ester
Methylene Chloride (**EPA 548.1**) or MTBE (**EPA 552.1**) extraction solvent
Sodium Sulfate drying agent
Surrogate Standard (required for **EPA 552.1**)
Internal Standards (required for both methods)
Acid or Base to adjust sample pH to 4.5-5.5 (**EPA 552.1**)

EPA 551.1 (GC-ECD)

MTBE or n-Pentane extraction solvent
Sodium Sulfate to salt aqueous phase when Disinfection By-Products are analyzed (NaCl not allowed)
Ammonium Chloride buffer to pH 5.2, to sequester free chlorine as a chloramine
Surrogate Standard

EPA 552.2, 552.3, 515.3, & 515.4; SM6233B (<=18th ed.), SM6251B (>=19th ed.), & SM6640B (>=19th ed.) (GC-ECD)

Sodium Hydroxide to adjust sample pH>12 & hydrolyze esters (**EPA 515.3, 515.4**)
10% MTBE/Hexane solvent wash (**EPA 515.4**), to separate Dacthal from its Acid Metabolites
Sulfuric Acid to acidify sample to pH<0.5
MTBE extraction solvent (TAME also allowed for **EPA 515.4**)
Copper Sulfate & Sodium Sulfate to salt aqueous phase
10% Sulfuric Acid/Methanol derivatizing agent (**EPA 552.2, 552.3**) (sodium bicarbonate to quench reaction)
Diazald to generate diazomethane derivatizing agent (**EPA 515.3 & 515.4; SM6233B, SM6640B (>=19th ed.), & SM6251B**) (silica gel to quench reaction)
Tetrabutylammonium Hydroxide & Methyl Iodide derivatizing agents (**EPA 515.3**) (Florisil cleanup sorbent)
1-Methyl-3-nitro-1-nitrosoguanidine (MNNG) derivatizing agent (**SM6233B, SM6251B, & SM6440B (>=19th ed.)**)
Surrogate Standards

EPA 617 (GC-ECD); 614, 614.1 (GC-NPD or FPD); SM6630B (GC-ECD); D3086-90 (GC-ECD or ELCD)

15% Methylene Chloride/n-Hexane extraction solvent
Sodium Sulfate drying reagent
n-Hexane exchange solvent

EPA 622.1, 633.1, 634 (GC-NPD)

Methylene Chloride extraction solvent
Sodium Sulfate drying reagent

EPA 630.1 (GC-ELCD)

Acid to adjust sample pH < 2 (hydrolysis to release CS2)
n-Hexane extraction solvent

EPA 633 (GC-NPD or MS)

Methylene Chloride extraction solvent
Sodium Sulfate drying agent
Acetone exchange solvent

EPA 1659 (GC-NPD)

Ethyl Acetate extraction solvent

EPA 3545 with EPA 8081, 8082, 8141

Pressurized Extraction Fluids: 1:1 Methylene Chloride/Acetone, 1:1 Hexane/Acetone, Hexane, or CH2Cl2

Note: Exchange solvents are based on clean-up method & determinative methods employed

EPA 3550 with EPA 8081, 8082, 8141

Ultrasonic Extraction Fluids: 1:1 Methylene Chloride/Acetone, 1:1 Hexane/Acetone, or Hexane

Note: Exchange solvents are based on clean-up method & determinative methods employed

EPA 3580 with EPA 8081, 8082, 8141, 8151

Waste Dilution Solvents: Methylene Chloride or Hexane

O-3104-83 (USGS Bk.5, Ch.A3, p.27) (GC-FPD & GC-ECD)

Hexane extraction solvent
Sodium Sulfate dehydrating agent
Alumina Clean-up Sorbent for organochlorine pesticides

SM6640B (<=18th ed.), USGS O-3105-83 (Bk.5, Ch.A3, p.40) (GC-ECD)

