EXTRACTABLE ORGANICS 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 ea	ch 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)		
 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	
	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 page 17 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: F	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))		
COMM	ENTS:			
	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		
	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:

the entire measurement system, and the analyst must then perform the first option above

CHEMISTRY TEST METHOD EVALUATED:

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 18 for such Demonstration of Capability procedural requirements & acceptance criteria	

CHEN	MISTRY 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 19 for acceptance criteria
 5.1.1	Does the laboratory fulfill additional requirements specified in the mandated test method or regulation Note: See page 20 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

Does the laboratory have a written procedure to **monitor the application of marginal exceedance allowances** to LCS control limits to **ensure random behavior**

D.1.1.2(e)

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

CHEMISTRY TEST METHOD ASSESSED:

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

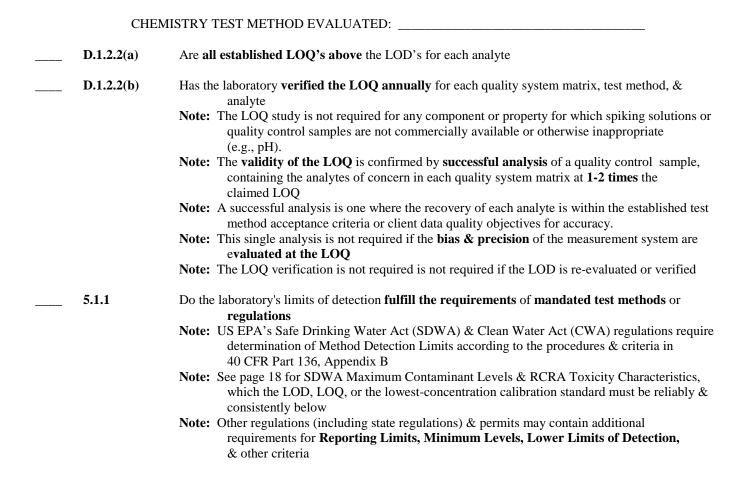
If a mass spectrometer is used, has the laboratory documented acceptance criteria for mass

spectral tuning

D.1.5(c)

CHEMISTRY TEST METHOD EVALUATED: _____

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). 		



COMMENTS: List analytes for which the above requirements for measurement sensitivity have not been fulfilled

EXTRACTABLE ORGANICS 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 506 (liq.-liq., GC-PID); 606, 612, (GC-ECD); 609 (GC-ECD & FID); 611 (GC-ELCD); EPA 3510, 3520, 3540, 3541 with 8061, 8091, 8111, 8121 (GC-ECD)

Methylene Chloride extraction solvent

Sodium Sulfate drying reagent

n-Hexane exchange solvent

Surrogate Standards (EPA 8000's)

Soxhlet extraction thimble (EPA 3540, 3541)

EPA 506 (liq.-sol., GC-PID); EPA 3535 with EPA 8061 (GC-ECD)

Reverse-phase C-18 solid-phase disks or cartridges

Ethyl Acetate, Methylene Chloride, Methanol solid-phase conditioning reagents

1:1 Methylene Chloride/Ethyl Acetate eluting solvent

Acetonitrile eluting solvent (EPA 8061)

Sodium Sulfate drying reagent

Surrogate standards (required for **EPA 8000's**)

EPA 551.1 (GC-ECD)

MTBE or n-Pentane extraction solvent

Surrogate Standard

EPA 556, 556.1 (GC-ECD)

KHP to adjust sample to pH 4

PFBHA (Pentafluorohydroxylamine) to derivatize analyte aldehydes to oximes (35 +/- 2 C for 2 hours)

Hexane extraction solvent

Sulfuric Acid to acid-wash extract

EPA 604, 8041; SM6420B (GC-FID or ECD)

EPA 3510, 3520, 3540, 3541 with EPA 8041 (GC-FID or ECD)

Methylene Chloride extraction

Sodium Sulfate drying agent

Isopropanol exchange solvent (Hexane prior to clean-up)

Pentafluorobenzyl Bromide derivatizing agent (if GC-ECD is used)

