NATIONAL ENVIRONMENTAL LABORATORY ACCREDITATION CONFERENCE (NELAC)

ON-SITE LABORATORY ASSESSMENT

CHEMISTRY CHECKLIST (103 PAGES TOTAL)

LABORATORY: _____

Physical Address: _____

_____

Mailing Address: _____
(if different from above)

_____

Telephone Number: _____ Facsimile Number: _____

E-mail address: _____

INSPECTED BY: (Name) (Affiliation)

_____ _____

_____ _____

_____ _____

INSPECTION DATES: _____

LABORATORY TECHNICAL DIRECTORS AND MANAGEMENT:
(Name) (Title)

_____ _____

_____ _____

_____ _____

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_____ _____

GENERAL INSTRUCTIONS: Before each item is a blank line and a NELAC Standard citation in **Bold Numerals**.

Place a check mark (__----) in the blank if the laboratory meets the NELAC Standard referenced.

Place an X-mark (X) in the blank if the Standard is not met and the laboratory must devise an acceptable Plan of Correction and estimated completion date. **The NELAC Standard reference must be cited in in the on-site assessment report.**

Mark **"N/A"** in the blank if the NELAC Standard is not applicable to this laboratory, either because of the nature of its business mission, because of the analytical tests it performs, or because of the situation never ever happening.

See individual **Technology Checklists** for specific requirements on Reagents & Standards, Test Method Standard Operating Procedures, Instrument Calibrations, Demonstrations of Capability, Quality Control, Detection Limits, and Selectivity, as applicable

Notes:

If the laboratory appears to meet a particular NELAC Standard but does not have the documentation to back up its claim, use the following:

_____ **5.0** Does the laboratory have **all items** identified in NELAC Chapter 5 Quality Systems **available** for on-site inspection or data audit

Comment: (list all applicable Standards where the accompanying data was not available for review)

CHEMISTRY LABORATORY TOUR

____ **5.5.3.2** Does the laboratory meet & document adherence to **laboratory facility requirements** specified by a test method or regulation

EPA 508A – fume hood for handling SbCl5 (PCB perchlorination)
EPA 515.1, 515.2, 615, 8151; SM6251B – fume hood with safety shield for diazomethane generation (Herbicides)
EPA 613, 1613, 8280, 8290 – controlled access work areas, adequate ventilation, analyst training (Dioxins)
EPA 9010, 9012 – fume hood & amber light with cyanogen chloride generation (Amenable Cyanide treatment)
EPA 9030, 9031 – fume hood (Sulfide distillations)
SM4500CN- B – fume hood (all sample manipulations that may contain toxic Cyanide)

____ **5.5.5.2.1(d)** Does the laboratory's **support equipment** checks meet the **following needs of the analysis or or application** for which the equipment is being used

EPA 245.1, 7470, 7471; SM3112B – 95 degrees Celsius to avoid Hg loss in boiling solution
EPA 245.2 – 105 degrees Celsius
EPA 351.x, SM4500Norg B & C – 365-380 degrees Celsius, Kjeldahl Nitrogen digestion until white SO3 fumes are expelled & for 30 minutes thereafter
SM4500F- B – discontinue HF distillation above 180 degrees Celsius to prevent H2SO4 carryover
SM5210B – 19-21 degrees Celsius during the 5-day BOD or CBOD incubations
Residues – 180 C (TDS), 103-105 C (TS & TSS), 550 C (Volatile Residue)
EPA 1110 – 55 degrees Celsius, stainless steel corrosivity test conducted for 24 hours
EPA 1311 – 21-25 degrees Celsius, extraction for 16-20 hours, tumbled end-over-end at 28-32 rpm
EPA 508A – 200-210 degrees Celsius for PCB perchlorination reaction
EPA 552.3 – at least 50 C for MTBE, 60 C for TAME, derivatization of HAA's for at least 2 hours
Herbicides Derivatization – 0-4 degrees Celsius if diazomethane solution is prepared from Diazald
Freon Solvent Evaporation (EPA 413.1, SM5520B) – 60-70 degrees Celsius
Methylene Chloride & Ethers (e.g. MTBE) Solvent Evaporation – 60-70 degrees Celsius
n-Hexane Solvent Evaporation – 85-90 degrees Celsius

____ **5.5.6.4(d)** Do **all** containers of prepared standards & reference materials bear a **unique identifier, expiration date, & link** to its specific **preparation record**

____ **5.5.6.4(e)** Are procedures in place to ensure that prepared reagents **meet the requirements of the test method**

Note: See the technology checklists for specific reagents required for particular test methods
Note: Reagents of appropriate quality must be selected and used. In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than specified in the test method shall not be used. Checks of the container label to verify that the purity of the reagents complies with the test method must be documented.