Ethyl Ether extraction solvent (MTBE also allowed in USGS mtd.)
Potassium Hydroxide as phenoxyacid ester hydrolysis reagent
Sulfuric Acid to adjust aqueous-phase pH
Acidified Sodium Sulfate dehydrating agent
Toluene exchange solvent
Boron Trifluoride in Methanol derivatizing agent
Magnesia / Silica Gel sorbent to quench excess derivatizing agent

EPA 3620 prior to EPA 8081, 8082, 8141, 8151

Florisil Clean-up Sorbent, activated by heating at 130 C overnight or deactivated by soaking in H2O for 2 hr
Hexane or Petroleum Ether conditioning solvent
20% Ethyl Ether in Hexane, to elute Phthalate Esters from deactivated Florisil
15% Ethyl Ether in Pentane, to elute Diphenylamine from activated Florisil (separate from Nitrosamines);
then 5% Acetone in Ethyl Ether, to elute Nitrosamines from activated Florisil; AND/OR
10% Acetone in Methylene Chloride, to elute Nitroaromatics & Isophorone from activated Florisil
6% Ethyl Ether in Hexane, to elute most Organochlorine Pesticides & PCB's from activated Florisil;
15% Ethyl Ether in Hexane, to elute Dieldrin, Endosulfan I, & Endrin from activated Florisil; then
50% Ethyl Ether in Hexane, to elute Endosulfan II, Endosulfan SO4, Endrin Aldehyde from Florisil
10% Acetone in Hexane, to elute all Organochlorine Pesticides & PCB's from Florisil cartridges
Hexane, to elute PCB's, Aldrin, DDE, & Heptachlor from Florisil cartridges;
26% Methylene Chloride in Hexane, to elute most other Organochlorine Pesticides; then
10% Acetone in Hexane, to elute Endosulfan II, Endrin Aldehyde, DDT, & remaining Methoxychlor
Petroleum Ether, to elute Chlorinated Aromatics from activated Florisil; then
6% Ethyl Ether in Petroleum Ether, to elute Haloethers from activated Florisil
50% Methylene Chloride in Hexane, to elute 2,4,6-Trichloroaniline from activated Florisil;
5% Isopropanol in Hexane, to elute most Aniline Derivatives; then
5% Methanol in Hexane, to elute the remaining Aniline & Dinitroanilines
10% Ethyl Ether in Hexane, to remove impurities from activated Florisil;
30% Ethyl Ether in Hexane, to elute Organophosphorus Pesticides from activated Florisil; then
40% Ethyl Ether in Hexane, to elute Tris(2,3-dibromopropyl) Phosphate
20% Methylene Chloride in Hexane, to elute Methyl Pentachlorophenate Ester from activated Florisil;
50%/0.35%/49.65% Methylene Chloride/Acetonitrile/Hexane, to elute most derivatized Herbicides;
then Ethyl Ether, to elute Picloram
Pesticide Check Solution (10 organochlorine pesticides), Herbicide Check Solution (3 chlorophenoxy
methyl esters), & 2,4,5-Trichlorophenol – used to test **each batch** of activated Florisil

EPA 3630 prior to EPA 8082

Silica Gel Clean-up Sorbent, activated at 130 C for 16 hours, conditioned with Pentane solvent
40% Methylene Chloride in Pentane, to elute Polynuclear Aromatic Hydrocarbons from silica gel
20% Benzene in Hexane or 100% Hexane, to elute Dibenzo-p-dioxins & Dibenzofurans from silica gel
15% Toluene in Hexane, to elute derivatized Pentachlorophenol from silica gel;
    40% then 70% Toluene in Hexane, to elute most Derivatized Phenols from silica gel; then
    15% Isopropanol in Toluene, to elute the derivatized Nitrophenols
25% Toluene in Hexane, to elute Derivatized Phenols from silica gel cartridge
Hexane, to elute PCB's, Heptachlor, Aldrin, & DDE from silica gel or silica gel cartridge; then
    Methylene Chloride, to elute remaining Organochlorine Pesticides from silica gel; or
    50% Ethyl Ether in Hexane, to elute remaining Organochlorine Pesticides from silica gel cartridge

