UV-VIS-IR SPECTROPHOTOMETRY (COLORIMETRY) & FLUORIMETRY

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 21-22 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 page 23 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 page 23 for acceptance criteria
____	**5.1.1**	Does the laboratory fulfill **additional requirements** specified in the **mandated test method** or **regulation** **Note:** See page 24 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 page 23 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

UV-VIS-IR SPECTROPHOTOMETRY (COLORIMETRY) & FLUORIMETRY

REQUIRED REAGANTS & STANDARDS

Alkalinity – EPA 310.2; USGS I-2030-85

Methyl Orange color reagent & KHP buffer (pH 3.1) for autoanalyzer (550 nm)

Aluminum – SM3500Al D (<=19th ed.), SM3500Al B (20th ed.)

Eriochrome Cyanine R color reagent (535 nm)
EDTA to serve as sample blank when added to a sample aliquot
Acetate Buffer to adjust sample pH around 6

Ammonia Distillation – SM4500NH3 B (required unless comparability data for representative effluents proves otherwise)

Sodium Hydroxide distillation reagent
Indicating Boric Acid receiver solution

Ammonia – EPA 350.2; ASTM D1426-98A; USGS I-3520-85; AOAC 973.49; SM4500NH3 C (<=18th ed.)

Nessler Reagent for colorimetry (Mercuric Iodide, Potassium Iodide, Sodium Hydroxide) (425 nm)

Ammonia – EPA 350.1; SM4500NH3 G (>=19th ed.), SM4500NH3 H (<=18th ed.); USGS I-4523-85; SM4500NH3 F (>=19th ed.), SM4500NH3 D (<=18th ed.) (manual)

Heating Bath on-line with Autoanalyzer
Sodium Phenate & Sodium Hypochlorite color reagents (630 nm)
EDTA or Sodium Potassium Tartrate to prevent precipitation of divalent metal ions
Sodium Nitroprusside catalyst

Arsenic – EPA 206.4; SM3500As C (<=19th ed.), SM3500As B (20th ed.); ASTM D2972-97A; USGS I-3060-85

Zinc Metal or Sodium Borohydride to produce arsine gas
Silver Diethyldithiocarbamate color reagent (510 nm)
Lead Acetate impregnated glass wool, to remove hydrogen sulfide interference

Beryllium – SM3500Be D (<=19th ed.)

Aluminon color reagent (Triammonium Aurintricarboxylate) (515 nm)
EDTA to complex transition metals (particularly copper)

Boron – EPA 212.3; SM4500B B; USGS I-3112-85

Curcumin color reagent (with oxalic acid) (540 nm), ethanol solvent
Cation Exchange Resin to remove metal interferences
Boric Acid standard (H3BO3)

Boron – SM4500B C

CARMINE/Sulfuric Acid color reagent (585 nm)
Alkaline digestion, ignition to dryness, HCl to dissolve
Boric Acid standard

Bromide – SM4500Br- B

Phenolsulfonephthalein (Phenol Red) / Chloramine-T / pH 4.5 Acetate Buffer color reagent (590 nm)
Thiosulfate to remove excess chlorine from chloramine-T

Cadmium – SM3500Cd D (<=19th ed.)

Dithizone color reagent (Diphenylthiocarbazone) (518 nm)
Chloroform extraction solvent

Chemical Oxygen Demand – EPA 410.4; SM5220D; ASTM D1252-95B; OIC Method; USGS I-3561-85; HACH8000

Digestion Reagent (Potassium Dichromate, Silver Sulfate to oxidize aliphatics, Mercuric Sulfate to precipitate halides, Sulfamic Acid to oxidize nitrites, Sulfuric Acid) (150 C for 2 hours)
KHP standard (Potassium Hydrogen Phthalate)
Closed Reflux digestion system

Chloride – EPA 325.1, 9250

Ferric Ammonium Sulfate & Nitric Acid color reagent for autoanalyzer (480 nm)
Mercuric Thiocyanate

Chloride – EPA 325.2, 9251; SM4500Cl- E; USGS I-1187-85 (manual method), I-2187-85

Ferric Nitrate & Mercuric Thiocyanate combined color reagent for autoanalyzer (480 nm)

Chlorine – EPA 330.5; SM4500CL G

DPD color reagent (515 nm)
Chlorine standards (KMnO4 as chlorine equivalent, or ClO- with KI added & standardized w/ Thiosulfate)

Free Chlorine – SM4500CL H

Syringaldazine color reagent in isopropanol (3,5-Dimethoxy-4-hydroxybenzaldehyde) (530 nm)
Phosphate Buffer to adjust sample pH to 6.5-6.8