____ **5.5.6.4(f)** Do containers of **prepared reagents** bear a **preparation date**

____ **5.5.6.4(f)** Is the expiration date for each prepared reagent **defined on the container or documented elsewhere** as indicated in the laboratory's quality manual or SOP

____ **5.5.8.3.1(a)(2)** Has the laboratory **checked samples** for **proper preservation** (e.g. pH, absence of free chlorine) prior to or during sample preparation or analysis

Note: See the Technology Checklists for holding times, sample containers allowed, & preservation required for each analyte

____ **D.1.3** Has the laboratory **documented procedures for data reduction** (e.g. use of linear regression)

____ **D.1.4(a)** Does the laboratory maintain records of **calibration certificates,** traceability to **national standards** of measurement, **measurement results** with associated uncertainties, or **correlations of results** for each standard source needed
Note: These Standards are from NELAC 5.5.6.2.2.2

____ **D.1.4(b)(1)** Does the laboratory **use analytical reagent grade** materials when the purity of reagents is not specified in the test method

____ **D.1.4(b)(1)** Does the laboratory **not use reagents of lesser purity** than those specified by the test method

____ **D.1.4(b)(1)** Does the laboratory **document** any checks it makes to **verify** that the **purity of reagents** meets the test method requirements

____ **D.1.4(b)(2)** Does the laboratory monitor & document the **quality** of its reagent **water sources**

____ **D.1.4(b)(2)** Does the quality of the water resources **meet method-specified requirements**

____ **D.1.4(b)(3)** Does the laboratory **verify titrant concentrations** according to written laboratory procedures

____ **D.1.6(a)** Does the laboratory's **test instruments** consistently **operate within the specifications** required of the applications for which they are used

____ **D.1.6(b)** Does the laboratory **clean glassware** to **meet the sensitivity** of the test method

____ **D.1.6(b)** Does the laboratory **document** in its records & standard operating procedures any **cleaning & storage procedures not specified** by the test method

COMMENTS:

US EPA REGULATIONS MANDATING PARTICULAR TEST METHODS

40 CFR Parts 122.21(g)(7), 122.21(h)(4), 403.7(b)(2)(v), 403.12(b)(5)(vi), & 403.12(g)(4) mandate the use of test methods approved in **40 CFR Part 136**

40 CFR Parts 122.41(j)(4) & 501.15(b)(10)(iv) mandate the use of test methods specified in **40 CFR Parts 136 or 503**

40 CFR Parts 141.23(k), 141.24(e), 141.30(e), 141.40(g), 141.40(n)(11), 141.74(a), 141.89(a), 141.131, 141.142(b), & 141.143(b) mandate the use of test methods contained therein for compliance with the **Safe Drinking Water Act**

40 CFR Part 141.40(n)(12) mandates the use of test methods in **40 CFR Part 143.4(b) for Sulfate**

40 CFR Parts 258.28(c)(1), 264.190(a), 264.314(c), 265.190(a), 265.314(d), 265.1081, & 268.32(i) mandate the use of **EPA 9095**

40 CFR Part 260.22(d)(1)(i) mandates the use of **SW-846 methods** to determine that **40 CFR Part 261 App. VII constituents** are absent

40 CFR Part 261.21 mandates the use of test methods that are directly referenced in **EPA 1010 & 1020 (Ignitability)**

40 CFR Part 261.22 mandates the use of **EPA 9040** or the method that is directly referenced in **EPA 1110 (Corrosivity)**

40 CFR Parts 261.24, 268.7(b)(1), & 268.7(c)(2) mandate the use of **EPA 1311 (TCLP)**

40 CFR Part 261 App. III cites **SW-846 Chapter 2** as specifying "appropriate analytical procedures" (as used in **EPA 1311, 7.2.14 &7.2.15)** to determine toxic constituents

40 CFR Part 266.104(e)(1) mandates the use of **EPA 0023A**

40 CFR Part 266.106(a) mandates the use of **SW-846 methods** to determine **Sb, Ba, Pb, Hg, Tl, Ag, As, Cd, Be, Cr, Ni, Se**

40 CFR Part 266.106(g) mandates the use of **EPA 0060 & 0061**

40 CFR Part 266.107(f) mandates the use of **EPA 0050 or 0051**

40 CFR Parts 266.112(b)(2)(i), 270.19(c)(1), 270.62(b)(2)(i), & 270.66(c)(2) mandate the use of **SW-846 methods** to determine **40 CFR Part 261 App. VIII toxic & hazardous constituents**

40 CFR Part 268.40(b) mandates the use of **EPA 1310 or 1311**

40 CFR Part 430.01(i) mandates the use of **EPA 1613, 1650, & 1653** to determine compliance with **Minimum Level requirements** in the **Pulp & Paper Industry**

40 CFR Part 455.50 mandates the use of test methods in **Table 7 to 40 CFR Part 455** for **Pesticide Active Ingredients**

40 CFR Part 503.8(b)(4) mandates the use of **SW-846 methods** to determine **Inorganic Pollutants in sludge**

Note: Make enough copies of **Pages x-xx** to **assess each test method** in use at the laboratory, one method at a time
In addition, copy **Page xx** to assess each **chromatography** method

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)** **Data Analysis & 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 xx-xx 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 xx for such Demonstration of Capability procedural requirements & acceptance criteria

CHEMISTRY TEST METHOD ASSESSED: _____

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

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

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

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

____ **5.1.1** Does the laboratory's **acceptance criteria** for blanks, laboratory control samples, duplicates, & matrix spikes **fulfill the requirements** in **mandated test methods**
Note: See pages xx-xx for acceptance criteria

____ **5.1.1** Does the laboratory fulfill **additional requirements** specified in the **mandated test method** or **regulation**
Note: See pages xx-xx 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 xx 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