EPA 3640 prior to EPA 8081

Gel Permeation Chrmoatography system with GPC Bio-Beads, UV Detector
GPC Calibration Solution (Corn Oil, Bis(2-ethylhexyl) Phthalate, Methoxychlor, Perylene, Sulfur)
    (store at 4 C, replace every 6 months)
Methylene Chloride eluting solvent
Semivolatile Organics collected within the Phthalate, Methoxychlor, & Perylene elution times
Organochlorine Pesticides & PCB's collected within the Methoxychlor & Perylene elution times

EPA 608; EPA 3660 prior to EPA 8081

Mercury, Activated Copper powder, or Tetrabutylammonium Sulfite (Tetrabutylammonium Bisulfate &
    Sodium Sulfite, stable for 1 month), to remove Sulfur from extracts

EPA 3665 prior to EPA 8082

Sulfuric Acid and/or KMnO4, to remove any color or emulsions from hexane phase for PCB's

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

7 Days to Extract Sample, 40 Days to Analyze Extract; glass container with Teflon-lined cap; 4 C

Organochlorine Pesticides (RCRA), PCB's

7 Days to Extract Sample, 40 Days to Analyze Extract; glass containers with Teflon-lined cap; 4 C; pH 5-9

Pesticides (CWA), Organophosphorus Pesticides (RCRA)

14 Days to Analyze Sample, 4 C, Sodium Sulfite & HCl to pH<2

SDWA Chlorinated Phenoxy Acids (Herbicides)

14 Days to Extract Sample, 14 Days to Analyze Extract; 4 C

SDWA Chlorinated Solvents & Disinfection By-Products

14 Days to Extract or Analyze Sample, 4 C

Other SDWA Pesticides & PCB's

14 Days to Extract Sample; 7-28 Days to Analyze Extract (depending on storage Temp.); Amber Glass container w/ Teflon-lined lid; 4 C; Ammonium Chloride

SDWA Haloacetic Acids

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 505, 10.2, calibration factor (if used) < 20% RSD
EPA 507, 508, 10.2 & 10.3, calibration factor or response factor (if used) < 20% RSD
EPA 515.1, 9.2, calibration factor or response factor (if used) < 20% RSD
EPA 515.2, 10.2, response factor (if used) < 30% RSD
EPA 552.1, 10.2, each standard processed & analyzed in triplicate, internal standard technique required
EPA 608, 608.1, 614, 617, 619, 622, 622.1, 627, 629, 631, 632, 633, 633.1, 634, 637, 7.2 & 7.3, calibration factors or response factors (if used) < 10% RSD
EPA 608.2, 614.1, 645, 8.1, calibration factors (if used) < 10% RSD
EPA 615, 7.2, calibration factors (if used) < 10% RSD
EPA 630.1, 7.3, calibration factors (if used) < 10% RSD
EPA 632.1, 8.2, calibration factors (if used) < 10% RSD
EPA 1656, 1657, 1658, 1659, 7.3, calibration factor (if used) < 20% RSD
EPA 1661, 7.3, calibration factor (if used) < 15% RSD
D5317-93, 11.2 & 11.3, calibration factor or response factor (if used) < 20% RSD
3M0222, 10.2 & 10.3, calibration factor or response factor (if used) < 10% RSD
O-3106-93, 6.15, performed **daily**

4 standards + blank

EPA 548.1, 10.1, each standard analyzed in triplicate, response factor < 30% RSD

5 standards + blank

EPA 8000, 7.4-7.5, calibration factor or response factor (if used) < 20% RSD, correlation coefficient >0.990 for non-linear calibration
Applies to **EPA 8081, 8082, 8141, 8151** (GC Pesticides)
Requires **client notification of analytes** quantitated from CF or RF when mtd. criteria not met (and mean RSD < 20%)
EPA 508A, 9.1, each standard injected in duplicate, response factors < 18% RSD, mid-point standard injected 7 times with response factors < 6% RSD
EPA 515.3, 10.1, relative response factors < 30% RSD, or correlation coefficient > 0.95 (1^{st} or 2^{nd} order)
EPA 515.4, 10.2, relative response factors (if used) < 30% RSD, each std. must be 70-130% of true value, Lowest std. may be 50-150% of true value
EPA 551.1, 10.1-10.3, calibration factor or response factor (if used) < 10% RSD
EPA 552.2, 10.1, relative response factor (if used) < 20% RSD
EPA 552.3, 10.2.2