Chlorine Dioxide & Chlorite – EPA 327.0

Lissamine Green B to react with Chlorine Dioxide (absorbance at 633 nm decreases)
Horseradish Peroxidase to reduce Chlorite to Chlorine Dioxide
Glycine-Citrate buffer to eliminate free chlorine interference

Chlorophylls – EPA 445.0, SM10200H

Magnesium Carbonate & Acetone to extract Chlorophylls from plant tissue
Hydrochloric Acid, used so that Chlorophyll may be measured in the presence of Pheophytin
SM10200H: High-resolution Spectrophotometer (0.5-2.0 nm bandpass) to measure:
664 nm before acidification, 665 nm after acidification, 750 nm to correct for turbidity;
664, 647, 630, & 750 nm (trichromic method)
EPA 445.0: Fluorimeter
SM10200H: Spectrofluorimeter (430 nm excitation wavelength, 663 nm emission wavelength)
SM10200H: HPLC (reverse-phase column, fluorescence detector)

Chromium & Chromium(VI) – EPA 218.4 (option), 7196; SM3500Cr D (<=19th ed.), SM3500Cr B (20th ed.); ASTM D1687-92A; USGS I-1230-85

1,5-Diphenylcarbazide color reagent (540 nm)
Cupferron, to remove Mo/V/Cu/Fe interferences
Potassium Permanganate (KMnO4), to oxidize Chromium to Cr(VI)
Sulfuric Acid, to adjust sample pH for color development (**ASTM**: Phosphoric Acid)
EPA 7196: Add color reagent first, then adjust pH to 1.5-2.5
SM, USGS: Adjust pH to 0.7-1.3, then add color reagent

Color – EPA 110.1; SM2120E; NCPI Tech. Bulletin 253

Photometer with Tristimulus Filters
Sulfuric Acid or Sodium Hydroxide to adjust sample pH to 7.6 (color reported for both pH 7.6 & original pH)

Color – EPA 110.2; SM2120B; USGS I-1250-85

Platinum-Cobalt Color standards (Potassium Chloroplatinate & Cobalt Chloride Hexahydrate)

Color – EPA 110.3; SM2120C

Spectrophotometer with bandpass < 10 nm (400-700 nm measured)
Sulfuric Acid or Sodium Hydroxide to adjust sample pH to 7.6 (color reported for both pH 7.6 & original pH)

Copper – SM3500Cu D (<=19th ed.), SM3500Cu B (20th ed.)

Neocuproine color reagent (2,9-Dimethyl-1,10-phenanthroline hemihydrate) (457 nm)
Chloroform extraction solvent
Methanol dilution solvent
Hydroxylamine Hydrochloride, to reduce Cu(II) to Cu(I)
Sodium Citrate, to complex metal ions that might precipitate when sample pH is raised to 6

Copper – SM3500Cu E (<=19th ed.), SM3500Cu C (20th ed.); HACH8506

Bathocuproine color reagent (Disodium 2,9-Dimethyl-4,7-diphenyl-1,10-phenanthroline disulfonate)
Hydroxylamine Hydrochloride, to reduce Cu(II) to Cu(I) (484 nm)
Sodium Citrate, to buffer sample pH around 4.3

Cyanide Distillation – EPA 335.4, 9010; SM4500CN- C

Sulfuric Acid, added to liberate HCN
Sodium Hydroxide, scrubber solution to trap HCN
Magnesium Chloride Hexahydrate, catalyst for the distillation
Lead Carbonate, added to scrubber solution to precipitate sulfides
Sulfamic Acid, added to distillation solution to eliminate nitrate & nitrite interferences
Bismuth Nitrate, added to distillation solution to precipitate sulfides
Sodium Arsenite, to remove chlorine & other oxidizing agents (that decompose cyanides)

Total Cyanide – EPA 335.2, 335.3, 335.4, 9012, 9014; SM4500CN- E; ASTM D2036-98A; USGS I-3300-85

Phosphate or Acetate Buffer, to adjust sample pH to 4.5
Chloramine-T, to generate cyanogen chloride (prepare weekly)
Pyridine-Barbituric Acid color reagent (578 nm)
Autoanalyzer (**EPA 335.3, 335.4, 9012**; optional for **ASTM D2036-91A**)
Silver Nitrate, to standardize Cyanide stock standard

Cyanide Amenable to Chlorination – EPA 335.1, 9010, 9012; SM4500CN- G; ASTM D2036-98B