FLAME EMISSION, ATOMIC ABSORPTION, & ATOMIC FLUORESCENCE SPECTROMETRY

Flame Photometric Methods – SM3500K D (<=19th ed.), SM3500K B (20th ed.);
SM3500Na D (<=19th ed.), SM3500Na B (20th ed.)
Flame AA Methods – EPA 2xx.1 & 7xx0 (generally); SM3111B,C,D; USGS I-3xxx-85 (generally);
ASTM Dxxxx-yyA or B (generally); AOAC 973.53, 973.54, 974.27; ANSI Photo. Effluents
Furnace AA Methods – EPA 2xx.2 & 7xx1 (generally), 200.9; SM3113B; ASTM Dxxxx-yyC or D (generally)
Hydride AA Methods – EPA 206.3, 270.3, 7061, 7741; SM3114B; ASTM D2972-97B, D3859-98A;
USGS I-3062-85, I-3667-85
Cold Vapor AA Methods – EPA 245.1, 245.2, 245.5, 245.6, 7470, 7471; SM3112B; ASTM D3223-91;
USGS I-3462-85; AOAC 977.22
Au Amalgamation Cold Vapor AA Methods – EPA 1631C, 7473
Atomic Fluorescence Methods – EPA 245.7, 1631E, 7474

REQUIRED REAGANTS & STANDARDS

EPA 200-series, 7000-series; SM3000-series

Stock Standards for each Metal
Hollow cathode or electrodeless discharge lamps for each Metal (Atomic Absorption Spectrometry)
Matrix modifiers for Graphite Furnace AA:
- Pd/Ni(NO3)2 – As, Cd, Pb, Se
- Pd/Mg(NO3)2 – Cd, Pb, Tl
- NH4H2PO4 or H3PO4 -
- H2 in Ar purge gas – Sb & Tl

Matrix Modifiers for Flame AA:
- LaCl3 – Ca (releasing agent to prevent pyrophosphate formation in flame)
- KCl – Ba & Na (K ionization more readily vs. Ba & Na)

Digestion reagents (unless sample is aqueous with turbidity < 1 NTU (SDWA); sample is filtered (to analyze for Dissolved Metals) and no COD or TOC (CWA); and not analyzed by cold-vapor AA, hydride AA, colorimetric, or chelation-extraction techniques)
- Nitric Acid (plus hydrochloric acid depending on the aqueous sample & metal analyzed) (plus HCl, H2O2, or HF depending on the metal analyzed & the solid/sludge/sediment sample)
- Permanganate & Sulfuric Acid to digest oils
- Xylene, MIBK, or Kerosene to dissolve oils, greases, or waxes
- **Note:** Microwave digestion not approved for SDWA; in CWA only approved for FL-AA & ICP analyses of Al, As, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Se, V, Zn
- Permanganate, Persulfate, HNO3, & H2SO4 required for aqueous Mercury digestions (waterbath for 2 hr at 95 C) (then hydroxylamine to decolorize & SnCl2 to generate Hg vapor)
- Bromate/Bromide & HCl required to digest for ultratrace concentrations of Mercury
- Sodium Carbonate & Sodium Hydroxide to digest for Chromium(VI)

Chelation-Extraction reagents: Ammonium Pyrrolidine Dithiocarbamate & MIBK
Coprecipitating reagents for Cr(VI): Ammonium Sulfate & Lead Nitrate
Reducing agents for Hydride Generation & Cold-vapor AA methods:
- Tin(II) Chloride to generate Hg vapor, or to reduce As(V) & Se(VI) to As(III) & Se(IV)
- Potassium Iodide or Urea (alternates to reduce As(V) & Se(VI) to As(III) & Se(IV))
- Zinc Metal/Hydrochloric Acid or Sodium Borohydride, to generate volatile metal hydrides

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

24-Hour Holding Time, plastic or glass containers, 4 C
Chromium(VI)

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

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 at least 16 hours prior to analysis)

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

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank
SM1020B, 5, applies to all mandated SM methods
EPA Metals, 10.2.1 refers to all EPA 200-series FL-AA & FUR-AA methods used for SDWA
EPA 200.9, 11.4.4
EPA 245.7, 9.1
EPA 7000, 8.2, applies to all EPA 7000-series FL-AA & FUR-AA methods, **daily**
SM3113B, 4c, **daily**
D3919, 11.2 refers to all ASTM FUR-AA methods
D4691, 11.2 refers to all ASTM FL-AA methods

4 standards + blank
EPA Metals, 8.2 refers to all EPA 200-series FL-AA & FUR-AA methods

5 standards + blank
EPA 245.1, 11.2.2 (**standards must contain same digestion reagents or be digested** along with samples)
EPA 245.5, 9.1 (**standards digested** along with samples)
EPA 1631E, 10.2 & 10.3, plus **3 system blanks**, CF<15%RSD, lowest std. 75-125% recovery, blanks <0.5 ng/L

6 standards + blank
I-3xxx-85, 6, **daily,** applies to all USGS FL-AA Metals mtds.

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 80-120%
EPA 1631E, 9.5.3 (every 12 hr & end of run), all procedural steps included, Table 2 says 77-123% recovery at 5 ng/L
EPA 7000, 8.3, for continuing calibration verifications done **after every 10 samples**

Recovery 90-110%
EPA 200.7 200.8, 200.9, 245.1, 9.3.4 after **every 10 samples & end of run**, calibration blank analysis also required each time
SM3020B (AA Metals)
EPA 7000, 8.2, for second-source initial calibration verification

Recovery 95-105%
EPA 200.9 & 245.1, 9.3.4 for midpoint std. after initial calib.