6 standards + blank

EPA 508.1, 7.13 & 10.5, response factor (if used) < 30% RSD

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 70-130%

EPA 505, 10.2.3
EPA 508.1, 10.7, every 12-hour work shift
EPA 515.2, 10.2.4
EPA 548.1, 10.4, absolute peak area for Internal Standard within 30% of area from most recent calibration verification & within 50% of area from most recent initial instrument calibration
EPA 515.3 & 552.2, 10.2.1, PLUS (**40 CFR 141.131(b)(2)(iv)**) 50-150% recovery for MRL verification std. at 1.0 ug/L (HAA's except 2.0 ug/L Bromoacetic Acid)
EPA 515.4, 552.3, 10.3, for mid- & high-level CCV's, 50-150% recovery for low-level CCV's; Every 10 samples & end of batch

Recovery 80-120%

EPA 507, 508, 515.1, 10.2.4
EPA 508A, 9.2, calibration verification performed at end of sample batch despite int. std. technique used
EPA 1656, 1657, 1658, 7.4, for resolved components on the GC
SM6020B, 1b (applies to SM Organics methods)
D5317-93, 11.2 & 11.3
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 75-125%

EPA 551.1, 10.4, plus recoveries 80-120% for 90% of the analytes, also every **10 samples & end of run**

Recovery 85-115%

EPA 552.1, 10.2.5, PLUS (**40 CFR 141.131(b)(2)(iv)**) 50-150% recovery for MRL verification std. at 1.0 ug/L (HAA's except 2.0 ug/L Bromoacetic Acid)
EPA 608, 7.4
EPA 8000, 7.7
3M0222, 10.4

Recovery 90-110%

EPA 608.1, 614, 617, 619, 622, 622.1, 627, 630, 630.1, 633, 633.1, 634, 637, 7.2 & 7.3
EPA 608.2, 614.1, 645, 8.1
EPA 615, 7.2
EPA 630, 7.5
EPA 630.1, 7.3
EPA 632.1, 8.2

Recovery within the Test Method QC Acceptance Criteria

EPA 1656, 13.5

PRECISION & ACCURACY ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS (INITIAL DEMONSTRATION OF CAPABILITY)

Method Detection Limit required for each analyte

EPA 508.1, 9.3

Mean Accuracy 60-140% for each analyte

EPA 515.2, 9.3, Precision < 30% RSD, MDL also required

Mean Accuracy 70-130% for each analyte

EPA 515.1, 10.3
EPA 552.1, 9.3, Precision RSD < 30% from mean
D5317-93, 19.3

Mean Accuracy 70-130%; Precision RSD < 20%

EPA 505, 507, 508, 9.3, MDL study also required
EPA 552.3, 9.2, MDL study also required

Mean Accuracy 80-120% for each analyte

EPA 515.3, 9.3

Mean Accuracy 80-120%; Precision RSD<30%

EPA 548.1, 9.3.2

Mean Accuracy 80-120% for each analyte; Precision RSD<20%

EPA 515.4, 9.2, MDL determination also required
EPA 552.2, 9.3, MDL determination also required

Mean Accuracy 80-120% for each analyte; Precision RSD<15%

EPA 551.1, 9.4, **7 replicates** required, MDL determination required

Mean Accuracy 80-120%; Precision RSD<10%

EPA 508A, 10.4 & 10.5, PCB Extract Matrix Effect evaluation also required with recovery within 80-120%

Average Recovery & Standard Deviation of Recovery compared to Acceptance Criteria in Table of Test Method