(required under SDWA if Total CN- is > 0.2 mg/L)
Calcium Hypochlorite, to generate excess chlorine
Sodium Arsenite or Ascorbic Acid, to remove excess chlorine after the 1-hour reaction time
Same reagents for Cyanide Distillation & for Total Cyanide (both aliquots must be distilled)

Fluoride Distillation – SM4500F- B (required under CWA unless comparability data for representative effluents shows that preliminary distillation is unnecessary; required for SDWA UV-VIS methods)

Sulfuric Acid, to liberate HF & Fluosilicic Acid
Soft Glass boiling beads, to convert HF to Fluosilicic Acid
Silver Sulfate, to eliminate chloride interference if necessary

Fluoride – EPA 340.1; SM4500F- D; ASTM D1179-93A

SPADNS color reagent (Sodium 2-(Parasulfophenylazo)-1,8-dihydroxy-3,6-naphthalene disulfonate)
Zirconyl-Acid reagent (Zirconyl Chloride Octahydrate & Hydrochloric Acid) (570 nm)

Fluoride – EPA 340.3; SM4500F- E; Technicon 129-71W

Complexone color reagent (Acetate Buffer, Acetone, t-Butanol, Alizarin Fluorine Blue, Lanthanum Nitrate; Added in this order for the combined reagent) (620 nm)

Formaldehyde – EPA 8520

Pararosaniline, Hydrochloric Acid, & Sodium Sulfite as color reagent (550 nm)

Hardness – EPA 130.1 (Autoanalyzer)

Ammonia Buffer, to adjust sample pH to 10
Magnesium-EDTA (releases Mg when Ca from the sample is preferentially complexed)
Calmagite Color Indicator (complexes with free Mg) (520 nm)

Iron – SM3500Fe D (<=19th ed.), SM3500Fe B (20th ed.); ASTM D1068-96D; HACH8008

Phenanthroline color reagent (1,10-Phenanthroline Monohydrate) (510 nm)
Hydroxylamine Hydrochloride, to reduce Fe(III) to Fe(II)
Ammonium Acetate Buffer, to adjust sample pH to 3.2-3.3

Kjeldahl Nitrogen Digestion – EPA 351.2; SM4500Norg B, SM4500Norg C; ASTM D3590-89B; USGS I-4515-91

Digestion reagent (Sulfuric Acid; Potassium Sulfate; Mercuric Sulfate, Copper Sulfate, or Selenium)

Kjeldahl Nitrogen – EPA 351.3; ASTM D3590-89A; PAI-DK02; SM4500NH3 C (<=18th ed.)

Sodium Hydroxide distillation reagent
Indicating Boric Acid receiver solution
Nessler Reagent for colorimetry (Mercuric Iodide, Potassium Iodide, Sodium Hydroxide)

Kjeldahl Nitrogen – EPA 351.1; SM4500NH3 G (>=19th ed.), SM4500NH3 H (<=18th ed.); USGS I-4551-78; SM4500NH3 F (>=19th ed.), SM4500NH3 D (<=18th ed.) (manual)

Digestion Block & Heating Bath on-line with Autoanalyzer
Sodium Phenate & Sodium Hypochlorite color reagents (630 nm)
EDTA or Sodium Potassium Tartrate to prevent precipitation of divalent metal ions
Sodium Nitroprusside catalyst

Kjeldahl Nitrogen – EPA 351.2; ASTM D3590-89B; USGS I-4515-91

Sodium Salicylate & Sodium Nitroprusside color reagent
Sodium Hypochlorite solution
Ammonium Chloride, Sodium Potassium Tartrate, Buffer solution

Kjeldahl Nitrogen – PAI-DK03

Gas-diffusion membrane on Autoanalyzer into proprietary color reagent (590 nm)

Lead – SM3500Pb D (<=19th ed.), SM3500Pb B (20th ed.)

Dithizone color reagent (510 nm)
Chloroform extraction solvent
Citrate-Cyanide reducing solution (Ammonium Citrate, Sodium Sulfite, Hydroxylamine Hydrochloride, KCN)
(Sample pH is 10-11.5 so that dithizone complexes of interferences are only partially extracted)

Manganese – SM3500Mn D (<=19th ed.), SM3500Mn B (20th ed.); AOAC 920.203; HACH8034 (periodate oxidizing agent)

Ammonium Persulfate, to oxidize Mn to permanganate (525 nm)

Mercury – SM3500Hg C (<=19th ed.)