Inclusion of both Standard & Calibration Blank Analysis
EPA 245.7, 10.5 (including end of the run)

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 200.9, 9.2.2, 6 stds. needed
EPA 245.1, 9.2.2, 3 stds. needed
EPA 245.5, 245,6, 10.2.1

Table Criteria of 79-121%, 21% RSD

EPA 1631E, 9.2.2, also requires **MDL <0.2 ng/L**

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample Recoveries within 85-115%

EPA 200.9, 245.1, 9.3.2-9.3.3 (Metals LFB)

2 concentrations required for QC Check Sample Analyses

ASTM D4691, 11.4.1 refers to all ASTM FL-AA methods

External QC Check Sample Recoveries within 80-120%

EPA 7000, 8.3, every 10 samples

External QC Check Sample Recoveries within 90-110%

EPA 7000, 8.2, **daily** after each initial calibration

EPA QC Check Sample Recoveries within 90-110%

EPA 200.9, 245.1, 9.2.3 analyzed quarterly (Metals)

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

EPA 200.9, 245.1, 9.4.2-9.4.3, analyzed **every 10 samples** (Metals)

Matrix Spike & Matrix Spike Duplicate Recoveries within Table Criteria of 71-125%, <24% RPD

EPA 1631E, 9.3, MS/MSD required **every 10 samples**

Matrix Spike Recoveries within 85-115%

EPA 7000, 8.6.2, criterion to avoid quantitation of metals by the method of standard additions
SM3020 (Metals) (SDWA: In-furnace addition recovery needed to avoid quantitation of Metals by the method of standard additions)

Matrix Spike Recoveries within 90-110%

EPA Metals, 5.2.1 for post-digestion spikes, refers to all EPA 200-series FL-AA & FUR-AA methods

Serial Dilution Results within 10%

EPA 7000, 8.6 analyzed each sample batch

Background Absorbance < 1.0

EPA 200.9, 9.4.6

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

Antimony	6.0 ug/L
Arsenic	10.0 ug/L
Barium	2000 ug/L
Beryllium	4.0 ug/L
Cadmium	5.0 ug/L
Chromium	100 ug/L
Copper	1300 ug/L (1.0 ug/L if analyzing composite samples) (20 ug/L if analyzing composite samples by FL-AA)
Lead	15.0 ug/L (1.0 ug/L if analyzing composite samples)
Mercury	2.0 ug/L
Nickel	100 ug/L
Selenium	50.0 ug/L
Sodium	20000 ug/L (reporting level)
Thallium	2.0 ug/L

RCRA TOXICITY CHARACTERISTICS

Arsenic	5.0 mg/L
Barium	100.0 mg/L
Cadmium	1.0 mg/L
Chromium	5.0 mg/L
Lead	5.0 mg/L
Mercury	0.2 mg/L
Selenium	1.0 mg/L
Silver	5.0 mg/L

ADDITIONAL REQUIREMENTS

Matrix Spikes, Control Standards, & Duplicates at least 15% of workload for any parameter
USGS Bk. 5, Ch. A1, p.7, applies to all USGS Metals & General Chemistry mtds.

Method of Standard Additions required to quantitate Metal analytes
EPA 7000, 8.7, for all EP-TOX extracts, analyses for RCRA delisting petitions, & analysis of new matrices

Matrix Spikes analyzed every 10 samples
EPA 245.5, 245.6, 245.7, 10.4, or **batch**

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
EPA 245.7, 10.3
D4691, 14.3, applies to ASTM FL-AA Metals mtds.

Matrix Spike & Matrix Spike Duplicate each batch of 20 samples or fewer
EPA 7000, 8.4 (applies to all 7000-series FL-AA & FUR-AA Metals mtds.)
EPA 7473, 9.3, may use sample dup. in place of MSD

Spike Duplicate analyzed every 10 samples
EPA 7195, 7197, 8.6

All Samples analyzed in Duplicate
SM3113B, 4d, repeat replicate analyses until variation < 10%

Calibration Verification every 10 samples
D3919, 12.3, applies to ASTM FUR-AA Metals mtds.
D4691, 11.3, applies to ASTM FL-AA Metals mtds.

Calibration Verification every 15 samples
EPA 7195, 7197, 8.5, second-source std.

INDUCTIVELY-COUPLED PLASMA (ICP) & DIRECT-COUPLED PLASMA (DCP) SPECTROMETRY; ICP MASS SPECTROMETRY (ICP/MS)

ICP Methods – EPA 200.7, 6010; SM3120B
DCP Methods – ASTM D4190-94; ARL AES0029
ICP-MS Methods – EPA 200.8, 6020; AOAC 993.14; SM3125B

REQUIRED REAGANTS & STANDARDS

EPA 200-series, 7000-series; SM3000-series

Stock Standards for each Metal
Digestion reagents (unless sample is aqueous with turbidity < 1 NTU (SDWA); sample is filtered (to analyze for Dissolved Metals) and no COD or TOC (CWA); and not analyzed by cold-vapor AA, hydride AA, colorimetric, or chelation-extraction techniques)
Nitric Acid (plus hydrochloric acid depending on the aqueous sample & metal analyzed) (plus HCl, H2O2, or HF depending on the metal analyzed & the solid/sludge/sediment sample)
Permanganate & Sulfuric Acid to digest oils
Xylene, MIBK, or Kerosene to dissolve oils, greases, or waxes
Note: Microwave digestion not approved for SDWA; in CWA only approved for FL-AA & ICP analyses of Al, As, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sb, Se, V, Zn
Interference Check Standards for ICP-AES
Tuning solutions & Internal Standards for ICP-MS

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

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

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 at least 16 hours prior to analysis)

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

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank
SM1020B, 5, applies to all mandated SM methods

4 standards + blank
D4190, 11.1 (DCP)

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 90-110%

EPA 200.7 200.8, 9.3.4 after **every 10 samples & end of run**, calibration blank analysis also required each time

EPA 6010, 8.6.1, also **every 10 samples & end of run**, calibration blank analysis also required