Diazald, to generate diazomethane derivatizing agent (optional)

Silica gel clean-up (if GC-ECD is used)

n-Hexane exchange solvent (if GC-ECD is used)

EPA 607 (GC-NPD); EPA 3510, 3520, 3540, 3541 with EPA 8070 (GC-NPD)

Methylene Chloride extraction solvent

Sodium Sulfate drying agent

Hydrochloric Acid for acid wash (EPA 607 only)

Methanol exchange solvent

Surrogate standards (EPA 8000's)

EPA 610, SM6440B (GC-FID or HPLC-UV & Fluorescence); EPA 3510, 3520, 3540, 3541 with EPA 8015, 8100 (GC-FID)

Methylene Chloride extraction solvent

Sodium Sulfate drying reagent

Cyclohexane exchange solvent prior to clean-up (GC-FID only)

Surrogate standards (EPA 8100, 8310)

EPA 8410, 8430 (GC-FTIR)

Methylene Chloride extraction solvent

Sulfuric Acid & Sodium Hydroxide to adjust aqueous-phase pH

Sodium Sulfate drying reagent

Internal Standards

Surrogate Compounds

Soxhlet extraction thimble (if solids are extracted)

EPA 7580 (GC-FPD or GC-NPD)

Diethyl Ether or Isooctane extraction solvent

White Phosphorus std. (P4)

EPA 8131 (GC-NPD)

1:1 Methylene Chloride/Acetone (solid samples) extraction solvent

Methylene Chloride (aqueous samples) extraction solvent

Sodium Sulfate drying agent

Toluene exchange solvent

Surrogate standards

EPA 3545 with EPA 8015, 8041, 8061, 8070, 8100, 8111, 8131, 8410, 8430

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 8015, 8041, 8061, 8070, 8100, 8111, 8121, 8410, 8430

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 3560 with EPA 8015 or 8440

Carbon Dioxide supercritical extraction fluid

Tetrachloroethylene collection solvent

EPA 3561 with EPA 8100

Carbon Dioxide supercritical extraction fluid, with Methanol, Water, & Methylene Chloride as modifiers

Reconstitution Solvents: 1:1 Acetonitrile/THF or 3:1 Methylene Chloride/Isooctane

EPA 3580 with EPA 8041, 8061, 8100, 8121, 8410

Waste Dilution Solvents: Methylene Chloride or Hexane

EPA 606, 607, 613, 1613, 8280, 8290; EPA 3610, 3611 prior to EPA 8061, 8070, 8100, 8270, 8310

Alumina Clean-up Sorbent, conditioned with Hexane

20% Ethyl Ether in Hexane, to elute Phthalate Esters from neutral alumina

30% then 50% Ethyl Ether in Pentane, to elute Nitrosamines from basic alumina

20% Methylene Chloride in Hexane, to elute Di-benzo-p-dioxins & Dibenzofurans from basic alumina

50% Methylene Chloride in Hexane, to elute Di-benzo-p-dioxins & Dibenzo-furans from acidic alumina

Hexane eluting solvent for Base-Neutral Aliphatics in petroleum waste

Methylene Chloride eluting solvent for Base-Neutral Aromatics in petroleum waste

Methanol eluting solvent for Base-Neutral Polars in petroleum waste

EPA 604, 610, 613, 1613, 1668, 8280, 8290; EPA 3630 prior to EPA 8041, 8082, 8100, 8310

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 606, 607, 608, 609, 611, 612, 1613, 1668

EPA 3620 prior to EPA 8061, 8070, 8081, 8082, 8091, 8111, 8121, 8131, 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 3650 prior to EPA 8041

Sodium Hydroxide, to remove water-soluble Organic Acids & Phenols form extract into aqueous phase Sulfuric Acid, to remove water-soluble Amines & Anilines from Dioxin extracts into aqueous phase Sulfuric Acid, to facilitate re-extraction of Organic Acids & Phenols into organic phase