Dithizone color reagent (492 nm)
Chloroform extraction solvent
Permanganate/Persulfate preliminary digestion reagent, the Hydroxylamine decolorizing reagent
H2SO4/KBr to put initial Hg-Dithizonate extraction complex back to aqueous phase
Phosphate/Carbonate Buffer

Nickel – SM3500Ni D (<=17th ed.)

Heptoxime color reagent (1,2-Cycloheptanedionedioxime) (445 nm)
Cupferron, to remove Cu & Fe interferences
Chloroform extraction solvent
Hydrochloric Acid, to re-extract Ni-heptoxime back to aqueous phase

Nitrate – EPA 352.1, 9200; AOAC 973.50; ANSI Photo. Effluents

Brucine-sulfanilic Acid color reagent (410 nm)
Sodium Arsenite to remove residual chlorine
Acid or Base to adjust sample pH to 7

Nitrate – calculation from Total Nitrate-Nitrite minus Nitrite

Total Nitrate-Nitrite – EPA 353.3; SM4500NO3- E; ASTM D3867-99B

Cadmium coated with Copper Sulfate, to reduce Nitrate to Nitrite
Ammonium Chloride & EDTA Buffer, to adjust sample pH to 8.5 & to keep Cd column in good condition
Sulfanilamide, Phosphoric Acid, & N-(1-naphthyl)-ethylenediamine dihydrochloride color reagent (543 nm)

Total Nitrate-Nitrite – EPA 353.2; SM4500NO3- F; ASTM D3867-99A; I-4545-85

Same reagents as above for Autoanalyzer

Total Nitrate-Nitrite – EPA 353.1; SM4500NO3- H

Hydrazine Sulfate reducing agent
Same color reagent for Total Nitrate-Nitrite above

Nitrite – EPA 354.1; SM4500NO2- B; ASTM D1254-67; USGS I-4540-85 (autoanalyzer); HACH8507

Sodium Oxalate or Ferrous Ammonium Sulfate, plus Potassium Permanganate, to standardize Nitrite stds
Same color reagent as Total Nitrate-Nitrite above

Total Organic Carbon – EPA 415.1, 415.2, 415.3, 9060; SM5310B, SM5310C, SM5310D; ASTM D2579-93A, D2579-93B; AOAC 973.47

KHP Organic Carbon standard (Potassium Hydrogen Phthalate)
Phosphoric Acid (or other acid), to purge out inorganic carbonate
Sodium Carbonate Inorganic Carbon standard
Sample Combustion – **EPA 415.1, 415.3, 9060; SM5310B**
Sample Persulfate Oxidation – **EPA 415.1, 415.3, 9060**; UV-assisted for **EPA 415.2, 415.3, SM5310C**
Sample Autoclaved in sealed ampules with Persulfate Oxidation at 116-130 C for 4 hr – **SM5310D**
Infrared Detection of CO2 – all methods
Conversion to Methane & Flame Ionization Detection – **EPA 415.1, 415.2, 9060; SM5310C**

Organic Nitrogen – calculation as Total Kjeldahl Nitrogen minus Ammonia

Ozone – SM4500O3 B

Indigo color reagent (Potassium Indigo Trisulfonate) (600 nm) (Indigo absorptivity is the standard here)
Malonic Acid, to control chlorine interference if necessary
Glycine, to compensate for Mn(II) interference, if necessary

Total Recoverable Petroleum Hydrocarbons – EPA 418.1, 8440, SM5520F (Oil & Grease EPA 413.2, SM5520C)

Trichlorotrifluoroethane extraction solvent (**EPA 418.1, 413.2, SM5520C, SM5520F**)
Supercritical Carbon Dioxide (**EPA 8440**)
Sodium Sulfate drying agent
Silica Gel clean-up material (Oil & Grease **EPA 413.2 & SM5520C** omit this cleanup step)
Isooctane, Hexadecane, & Benzene reference standard (3200-2700 cm-1, max at 2930 cm-1) (**SM5520C**)
Isooctane, Hexadecane, & Chlorobenzene reference oil (2800-3000 cm-1 hydrocarbon range, 1600-1800 cm-1 ester range) (**EPA 413.2, 418.1, 8440**)

Total Phenols – EPA 420.1, 9065, SM5530C, SM5530D (Autoanalyzer EPA 420.2, 420.4, 9066)

Copper Sulfate & Phosphoric Acid, or Sodium Hydroxide, to adjust sample pH to 4 prior to distillation
Sodium Hydroxide scrubber solution
4-Aminoantipyrene color reagent (500 or 510 nm) (direct read for all methods except SM5530C)
EPA 410 (optional), **SM5530C**: Chloroform extraction solvent (for enhanced sensitivity) (460 nm)