EPA 6020, 8.8, also **every 10 samples & end of run**, calibration blank analysis also required

Recovery 95-105%

EPA 200.7, 9.3.4, calibration blank analysis also required

Note: For **40 CFR 136 App. C**, 12.1.1, above also applies after **every 10 samples & end of run**)

SM3120B, 4e, **every 10 samples**

Inclusion of both Standard & Calibration Blank Analysis

EPA 200.8, 10.4

EPA 6020, 7.6

ICP-MS Tune Solution Precision within 5%

EPA 200.8, 10.2.2 for 5 daily analyses

EPA 6020, 7.4 for 4 daily analyses

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 200.7, 9.2.2

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample Recoveries within 85-115%
EPA 200.7, 200.8, 9.3.2-9.3.3 (Metals LFB)

External QC Check Sample Recoveries within 90-110%
EPA 6020, 7.8 (Initial Calibration Verification)

EPA QC Check Sample Recoveries within 90-110%
EPA 200.7, 200.8, 9.2.3 analyzed quarterly (Metals) (recoveries 95-105% for **EPA 200.7**)

Outside Source QC Standard Recoveries within 95-105%
EPA 200.7 (from **40 CFR 136, App. C**), 12.1.3 analyzed weekly (CWA)

Interference Check Sample (ICS) Analysis
EPA 200.7, 9.3.5
EPA 6010, 8.6.2
EPA 200.7 (40 CFR 136 App. C), 12.1.2, analyzed at beginning & end of run, results within 1.5 std. dev. of mean

Matrix Spike (SMS or LFM) Recoveries within 70-130%
EPA 200.7, 200.8, 9.4.2-9.4.3, analyzed **every 10 samples** (Metals)

Matrix Spike Recoveries within 75-125%
EPA 6010, 8.4, analyzed every 20 samples or batch, historical limits also allowed

Duplicate Precisions within 20%
EPA 6010, 8.4, for matrix spike duplicate, analyzed every 20 samples or batch, historical limits also allowed
EPA 6020, 8.10, every 20 samples or batch, for analyte concentrations over 100 times the instrument detection limit

Serial Dilution Results within 10%
EPA 6020, 8.5 analyzed every 20 samples

Internal Standards Responses
EPA 200.8, 9.4.5 & 10.3, **60-125%** of values from the calibration blank
EPA 6020, 8.3, **30-120%** of values from the initial calibration standard in samples, **80-120%** in CCV/CCB's

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

Antimony 6.0 ug/L
Arsenic 10.0 ug/L
Barium 2000 ug/L
Beryllium 4.0 ug/L
Cadmium 5.0 ug/L
Chromium 100 ug/L
Copper 1300 ug/L (1.0 ug/L if analyzing composite samples) (20 ug/L if analyzing composite samples by FL-AA)
Lead 15.0 ug/L (1.0 ug/L if analyzing composite samples)
Mercury 2.0 ug/L
Nickel 100 ug/L
Selenium 50.0 ug/L
Sodium 20000 ug/L (reporting level)
Thallium 2.0 ug/L

RCRA TOXICITY CHARACTERISTICS

Arsenic 5.0 mg/L
Barium 100.0 mg/L
Cadmium 1.0 mg/L
Chromium 5.0 mg/L
Lead 5.0 mg/L
Mercury 0.2 mg/L
Selenium 1.0 mg/L
Silver 5.0 mg/L

ADDITIONAL REQUIREMENTS

Instrument Detection Limit evaluated quarterly
EPA 6020, 8.2

ICP Calibration Blank Results within 3 Std. Dev. of Background Mean
EPA 200.7, 9.3.4
EPA 200.7 (40 CFR 136 App. C), 12.1.1, 2 std. dev.
EPA 6010, 8.6.1, also < 3 times the instrument detection limit

ION CHROMATOGRAPHY (IC)

REQUIRED REAGANTS & STANDARDS

Br-, Cl-, F-, NO3-, NO2-, HPO4=, SO4= - EPA 300.0, 300.1, 9056, 9057; SM4110B; ASTM D4327-97; AOAC 993.30

HPLC with anion exchange column, suppressor, & conductivity detector
Sulfuric Acid or alternate column regenerating system
Sodium Bicarbonate & Sodium Carbonate mobile phase

Ca++, NH4+, Mg++, K+, Na+ - EPA 300.7

HPLC with cation exchange column & conductivity detector
Sodium, Barium, or Tetramethylammonium Hydroxides, or alternate column regenerating system
Hydrochloric Acid & m-Phenylenediamine mobile phase

Chromium(VI) – EPA 218.6, 7199; SM3500Cr E (19th ed.), SM3500Cr C (20th ed.); ASTM D5257-93; AOAC 993.23

HPLC with anion exchange column & UV detector (530 nm)
Ammonium Sulfate & Ammonia-water eluent & buffer to adjust sample pH to 9.0-9.5
1,5-Diphenylcarbazide post-column colorimetric reagent

Oxyhalides Disinfection By-Products (Br-, BrO3-, ClO2-, ClO3-) – EPA 317.0, 326.0 (also see EPA 300.1)