EPA 608, 608.1, 614, 615, 617, 619, 622, 622.1, 627, 630, 630.1, 633, 633.1, 634, 637, 8.2
3M0222, 9.2
EPA 1656, 1657, 1658, 1659, 1661, 8.2
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 508A, 10.6
EPA 508.1, 9.6
EPA 552.1, 9.6

QC Check Sample Recoveries within 80-120%

EPA 548.1, 9.6.2

QC Check Sample or Matrix Spike Recoveries within the Test Method QC Acceptance Criteria for each Analyte

EPA 608, 608.1, 608.2, 1656, 1657, 1658, 8.4
EPA 1659, 1661, 8.3
EPA 8000, 8.8, applicable to **EPA 8081, 8082, 8141, 8151**
3M0222, 9.3 & 9.4

External QC Check Sample Analyzed Quarterly

EPA 552.2, 9.10
EPA 507, 508, 515.2, 9.7
EPA 508A, 10.8
EPA 552.1, 9.8

External QC Check Sample Recoveries within 70-130%

EPA 508.1, 9.9, analyzed quarterly
EPA 552.3, 9.10, analyzed quarterly

External QC Check Sample Recoveries within 80-120%

EPA 508A, 10.8, analyzed quarterly

Matrix Spike Recoveries 50-150%

EPA 515.4, 9.9, analyzed every 20 samples or batch
EPA 552.3, 9.8, analyzed every 20 samples or batch

Matrix Spike Recoveries 65-135%

EPA 507, 9.8, analyzed every 20 samples
EPA 505, 9.6 & **508**, 9.8, analyzed every 10 samples or batch
EPA 508.1, 9.7, analyzed every **sample type** but no frequency specified

Matrix Spike (SMS or LFM) Recoveries within 70-130%

EPA 515.3, 9.7, analyzed **every 10 samples** (Herbicides)
EPA 552.1 & 552.2, 9.7 (Haloacetic Acids)

Matrix Spike Recoveries within 75-125%

EPA 551.1, 9.6, plus recoveries within 80-120% for 90% of the target analytes

Matrix Spike Recoveries within 80-120%
EPA 548.1, 9.7.1 (Endothall)

Surrogate Recoveries within 40-120%
EPA 1656, 1657, 1658, 8.3

Surrogate Recoveries within 60-140%
EPA 515.2, 9.5

Surrogate Recoveries within 70-130%
EPA 507, 508, 9.5
EPA 515.1, 10.5
EPA 515.3, 515.4, 552.2, 9.8
EPA 552.1, 9.4
EPA 552.3, 9.7
D5317-93, 19.5

Surrogate Recoveries 80-120%
EPA 551.1, 9.8

Analyte Concentrations in Blank
EPA 508A, 10.1, <0.025 ug/L for Decachlorobiphenyl

Internal Standards Responses
EPA 507, 508, 515.2, 9.6 & **515.1**, 10.6, 70-130% from last Calibration Verification
EPA 515.3, 552.2, 9.9, 70-130% from last Initial Calibration
EPA 515.4, 9.7, 50-150% from last Initial Calibration
EPA 551.1, 9.9, 80-120% from avg. of last 5 Calib. verifications
EPA 508.1, 9.4, 70-130% from last Calib. Verification & 50-150% from last Initial Calibration
EPA 548.1, 552.1, 9.5, 70-130% from last Calibration Verification
EPA 552.3, 9.6, 50-150% from last Initial Calibration
D5317-93, 19.6, decrease < 30% from last Calib. Verification

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

RCRA TOXICITY CHARACTERISTICS

Endrin	0.02 mg/L
Heptachlor & its Epoxide	0.008 mg/L
Lindane	0.4 mg/L
Methoxychlor	10.0 mg/L
Chlordane	0.03 mg/L
Hexachlorobenzene	0.13 mg/L
2,4-D	10.0 mg/L
Pentachlorophenol	100.0 mg/L
2,4,5-TP (Silvex)	1.0 mg/L