Total Phenols – EPA 420.3, 9067

Copper Sulfate & Phosphoric Acid, or Sodium Hydroxide, to adjust sample pH to 4 prior to distillation
Sodium Hydroxide scrubber solution
MBTH color reagent (3-Methyl-2-benzothiazolinone Hydrazone Hydrochloride) (520 nm)
Ceric Ammonium Sulfate oxidizing agent
EDTA & Borate Buffer

Orthophosphate – EPA 365.1; SM4500P F; USGS I-2598-85, I-2601-85, I-4601-85 (sample unfiltered); AOAC 973.56

Combined color reagent (Sulfuric Acid, Potassium Antimony Tartrate, Ammonium Molybdate, Ascorbic Acid; added together in this order; good only for 4 hours) (880 nm) (Autoanalyzer)
Sodium Hydroxide & Phenolphthalein indicator, to adjust sample pH to 7
Potassium Dihydrogen Phosphate standard

Orthophosphate – EPA 365.2; SM4500P E; ASTM D515-88A; USGS I-1602-85; AOAC 973.55

Same reagents as above, for manual technique

Orthophosphate – EPA 365.3

Double color reagent (Ascorbic Acid separate from the Acid, Tartrate, & Molybdate reagent) (manual method)
Sodium Hydroxide & Phenolphthalein indicator, to adjust sample pH to 7
Potassium Dihydrogen Phosphate standard

Orthophosphate – SM4500P C

Ammonium Molybdate & Ammonium Metavanadate color reagent (manual method) (400, 420, or 470 nm)
Sodium Hydroxide & Phenolphthalein indicator, to adjust sample pH to 7
Potassium Dihydrogen Phosphate standard

Orthophosphate – SM4500P D

Color reagent (Sulfuric Acid Potassium Antimony Tartrate, Ammonium Molybdate) (manual method) (690 nm)
Tin(II) Chloride reducing agent (instead of Ascorbic Acid)
Sodium Hydroxide & Phenolphthalein indicator, to adjust sample pH to 7
Potassium Dihydrogen Phosphate standard

Total Phosphorus – EPA 365.1, 365.2, 365.3; SM4500P C, SM4500P D, SM4500P E, SM4500P F; ASTM D515-88A; USGS I-4600-85; AOAC 973.55, 973.56

Sulfuric Acid & Ammonium Persulfate digestion solution
Same reagents as for the corresponding Orthophosphate methods above

Total Phosphorus – EPA 365.4; ASTM D515-88B

Kjeldahl Nitrogen digestion solution (Sulfuric Acid, Potassium Sulfate, Mercuric Oxide or Copper Sulfate)
Autoanalyzer with combined color reagent (see EPA 365.1 above)

Selenium – SM3500Se D (<=19th ed.), SM3500Se C (20th ed.) (UV-VIS); SM3500Se E (<=19th ed.) (Fluorimetry)

2,3-Diaminonaphthalene color/fluorimetric reagent (480 nm absorption; 369 nm excitation, 525 nm emission)
Cyclohexane extraction solvent
Persulfate digestion to oxidize organic interferences
Peroxide/Hydroxide digestion reagent to oxidize Selenium species to Se(VI)
Permanganate digestion reagent, then Hydroxylamine to decolorize
Conc. HCl to reduce Se(VI) to Se(IV)
HCl & Ammonia-water to adjust sample pH to 1.2-1.8 (UV-VIS) or 1.7-2.0 (Fluorimetry) prior to color formation

Dissolved Silicate – EPA 366.0 (autoanalyzer)

Sodium Hexafluorosilicate for calibration standards
Ammonium Molybdate & Oxalic Acid color reagent (660 nm)
Ascorbic Acid reducing agent

Dissolved Silica – EPA 370.1; SM4500Si D (<=19th ed.), SM4500SiO2 C (20th ed.); ASTM D859-94; USGS I-1700-85; USGS I-2700-85 (autoanalyzer)

Ammonium Molybdate & Oxalic Acid color reagent (410 nm)

Dissolved Silica – EPA 370.1 (Si < 1 mg/L); SM4500Si E (<=19th ed.), SM4500SiO2 D (20th ed.); EPA 366.0, SM4500Si F (<=19th ed.), SM4500SiO2 E (20th ed.) (autoanalyzer)

Ammonium Molybdate & Oxalic Acid color reagent (650 or 815 nm)
1-Amino-2-naphthol-4-sulfonic Acid reducing agent

Silver – SM3500Ag D (<=19th ed.)