HPLC with anion exchange column, suppressor, conductivity detector, post-column derivatization, & UV-VIS detector (450 nm for **EPA 317.0**, 352 nm for **EPA 326.0**)
EPA 300.1: BrO3- & ClO2- must be analyzed separately from F-, Cl-, NO3-, NO2-, & SO4=
EPA 317.0, 326.0: Chlorite & Bromate must be analyzed separately (ClO2- overwhelms PCR/UV-VIS response)
HNO3, KBr, & o-Dianisidine post-column derivatizing agent for Bromate (**EPA 317.0**), prepared fresh daily
Potassium Iodide post-column derivatization agent, with Ammonium Molybdate catalyst, for Bromate (**EPA 326.0**)
Sodium Carbonate mobile phase
Dichloroacetic Acid surrogate
Ethylenediamine preservation solution, also chelates iron interference & binds free chlorine

Perchlorate – EPA 314.0

HPLC with anion exchange column & conductivity detector
Anion suppressant or alternate column regenerating system
Sodium Hydroxide mobile phase
Chloride, Sulfate, & Carbonate (sodium salts) for synthetic sample matrix solution

Bromate (IC / ICP-MS) – EPA 321.8

HPLC with anion exchange column & ICP/MS detector (m/z 79 & 81, but m/z 81 has Ar2H+ interference)
Anion suppressant or alternate column regenerating system
Pre-treatment cartridge, to remove trihaloacetic acid interferences
Ammonium Nitrate / Nitric Acid mobile phase
Bromate also used as MS tuning solution & instrument drift solution

Perchlorate (IC / MS) – EPA 331.0, 332.0

HPLC with anion exchange column, negative electrospray ionization interface, & MS detector (m/z 99 & 101 parent ions for the SIM mode, m/z 83 & 85 daughter ions for the MRM mode (**EPA 331.0**))
Anion suppressant (**EPA 332.0**)
Oxygen-18 labeled internal standard (m/z 107 & 109 for SIM mode, m/z 89 & 91 for MRM mode)
Methylamine solution (**EPA 331.0**) or Potassium Hydroxide (**EPA 332.0**) as mobile phase
Chloride, Sulfate, & Bicarbonate (or Carbonate (**EPA 332.0**)) (sodium salts) for synthetic sample matrix solution

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

Analyze Immediately in the field or upon arrival at the laboratory, plastic or glass containers
Orthophosphate (filtration step only)

24-Hour Holding Time, plastic or glass containers, 4 C
Chromium(VI)

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

14-Day Holding Time, plastic or glass containers, 4 C
Nitrate (SDWA chlorinated samples)

14 Days to Extract Sample, Opaque containers; 4 C
SDWA Chlorite

28-Day Holding Time, plastic or glass containers, 4 C
Bromide, Chloride, Sulfate
Perchlorate (must be <10 C during 1st 48 hr after collection)

28 Day Holding Time, Opaque containers, 4 C
SDWA Bromate

28-Day Holding Time, plastic container (only)
Fluoride

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

6-Month Holding Time, plastic or glass containers, Nitric Acid to pH<2
Metals (add HNO3 if sample unpreserved analysis)

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank
SM1020B, 5, applies to all mandated SM methods
EPA 300.0, 10.2
EPA 300.1, 314.0, 317.0, 10.2.2, **5 stds.** required if > 2 orders of magnitude in conc., CF (if used) < 15% RSD
EPA 7199, 7.2
EPA 9056, 7.1.2
D5257-93, 10.2

5 standards + blank
EPA 300.7, 9.6.1
EPA 326.0, 10.2.3-10.2.4
EPA 331.0, 10.3.1
EPA 332.0, 10.3.3, each concentration level > MRL must be within 80-120% of resultant regression curve value, 50-150% for concentration level < MRL

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 80-120%

EPA 331.0, 332.0, 10.4, for Mid-point Std., Std. also required at or below MRL with recovery 50-150%, also after **every 10 samples & end of run**

Recovery 85-115%

EPA 300.1, 10.5.1, 75-125% allowed for conc. 1-100x the MDL

EPA 314.0, 317.0, 326.0, 10.3.2, 75-125% allowed at MRL, also after **every 10 samples & end of run**, CCB also required for EPA 314.0 & 317.0

Recovery 90-110%

EPA 300.0, 9.3.4 after **every 10 samples & end of run**, calibration blank analysis also required each time

EPA 7199, 7.3, also **every 10 samples & end of run**

EPA 9056, 7.1.4 & 8.2, plus after **every 10 samples**, recalibrate if not within 5% of previous result

Recovery 95-105%

EPA 218.6, 9.3.4 for midpoint std. **every 10 samples**

Inclusion of both Standard & Calibration Blank Analysis

EPA 300.7, 10.5 (including end of the run)

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 – upper limit within 10% of extrapolated value

EPA 218.6, 10.2.3, 5 stds. needed with log-log plot slope within 0.98-1.02

EPA 300.0, 9.2.2, 3 stds. needed & verified at least **every 6 months**

EPA 321.8, 9.2.2 & 9.2.4

Method Detection Limit required for each analyte

EPA 300.1, 9.2.3

EPA 314.0, 317.0, 9.2.6

EPA 326.0, 9.2.4

Matrix Conductivity Threshold (MCT) evaluation

EPA 314.0, 9.2.8 & 9.3.2, at 25 ug/L ClO4- under increasing anion concentrations, MCT verified each batch

Mean Accuracy 80-120%; Precision RSD < 20%

EPA 331.0, 332.0, 9.2, from 7 replicates, required from both Fortified Blanks & Fortified Synthetic Sample Matrices, MRL verification also required

Mean Accuracy 85-115%; Precision RSD < 20%

EPA 317.0, 326.0, 9.2, from 7 replicates

Mean Accuracy 90-110%, Precision RSD < 10%

EPA 314.0, 9.2.3 & 9.2.4, from 7 replicates

EPA 321.8, 9.2.3, from 3 replicates (no precision criteria specified)