EPA 3640 prior to EPA 8041, 8061, 8070, 8091, 8100, 8111, 8121, 8131

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

Diesel Range Organics

CH2Cl2 extraction solvent (MA-EPH, ME 4.1.25, AK-102, MS-DRO, MT-DRO, OK-DRO, TN-EPH, NWTPH-Dx)

n-Hexane exchange solvent (MA-EPH)

choice of Methylene Chloride or Freon-113 extraction solvent (OA-2)

choice of CH2Cl2, Hexane, or CS2 extraction solvent (WI-DRO)

Freon extraction solvent (**RI-DRO**)

Silica Gel fractionation or cleanup sorbent (MA-EPH, RI-DRO)

Diesel Fuel std. (EPA 8015, CA-LUFT, AK-102, MS-DRO)

Choice of Diesel Fuel or Fuel Oil #2 as std. (**RI-DRO**)

Even-numbered n-Alkane stds. C10 to C28 (WI-DRO, OK-DRO, MT-DRO, ME 4.1,25; AK-102 as RT std.)

#2 Diesel Fuel & 10W30 Motor Oil std. (TN-EPH)

choice of Kerosene, Diesel, Fuel Oil, Motor Oil, Transformer Oil, etc. stds. (OA-2, NWTPH-Dx, CT-ETPH)

Even-numbered n-Alkane stds. C10 to C36, 16 PAH's, n-C9, 2-Methylnaphthalene std. (MA-EPH)

n-Alkane stds. C9 to C36 for calibration and GC system performance (CT-ETPH)

o-Terphenyl Surrogate (MA-EPH, MS-DRO, MT-DRO, TN-EPH)

a-Androstane Surrogate (MS-DRO)

1-Chloro-octadecane Surrogate (MA-EPH)

Total Petroleum Hydrocarbons

Methylene Chloride extraction solvent (FL-PRO, NWTPH-HCID, AK-103)

Pentane extraction solvent for waters, 1:1 Pentane-Methanol for soils (TX1005)

Choice of Methylene Chloride or Hexane extraction solvent (8015AZ)

Silica Gel cleanup sorbent (**FL-PRO**)

Even-numbered n-Alkane std. C8 to C40 (FL-PRO)

Gasoline-Diesel-Motor Oil std. (NWTPH-HCID)

Gasoline-Diesel std. (TX1005)

Diesel-10W30 Oil std. (8015AZ)

Motor Oil std. (AK-103)

o-Terphenyl Surrogate (FL-PRO, 8015AZ)

n-C39 Surrogate (FL-PRO)

Bromofluorobenzene & n-C25 Surrogates (NWTPH-HCID)

n-C6, n-C10, n-C28 retention time stds. (**TX1005**)

n-C10, n-C22, n-C32 retention time stds. (8015AZ)

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

Chlorinated Hydrocarbons, Organochlorine Pesticides (RCRA), PCB's, Phthalate Esters

7 Days to Extract Sample, 40 Days to Analyze Extract; glass containers with

Teflon-lined cap; 4 C; 0.008% Sodium Thiosulfate

Phenols, Haloethers, Benzidines (RCRA); Polynuclear Aromatic Hydrocarbons (CWA)

7 Days to Extract Sample, 40 Days to Analyze Extract; glass containers with

Teflon-lined cap; 4 C; 0.008% Sodium Thiosulfate; store in the dark

Nitrosamines, Nitroaromatics & Cyclic Ketones,

Polynuclear Aromatic Hydrocarbons (SDWA & RCRA)

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

SDWA Chlorinated Solvents & Disinfection By-Products

28 Days to Extract Sample; 48 Hours to Analyze Extract; 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 506, 10.2, calibration factor (if used) < 20% RSD

EPA 604, 606, 607, 609, 610, 611, 612, 7.2 & 7.3, calibration or response factors (if used) < 10% RSD

OA-2, ME 4.1.25, calibration factor (if used) < 20% RSD

OK-DRO, correlation coefficient > 0.990

AK-102, AK-103, calibration factor (if used) < 25% RSD

5 standards + blank

EPA 7580, 7.2, calibration factor (if used) < 15% RSD

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 8015** (GC Gasoline Range & Diesel Range Organics)