Dithizone color reagent (620 or 462 nm)
Carbon Tetrachloride extraction solvent
Digestion Reagents: Nitric/Sulfuric Acid, Sulfuric Acid, Urea/Hydroxylamine
Ammonium Thiocyanate, to put initial Ag-Dithizone extraction complex back to aqueous phase

Sulfate – EPA 375.1, 9035

Barium Chloranilate color reagent (520 nm)
Acetate Buffer, to adjust sample pH to 4.63
Cation Exchange Resin, to remove Ca, Al, & Fe interferences (which precipitate the chloranilate)

Sulfate – EPA 375.2, 9036; SM4500SO4= F

Barium Chloride & Methylthymol Blue (3,3'-Bis-N,N-bis(carboxymethylamino)methylthymolsulfone-phthalein, Pentasodium salt) & Hydrochloric Acid color reagent (460 nm for autoanalyzer)
Cation Exchange Resin, to remove multivalent cation interferences
Ammonia or EDTA Buffer, to adjust sample pH around 10.5

Sulfide – EPA 376.2; SM4500S= D

Methylene Blue standard
Amino-sulfuric Acid color reagent (N,N-Dimethyl-p-phenylenediamine Oxalate in Sulfuric Acid) (664 nm)

Sulfite – SM4500SO3= C

Ferric Ammonium Sulfate & 1,10-Phenanthroline color reagent & absorber solution (510 nm)
Ammonium Bifluoride to remove excess Fe(III)
K2HgCl4 to stabilize Na2SO3 standard
HCl & Sulfamic Acid to remove SO3= from sample as SO2

Surfactants – EPA 425.1; SM5540C; ASTM D2330-88

Linear Alkylbenzenesulfonate standard, with number-average Molecular Weight documented
Methylene Blue ion-pairing agent (652 nm)
Chloroform extraction solvent
Sodium Sulfate drying agent

Tannin & Lignin – SM5550B

Folin Phenol color reagent (Sodium Tungstate, Sodium Molybdate, Phosphoric Acid, Hydrochloric Acid, Lithium Sulfate, & Bromine-water) (700 nm)
Sodium Carbonate & Sodium Tartrate reagent

UV 254 – EPA 415.3; SM5910B

Potassium Hydrogen Phthalate standard

Vanadium – SM3500V D (<=19th ed.), SM3500V B (20th ed.)

Gallic Acid color reagent (415 nm)
Mercuric Nitrate, to eliminate bromide & iodide interferences
Ammonium Persulfate & Phosphoric Acid oxidizing agent (Vanadium is the catalyst)

Waste Reactivity Distillation – Section 7.3 of the SW-846 Manual

Sulfuric Acid, to release reactive gases (30-minute test period, no heating, constant stirring)
Sodium Hydroxide, scrubber solution to collect reactive gases

Zinc – SM3500Zn E (<=19th ed.)

Dithizone color reagent (535 nm)
Carbon Tetrachloride extraction solvent
Bis(2-hydroxyethyl)dithiocarbamate (prepared from Diethanolamine & Carbon Disulfide),
to prevent other metals from reacting with dithizone

Zinc – SM3500Zn F (<=19th ed.), SM3500Zn B (20th ed.); HACH8009

Zincon color reagent (2-Carboxy-2'-hydroxy-5'-sulfoformazyl benzene) (620 nm)
Borate Buffer, to adjust sample pH to 9
Ascorbic Acid, Borate Buffer, Potassium Cyanide, & Zincon added to sample in this order

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

Analyze Immediately in the field or upon arrival at the laboratory, plastic or glass containers

Total Residual Chlorine, Orthophosphate (filtration step only)

Analyze Immediately in field or upon arrival at the laboratory, glass bottle & top

Ozone

24-Hour Holding Time, plastic or glass containers, 4 C

Chromium(VI)

24-48 Hour Holding Time, plastic or glass containers, store in the dark at 4 C, unfiltered

Chlorophyll

48-Hour Holding Time, plastic or glass containers, 4 C, unpreserved

Color, Nitrate, Nitrite, Orthophosphate, Surfactants

7-Day Holding Time, plastic or glass container, 4 C, Zinc Acetate & NaOH to pH>9

Sulfide (analyze immediately if sample unpreserved)

14-Day Holding Time, plastic or glass containers, 4 C

Acidity, Alkalinity, Nitrate (SDWA chlorinated samples)