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample Recoveries within 80-120%

EPA 331.0, 332.0, 9.3.3, for concentrations > MRL, 50-150% for concentrations < MRL

QC Check Sample Recoveries within 85-115%

EPA 300.1, 317.0, 9.3.2, 75-125% allowed for concentrations 1-100 times the MDL
EPA 314.0, 321.8, 9.3.3
EPA 326.0, 9.6, 75-125% allowed for concentrations 1-2 times the MRL

QC Check Sample Recoveries within 90-110%

EPA 218.6, 10.3.3
EPA 300.0, 9.3.2-9.3.3 (Inorganics)
EPA 9057, 8.1.1, analyzed every **10 samples & end of run**

External QC Check Sample Recoveries within 80-120%

EPA 317.0, 9.2.5, analyzed quarterly

External QC Check Sample Recoveries within 85-115%

EPA 326.0, 9.11, analyzed quarterly and each time new calibration stds. are prepared

External QC Check Sample Recoveries within 90-110%

EPA 314.0, 9.2.5

EPA QC Check Sample Recoveries within 90-110%

EPA 218.6, 10.5 analyzed quarterly
EPA 300.0, 9.2.3 analyzed quarterly (Inorganics)

Matrix Spike Recoveries within 70-130%

EPA 321.8, 9.4, analyzed **every 10 samples**

Matrix Spike Recoveries within 75-125%

EPA 300.1, 9.4.1, analyzed **every 10 samples**
EPA 317.0, 9.4.1, analyzed every 20 samples or batch
EPA 326.0, 9.8, analyzed every 20 samples or batch

Matrix Spike Recoveries within 80-120%

EPA 314.0, 9.4.1, every 20 samples or batch

Matrix Spike Recoveries within 90-110%

EPA 218.6, 10.4, analyzed **every 10 samples**
EPA 300.0, 9.4, analyzed **every 10 samples** (Inorganics)

Duplicate Precisions within 15%

EPA 314.0, 9.4.2, analyzed every 20 samples or batch

Duplicate Precisions within 10%

EPA 300.1, 9.4.3, 20% allowed for conc. 1-100x the MDL, analyzed **every 10 samples** or batch
EPA 317.0, 9.4.3, 20% allowed for conc. 1-5x the MRL, analyzed every 20 samples or batch
EPA 326.0, 9.9, 20% allowed for conc. 1-5x the MRL, analyzed every 20 samples or batch

Surrogate Recoveries within 90-115%

EPA 300.1, 317.0, 9.4.2, for Dichloroacetic Acid as surrogate
EPA 326.0, 9.7, for Dichloroacetic Acid as surrogate

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
Fluoride	4.0 mg/L
Chlorite	1.0 mg/L
Bromate	0.010 mg/L

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
EPA 9056, 8.3, not necessarily required each batch

Matrix Spike & Sample Duplicate every 20 samples
EPA 331.0, 332.0, 9.3.7-9.3.8, or **batch**, MSD allowed in lieu of sample duplicate
EPA 7199, 8.6 or **batch**

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)

Internal Standard Responses
EPA 331.0, 332.0, 9.3.4, 70-130% from last Calib. Verification

Peak Gaussian Factor evaluated each analytical batch
EPA 300.1, 317.0, 9.3.3
EPA 326.0, 9.10

Analysis of Synthetic Sample Matrix Blank & Fortified Synthetic Sample Matrix quarterly
EPA 331.0, 332.0, 9.4

Criteria for Qualitative Identification of Perchlorate (EPA 331.0, 332.0)
GC retention times for target analytes agree match the retention times for the isotopically labeled analogs (within 2%)
Integrated peak areas for both quantitation mass ions meet isotope abundance ratio criteria (within 25%)

Minimum Reporting Level (MRL) Standard each analytical batch for Bromate & Chlorite
40 CFR 141.131(b)(2)(iv), 50-150% at 20.0 ug/L Chlorite and 5.0 ug/L Bromate
(1.0 ug/L Bromate for **EPA 317.0, 326.0, 321.8**)

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 Mercuric 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

TURBIDIMETRIC MEASUREMENTS

REQUIRED REAGANTS & STANDARDS

Sulfate – EPA 375.4, 9038; SM426C (15th ed.), SM4500SO4= E; ASTM D516-90

Conditioning reagent (Magnesium Chloride, Acetate Buffer, Potassium Nitrate) (SM4500SO4= E)
Conditioning reagent (HCl, Ethanol or Isopropanol, NaCl, Glycerol) (all other methods)
Barium Chloride, to form Barium Sulfate suspension
Maximum Turbidity measurement during a 5-minute interval recorded (except SM4500SO4= E, where the turbidity after 5 minutes is recorded)

Turbidity – EPA 180.1; SM2130B; ASTM D1889-94A; USGS I-3860-85

Hydrazine Sulfate & Hexamethylenetetramine standard (Formazin or Polystyrenedivinylbenzene)

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

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

Turbidity

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

Sulfate

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank

SM1020B, 5, applies to all SM methods unless more stringent requirements found in individual mtds
EPA 375.4, 7.1

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 180.1, 9.2.2, 3 stds. needed & verified at least **every 6 months**

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 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)

Calibration Verification every 3-4 samples

EPA 375.4, 6.3.4

Turbidities measured in 30-second intervals for 1-5 minutes & maximum reading taken

