PHYSICAL PROPERTIES

Note: Make enough copies of Pages 1-9 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 Does the laboratory clearly indicate in its methods manual any modifications made to the 5.5.4.1.2(b) 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** Scope & application, including components to be analyzed 5.5.4.1.2(b)(4) 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 **Equipment & supplies** 5.5.4.1.2(b)(9) 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:

 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

 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

 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
 5.1.1	 Does the laboratory fulfill additional requirements specified in the mandated test method or regulation Note: See page 11 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

 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

	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 OC 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
		 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:

 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

	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).

	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 LOO
		Note: A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria or client data quality objectives for accuracy.
		Note: This single analysis is not required if the bias & precision of the measurement system are evaluated at the LOQ
		Note: The LOQ verification is not required 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: 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

PHYSICAL PROPERTIES

REQUIRED REAGANTS & STANDARDS

Corrosivity (Langlier Index) – SM2330B (calculation from pH, TDS, Ca, Temperature, Alkalinity)

Hardness - calculation from Calcium & Magnesium

pH - EPA 9041

Wide & narrow-range pH paper pH Standard Buffers

Ignitability (Flash Point) - EPA 1010, 1020, 1030

Pensky-Martin Closed-cup tester w/ working stirrer & motor (EPA 1010) Setaflash Closed Tester (EPA 1020, not for use for liquids that produce surface films) Burn rate of solid along an unbroken strip (EPA 1030)

Magnesium – SM3500Mg E (<=19th ed.), SM3500Mg B (20th ed.) – calculation from Total Hardness minus Calcium

Nitrate - calculation from Total Nitrate-Nitrite minus Nitrite

Odor – EPA 140.1, SM2150B

EPA: At least **2 people needed**, one to make dilutions and at least 1 additional person(s) to sniff samples **SM**: Record **temperature** during test conditions, but 40 C is the standard test temperature (cold), or 60 C (hot)

Organic Nitrogen - calculation as Total Kjeldahl Nitrogen minus Ammonia

Salinity - SM2520C

Hydrometers, checked for accuracy annually, or Densitometer

Temperature - EPA 170.1; SM2550B

NIST-traceable thermometer, with scale graduations of 0.1 degrees Celsius

Un-Ionized Ammonia - DEP SOP 10-3-83 (calculation from Ammonia, pH, and Temperature)

(RCRA Characterizations)

Cation Exchange Capacity of Soils – EPA 9080

pH 7.0 Ammonium Acetate, to substitute for exchangeable cations in the soil Ammonium Chloride leaching solutions Aeration Method: Sodium Carbonate, to release ammonia, Sulfuric Acid acrubber solution, Sodium Hydroxide Titrant, Methyl Red indicator (red to yellow)

Acid-NaCl Method: Sodium Chloride acidified with Hydrochloric Acid, Boric Acid collection solution, Sulfuric Acid titrant, Bromcresol Green-Methyl Red mixed indicator (blue-green to blue-violet to pink)

Cation Exchange Capacity of Soils – EPA 9081

Sodium Acetate, to substitute for exchangeable cations in the soil Isopropanol wash solution Ammonium Acetate, to release adsorbed Sodium (which is subsequently determined)

Dermal Corrosion – EPA 1120

Macromolecular Biobarrier, Diluent, & Confirmation Test Solution

EP-TOX Extraction – EPA 1310

Acetic Acid, to adjust sample pH to 5.0 or below before & during extraction (24 hours)

Extraction Procedure for Oily Wastes (to determine Mobile Metal Concentration) – EPA 1330

THF & Toluene Soxhlet extraction reagents (EP-TOX extraction then conducted)

Toxicity Characteristic Leaching Procedure (TCLP) – EPA 1311

Acetic Acid & Sodium Hydroxide, to prepare extraction fluids (pH 4.88-4.98 for Fluid #1, pH 2.83-2.93 for Fluid #2, test before each use & prepare fresh Fluids if pH specifications not met)

Synthetic Precipitation Leaching Procedure (SPLP) – EPA 1312

Sulfuric & Nitric Acids, to prepare extraction fluids (pH 4.15-4.25 for Fluid #1, pH 4.95-5.05 for Fluid #2, Reagent water used for Fluid #3)

Multiple Extraction Procedure (MEP) – EPA 1320

Sulfuric & Nitric Acids, to prepare Synthetic Acid Rain Extraction Fluid (pH 2.8-3.2) (EP-TOX first performed, then this test is conducted **9 times**) (24-hour extraction period)

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

Analyze Immediately in field or upon arrival at the laboratory, glass bottle & top Salinity (6-month holding time if wax seal is used)

- 24-Hour Holding Time, plastic or glass containers, 4 C pH (RCRA)
- **48-Hour Holding Time, plastic or glass containers, 4 C, unpreserved** Nitrate, Nitrite
- **14-Day Holding Time, plastic or glass containers, 4** C Nitrate (SDWA chlorinated samples)
- **28-Day Holding Time, plastic or glass containers, 4 C, Sulfuric Acid to pH<2** Ammonia, Total Kjeldahl Nitrogen, Organic Nitrogen, Total Nitrate-Nitrite

ADDITIONAL REQUIREMENTS

- 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)