14-Day Holding Time, plastic or glass containers, 4 C, NaOH to pH>12

Total & Amenable Cyanide (24-Hour Holding Time if Sulfide is present)
(Add NaAsO2 or Ascorbic Acid if oxidizing agents present (RCRA))

28-Day Holding Time, plastic or glass containers, 4 C

Chloride, Sulfate

28-Day Holding Time, plastic container (only)

Fluoride

28-Day Holding Time; plastic, Teflon, or quartz-glass containers, 4 C

Dissolved Silica

28-Day Holding Time, plastic or glass containers, 4 C, Sulfuric Acid to pH<2

Ammonia, Chemical Oxygen Demand, Total Kjeldahl Nitrogen, Organic Nitrogen,
Total Nitrate-Nitrite, Total Phosphorus

28-Day Holding Time, glass container (only), 4 C, Sulfuric Acid to pH<2
Total Phenols

28-Day Holding Time; plastic or glass containers; 4 C; HCl, H2SO4, or H3PO4 to pH<2
Total Organic Carbon

28-Day Holding Time, plastic or glass containers, store in the dark frozen at –20 C, filtered
Chlorophylls

6-Month Holding Time, plastic or glass containers, Nitric Acid to pH<2
Metals (except Cr(VI) & Hg; add HNO3 if sample unpreserved & let stand for 16 hours prior to analysis)

6-Month Holding Time, plastic or glass containers, Nitric or Sulfuric Acid to pH<2
Hardness

6-Month Holding Time; plastic, Teflon, or quartz-glass containers; HNO3 to pH<2
Boron

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank

SM1020B, 5, applies to all mandated SM methods unless specified in individual methods
EPA 327.0, 10.2, must use **linear regression**
EPA 335.4, 350.1, 351.2, 353.2, 365.1, 410.4, 420.4, 10.1
EPA 352.1, 353.1, 353.3, 354.1, 375.1, 8.1
EPA 350.2, 7.4
EPA 351.1, 365.2, 365.3, 9.1
EPA 351.3, 8.4
EPA 365.4, 7.1
EPA 630, 7.4
EPA 8520, 7.1.3, calibration blank required between each std.
EPA 9035, 9036, 9065, 9066, 9067, 9250, 9251, 8.2
D1687-92A, 12.4, **daily**
PAI-DK02

4 standards + blank

SM4500B B, 4b
EPA 415.3, 10.2
D859-94, D2579-93, 10.1 (Si & TOC, respectively)
I-4540-85, 5.4 & 6.4, **daily** (Nitrite)

5 standards + blank

EPA 420.1, 8.2 or 8.3
EPA 9036, 8.2
SM3500Hg C, SM4500B C, 4b
SM5540C (20th ed.), 4a, linear regression required with r>0.995
D1426-93A, 12.1, for NH3 by Nesslerization
D1252-95B, 23.1 & 23.2, for both high-level & low-level COD
D515-88A, 13.1
I-4523-85, 5.4 & 6.5, **daily**
I-1187-85, 6.2, **daily**
I-2598-85, 5.7 & 6.2, **daily**
I-2601-85, I-4601-85, 5.8 & 6.4, **daily**
I-4600-85, 5.8 & 6.5, **daily**

6 standards + blank

SM4500Br- B, 4a
SM4500P E, 4c
D3867-90B, 21.1, for Nitrate by manual cadmium reduction
D2330-88, 11.1
EPA 9012, 7.4 & **EPA 9014**, 7.3

7 standards + blank

D3590-89A, 12.4.1.1, for TKN by Nesslerization
D516-90, 10.1
AOAC973.48, AOAC973.49, refers to AOAC973.48F, **daily**
AOAC973.56, C(e) & D(c), **daily**

8 standards

D3867-90A, 13.1, for Nitrate by autoanalyzer cadmium reduction
I-2187-85, 5.2 & 6.4, blank also required, **daily**
I-2700-85, 5.6 & 6.4, blank also required, **daily**
AOAC973.55, D(g) & E(d), blank also required, **daily**
AOAC973.47, D(b) & F(a), for TOC, blank also required, **daily, 5 TIC stds.** plus blank also required daily

9 standards + blank

I-4545-85, 5.9 & 6.9, **daily**
PAI-DK03

10 standards

SM5540C (<=19th ed.), 4a

12 high-level & low-level working standards (6 standards for each level)