Applies to EPA 8041, 8061, 8070, 8091, 8100, 8111, 8121, 8131 (GC Extractable Organics)

Applies to EPA 8410, 8430 (GC-FTIR Organics)

Requires **client notification of analytes** quantitated from CF or RF when mtd. criteria not met (and mean RSD < 20%)

EPA 551.1, 10.1-10.3, calibration factor or response factor (if used) < 10% RSD

FL-PRO, TX1005, MA-EPH, MT-DRO, TN-EPH, calibration factor (if used) < 25% RSD

8015AZ, MS-DRO, calibration factor (if used) < 20% RSD

WI-DRO, NWTPH-Dx, correlation coefficient > 0.990

CT-ETPH, calibration factor (if used) < 30% RSD

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 70-130%

EPA 556, 556.1, 10.3, 50-150% allowed for low-level stds.; CCV also required every 10 samples & end of run **8015AZ, CT-ETPH**

Recovery 75-125%

EPA 551.1, 10.4, plus recoveries within 80-120% for 90% of the analytes,

also every 10 samples & end of run

FL-PRO, MA-EPH, AK-102, AK-103, MT-DRO, TN-EPH

Recovery 80-120%

EPA 506, 10.2.3

SM6020B, 1b (applies to SM Organics methods)

40 CFR 141.40, App. A, 3, mid-range std. **plus** 60-140% recovery for std. at or below Minimum Reporting Level (applies to SDWA Unregulated Contaminants)

OA-2, ME 4.1.25, WI-DRO

8015AZ, for second-source std.

Recovery 85-115%

EPA 604, 606, 607, 609, 610, 611, 612, 7.4

EPA 7580, 8.4, and after every 10 samples

EPA 8000, 7.7

Recovery 90-110%

CA-LUFT

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

Mean Accuracy 30-130%; Precision RSD<30% EPA 7580, 8.5.5

Mean Accuracy 40-140%; Precision RSD < 25%

MA-EPH

Mean Accuracy 60-140%; Precision RSD < 30%

OK-DRO

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

EPA 506, 9.3, MDL study also required

Mean Accuracy 75-125%; Precision RSD < 20% RSD

TX1005

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

EPA 556, 556.1, 9.2, MDL study also required & at least 2 days required (i.e., not all aliquots extracted same day)

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

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

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

EPA 604, 606, 607, 609, 610, 611, 612, 8.2

EPA 8000, 8.6, applicable to all EPA 8000-series methods

FL-PRO

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

Each Regulated VOC (MDL requirement)	0.5	ug/L
Total Trihalomethanes	80	ug/L
Bis(2-ethylhexyl) Adipate	400	ug/L
Bis(2-ethylhexyl) Phthalate	6.0	ug/L

RCRA TOXICITY CHARACTERISTICS

o-Cresol	200.0	mg/L
m-Cresol	200.0	mg/L
p-Cresol	200.0	mg/L
Total Cresols	200.0	mg/L
2,4-Dinitrotoluene	0.13	mg/L
Hexachlorobenzene	0.13	mg/L
Hexachlorobutadiene	0.5	mg/L
Hexachloroethane	3.0	mg/L
Nitrobenzene	2.0	mg/L
Pentachlorophenol	100.0	mg/L
Pyridine	5.0	mg/L
2,4,5-Trichlorophenol	400.0	mg/L
2,4,6-Trichlorophenol	2.0	mg/L

OUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample or Matrix Spike Recoveries within the Test Method QC Acceptance Criteria for each Analyte EPA 604, 606, 607, 609, 610, 611, 612, 8.4

EPA 8000, 8.8, applicable to EPA 8041, 8061, 8070, 8091, 8100, 8111, 8121, 8131

FL-PRO, Duplicate Precision & Surrogate Recovery acceptance criteria also listed in Tables

External QC Check Sample Recoveries within 60-140%

EPA 556, 10.2.9, analyzed after initial instrument calibration **ME 4.1.25**, MSD Precision < 20% RPD