SDWA MAXIMUM CONTAMINANT LEVELS

Alachlor	2.0 ug/L
Atrazine	3.0 ug/L
Chlordane	2.0 ug/L
Endrin	2.0 ug/L
Heptachlor	0.4 ug/L
Heptachlor Epoxide	0.2 ug/L
Hexachlorobenzene	1.0 ug/L
Hexachlorocyclopentadiene	50 ug/L
Lindane	0.2 ug/L
Methoxychlor	40 ug/L
Simazine	4.0 ug/L
Toxaphene	3.0 ug/L
PCB's (as Decachlorobiphenyl)	0.5 ug/L
2,4-D	70 ug/L
Pentachlorophenol	1.0 ug/L
2,4,5-TP (Silvex)	50 ug/L
Dalapon	200 ug/L
Dinoseb	7.0 ug/L
Picloram	500 ug/L
Endothall	100 ug/L
Total Trihaloacetic Acids	60 ug/L

ADDITIONAL REQUIREMENTS

Matrix Spikes, Control Standards, & Duplicates at least 15% of workload for any parameter

USGS Bk. 5, Ch. A1, p.7, applies to all USGS Metals & General Chemistry mtds.

Matrix Spikes analyzed every 10 samples

EPA 505, 551.1, 9.6
EPA 515.1, 10.8
EPA 548.1, 552.1, 552.2, 9.7
EPA 508, 515.2, 9.8
EPA 608, 8.3
EPA 608.1, 614, 615, 617, 619, 622, 622.1, 627, 630, 630.1, 633, 633.1, 634, 637, 8.4, or Monthly
EPA 1656, 1657, 1658, 8.4. for **each site type**
EPA 1659, 1661, 8.3
SM6020B, 3c, or Monthly (whichever is more frequent) (applies to all SM Organics methods)
3M0222, 9.3
D5317-93, 19.8, or **batch**
USGS Bk.5, Ch.A3, p.5, applies to all USGS Organics mtds., not required if **Surrogates** analyzed **each sample**

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 515.4, 9.9
EPA 8000, 8.5, applies to all 8000-series Organics mtds., may use sample dup. in place of MSD

Matrix Spike every 20 samples

EPA 508A, 10.7

Quality Control Check Samples analyzed every 10 samples

EPA 608, 8.1.5, frequency may be reduced if Matrix Spike recoveries meet all specified QC criteria
EPA 608.2, 614.1, 632.1, 645, 9.2

Field Duplicates analyzed

EPA 515.4, 9.10, analyzed each extraction batch, matrix spike duplicate also allowed as alternative
EPA 551.1, 9.7, ALL samples are collected in duplicate; 10% are analyzed
EPA 552.3, 9.9, analyzed each extraction batch
EPA 608.2, 614.1, 632.1, 645, 9.3, ALL samples are collected in duplicate; 10% are analyzed

Reagent Blank analyzed every 10 samples

EPA 508A, 10.1

GC Injector Port Degradation < 20% for Endrin & DDT (evaluated each initial calibration)

EPA 505, 10.1 & **551.1**, 9.2 (Endrin only); **EPA 508**, 10.1, **EPA 508.1**, 10.3; & **EPA 1656**, 13.4

GC Injector Port Degradation < 15% for Endrin & DDT

EPA 8081, 8.3

GC Retention Time Windows established for each analyte

EPA 508A, 9.1.3 for Decachlorobiphenyl, <0.2% RSD required
EPA 8000, 7.6

Chromatographic Resolution Checks

EPA 551.1, 9.2, Bromacil & Alachlor, Bromodichloromethane & Trichloroethene each batch

GPC Column Calibration Acceptance Criteria (EPA 3640) (performed weekly)

Retention time shift < 5% compared with the previous calibration
Symmetrical peaks observed for all compounds
Resolution between Corn Oil, Bis(2-ethylhexyl) Phthalate, Methoxychlor, & Perylene peaks all > 85%
Resolution between Perylene & Sulfur peaks > 90% & neither peak is saturated in response

Initial Instrument Calibration Concentration Range Must Encompass Minimum Reporting Level of 1.0 ug/L

40 CFR Part 141, applies to Haloacetic Acids, 2.0 ug/L OK for Chloroacetic Acid