EPA 375.2, 10.1

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 70-130%

EPA 327.0, 10.3

Recovery 85-115%

EPA 415.3, 10.3, for 5-50 mg/L TOC, 80-120% for 10-50 mg/L TOC, 50-150% for TOC<0.7 mg/L, CCV analyzed **every 10 samples & end of run**
EPA 9012, 8.2

Recovery 90-110%

PAI-DK02, PAI-DK03, also after **every 10 samples & end of run**
EPA 335.4, 350.1, 351.2, 353.2, 365.1, 375.2, 410.4, 420.4, 9.3.4 after **every 10 samples & end of run**, calibration blank analysis also required each time
SM5540C (20th ed.), 4a, also requires **Reporting Limit std.** each day with recovery 75-125%

Recovery 98-102%

AOAC973.55, E(d), must use at least **2 stds.**

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

Method Detection Limits & Linear Dynamic Range (or Linear Calibration Range) evaluations required for each analyte & wavelength used – upper limit within 10% of extrapolated value
EPA 335.4, 350.1, 351.2, 353.2, 365.1, 375.2, 410.4, 420.4, 9.2.2, 3 stds. needed & verified at least **every 6 months**
EPA 366.0, 445.0, 9.2.1

Mean Recovery 70-130%, Precision < 20% RPD
EPA 327.0, 9.2, from 5 replicates, **MDL also required**

Mean Recovery 80-120%, Precision < 20% RPD
EPA 415.3, 9.3.5-9.3.6, from 5 replicates at 2-5 mg/L TOC, **MDL also required**

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample Recoveries within 80-120%
EPA 415.3, 9.7
PAI-DK02, PAI-DK-03

QC Check Sample Recoveries within 85-115%
EPA 9010/9014, 8.3

QC Check Sample Recoveries within 90-110%
EPA 335.4, 350.1, 351.2, 353.2, 365.1, 366.0, 375.2, 410.4, 420.4, 9.3.2-9.3.3 (Inorganics)

External QC Check Sample Recoveries within 80-120%
EPA 415.3, 9.11, analyzed quarterly **in triplicate**

External QC Check Sample Recoveries within 90-110%
PAI-DK02, PAI-DK03

EPA QC Check Sample Recoveries within 90-110%
EPA 335.4, 350.1, 351.2, 365.1, 375.2, 410.4, 420.4, 9.2.3 analyzed quarterly (Inorganics)

Matrix Spike Recoveries within 80-120%
PAI-DK-02, PAI-DK03, after **every 10 samples**

Matrix Spike Recoveries within 90-110%
EPA 335.4, 350.1, 351.2, 353.2, 365.1, 375.2, 410.4, 420.4, 9.4, analyzed **every 10 samples** (Inorganics)

Method Blank Results
EPA 415.3, 9.9, < 0.35 mg/L TOC or DOC and < 0.01 cm-1 for UVA

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

Nitrate	10.0 mg/L as N	Nitrite	1.0 mg/L as N
Free Cyanide	0.2 mg/L	Fluoride	4.0 mg/L
Chlorine	4.0 mg/L as Cl2		

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 9012, 8.3

Matrix Spike every 10 samples, or Matrix Spike & Duplicate every 20 samples
SM1020B, 2 (applies to all SM methods unless more stringent requirements appear elsewhere)

Duplicate every 10 samples or analytical batch
SM2020 (applies to all SM2000-series methods)
D2579-93, 14.2 & 14.4, or **daily**

Matrix Spike & Sample Duplicate every 20 samples
EPA 415.3, 9.6 & 9.8, field duplicate in lieu of sample duplicate
EPA 9010/9014, 8.4 & 8.5

Matrix Spike each day of analysis
D2579-93, 14.3

Independent Quality Control Check Samples analyzed every 15 samples
EPA 9060, 8.3

Spike Duplicate analyzed every 10 samples
EPA 7196, 9250, 9251, 8.5, sample duplicate also allowed for Cr(VI)
EPA 9035, 9036, 9065, 9066, 9067, 8.6
EPA 9060, 8.4

All Samples analyzed in Duplicate
EPA 9060, 7.6, quadruplicate analyses required for TOC
AOAC973.47, F(a) & G

Calibration Verification every 15 samples
EPA 9035, 9036, 9065, 9066, 9067, 8.5, second-source std.
EPA 7196, 9250, 9251, 8.4, second-source std.
EPA 9060, 8.3

Reduction Efficiency of Nitrate to Nitrite checked each analytical batch
EPA 353.1, 7.3
SM4500NO3- E, 4c
SM4500NO3- F, 3k
D3867-90A, 13.2 & 14.5
D3867-90B, 21.2-21.3