External QC Check Sample Recoveries within 70-130%

EPA 556.1, 9.9, analyzed quarterly

EPA 556.1, 10.2.9, analyzed after initial instrument calibration

Control Standard Recoveries 75-125%

AK-102, AK-103, also at end of run, LCSD Precision < 20% RPD

Control Standard Recoveries 50-100%

TN-EPH, duplicate precisions < 20% RPD

Matrix Spike Recoveries within 40-140%

MA-EPH

Matrix Spike Recoveries within 65-135%

EPA 506, 9.6, analyzed every 10 samples or batch

Matrix Spike Recoveries within 70-130%

8015AZ

Matrix Spike Recoveries within 75-125%

EPA 551.1, 9.6, plus recoveries within 80-120% for 90% of the target analytes **TX1005**, MSD Precision < 20% RPD

Matrix Spike Recoveries within 80-120%

WI-DRO (MSD Precisions < 20% RPD), OK-DRO (for waters, 60-140% for soils)

Surrogate Recoveries within 50-150%

NWTPH-HCID, TN-EPH

Surrogate Recoveries within 70-130%

EPA 556, 556.1, 9.6 **8015AZ**

Surrogate Recoveries 80-120%

EPA 551.1, 9.8

Internal Standards Responses

EPA 551.1, 9.9, 80-120% from avg. of last 5 Calib. verifications

Method Detection Limit Acceptance Criteria

8015AZ: 30 ppm for C10-C22, 100 ppm for C22-C32, 130 ppm for C10-C32

CA-LUFT: 0.5 ppm waters, 10 ppm soils **WI-DRO**: 0.1 ppm waters, 10 ppm soils

ADDITIONAL REQUIREMENTS

Matrix Spikes analyzed every 10 samples

EPA 506, 551.1, 9.6

EPA 604, 606, 607, 609, 610, 611, 612, 8.3

SM6020B, 3c, or Monthly (whichever is more frequent) (applies to all SM Organics methods)

Matrix Spike & Matrix Spike Duplicate each batch of 20 samples or fewer

40 CFR 141.40, App. A, 6, must also **alternate between mid- & low-level** concentrations for spikes (applies to SDWA Unregulated Contaminants)

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

Matrix Spike every 20 samples

EPA 556, 556.1, 9.7, or each batch whichever more frequent

Matrix Spike & Matrix Spike Duplicate

EPA 7580, 8.6, monthly

Quality Control Check Samples analyzed every 10 samples

EPA 604, 606, 607, 609, 610, 611, 612, 8.1.5, frequency may be reduced if Matrix Spike recoveries meet all specified QC criteria

Field Duplicates analyzed

EPA 551.1, 9.7, ALL samples are collected in duplicate; 10% are analyzed

GC Retention Time Windows established for each analyte

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

GC Retention Time Definitions for Total Petroleum Hydrocarbons (forced baseline integrations)

FL-PRO: n-C8 to n-C40

NWTPH-HCID: GRO as Toluene to n-C12, DRO as n-C12 to n-C24, then Lube Oil as anything > n-C24

TX1005: TPH as n-C6 to n-C35, GRO as n-C6 to n-C10, DRO as n-C10 to n-C28 **8015AZ**: GRO as n-C6 to n-C10, DRO as n-C10 to n-C22, ORO as n-C22 to n-C32

AK-103, C25 to C45 as Residual Range Organics

GC Retention Time Definitions for Diesel Range Organics (forced baseline integrations)

EPA 8015, **ME 4.1.25**, **MT-DRO**, **OK-DRO**, **WI-DRO**: n-C10 to n-C28

MA-EPH: C9 to C18 Aliphatics, C19 to C36 Aliphatics, C11 to C22 Aromatics

AK-102, MS-DRO: n-C10 to n-C25

NWTPH-Dx: n-C12 to n-C24

CT-ETPH: C9 to C36 **TN-EPH**: C12 to C40

GC System Performance

CT-ETPH, all n-Alkanes C9 to C36 have RF's within 20% of each other, recalibrate GC if not