EPA 375.4, 6.2

ELECTROCHEMICAL METHODS
POTENTIOMETRY – ION SPECIFIC ELECTRODES; CONDUCTIMETRY; AMPEROMETRY – POLAROGRAPHY, ANODIC STRIPPING VOLTAMETRY

REQUIRED REAGANTS & STANDARDS

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, 350.3; SM4500NH3 D, SM4500NH3 E (both >=19th ed.); ASTM D1426-98B; SM4500NH3 F, SM4500NH3 G (both <=18th ed.)
- Ammonia membrane electrode & filling solution
- Sodium Hydroxide to adjust pH above 11 (immerse electrode in solution FIRST)
- **SM4500NH3 G (<=18th ed.) & SM4500NH3 E (>=19th ed.)**: standard additions methods for NH3

Ammonia – Technicon 378-75WE
- Autoanalyzer with Ammonia sensitive electrode detector

Arsenic – EPA 7063
- Gold metal film deposited on glassy carbon electrode (+145 mv applied potential vs. SCE)
- Saturated Calomel reference electrode (SCE)
- Hydrochloric Acid, to acidify sample (2 M)

Biochemical Oxygen Demand – EPA 405.1; SM5210B; USGS I-1578-78; AOAC 973.44; ANSI Photo. Effluents
- Oxygen Membrane Electrode
- Glucose-Glutamic Acid standard
- Sulfuric Acid or Sodium Hydroxide to adjust sample pH to 6.5-7.5 (if sample pH not 6.0-8.5 prior to testing)
- Sodium Sulfite (prepared fresh daily) to remove residual Cl2 if present (starch-iodine titrimetric endpoint)
- Phosphate Buffer, Calcium Chloride, Ferric Chloride, & Magnesium Sulfate for dilution water
- Seed (if chlorinated effluents are analyzed)

Carbonaceous Biochemical Oxygen Demand – SM5210B
- Same reagents as for BOD above, plus:
- Nitrification Inhibitor, added to all samples & quality control items (2-Chloro-6-(trichloromethyl)pyridine)

Bromide – EPA 9211
- Bromide Specific Electrode
- Sulfuric Acid, to raise sample pH to 4 (eliminate cyanide, sulfide, ammonia interferences)
- EDTA, to remove interferences from multivalent metal ions
- Phosphoric Acid, to remove Fe interference
- Sodium Nitrate, ionic strength adjustment reagent

Cadmium – ASTM D3557-95C; SM3130B
- Hanging drop Hg electrode & Calomel ref. electrode (-0.8 v to deposit Cd onto Hg, re-oxidized at –0.6 v)
- Nitric Acid digestion solution
- Ammonium Citrate buffer, to adjust sample pH to 3.0
- Hydroxylamine, to reduce interfering Fe(III) to Fe(II)

Chloride – EPA 9212
- Chloride Specific Electrode
- EDTA, to remove interferences from polyvalent cations
- Sulfuric Acid, to adjust sample pH to 4 (eliminate sulfide, cyanide, ammonia interferences)
- Potassium Bromate, to remove interferences from bromide & iodide
- Sodium Nitrate, ionic strength adjustment reagent

Chlorine – SM4500CL I

Platinum & Iodide Selective Electrodes
Potassium Iodate standard
Potassium Iodide, to release iodine upon reaction with chlorine
Acetate Buffer to adjust sample pH to 4-5

Chlorine – Orion 97-70 instruction manual

Chlorine membrane electrode

Chromium(VI) – EPA 7198

Dropping Hg electrode & Ag/AgCl reference electrode (Cr(VI) reduces to Cr(III) at -0.250 v)
Ammonia Buffer, as supporting electrolyte (also reduces Cu(II) interference)

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 9213; SM4500CN- F

Cyanide Specific Electrode with Potassium Nitrate filling solution
Lead Carbonate, to remove sulfide interference
Sodium Hydroxide, ionic strength adjustment reagent

Total Cyanide – OIA 1677

Autoanalyzer with Cyanide Specific Electrode
Gas-diffusion membrane into proprietary receiver solution

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

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

Fluoride Distillation – SM4500F- B (required unless comparability data for rep. effluents shows otherwise)

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 340.2, 9214; SM4500F- C; ASTM D1179-93B; USGS I-4327-85 & Technicon 380-75WE (automated)

Fluoride Specific Electrode
Ionic Strength Adjustment Buffer (Acetic Acid, Sodium Chloride, Cyclohexylenediaminetetraacetic Acid)

pH – EPA 150.1, 9040, 9045; SM4500H+ B; ASTM D1293-95A, D1293-95B; USGS I-1586-85; AOAC 973.41

pH Glass Electrode
pH Standard Buffers
SW-846: Use EPA 9040 if Aqueous Phase > 20% of sample; otherwise, must use EPA 9045

pH – EPA 150.2; Technicon 378-75WA

pH Glass Electrode
Autoanalyzer or continuous readout flow cell
pH Standard Buffers

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; SM4500NH3 D, SM4500NH3 E (both >=19th ed.); SM4500NH3 F, SM4500NH3 G (<= 18th ed.)

Sodium Hydroxide distillation reagent
Indicating Boric Acid receiver solution
Ammonia membrane electrode & filling solution
Sodium Hydroxide to adjust pH above 11 (immerse electrode in solution FIRST)
SM4500NH3 G (<=18th ed.) & SM4500NH3 E (>=19th ed.): standard additions methods for NH3

Kjeldahl Nitrogen – EPA 351.4; ASTM D3590-89A

Ammonia membrane electrode & filling solution
Sodium Hydroxide, EDTA, & Sodium Iodide to adjust pH above 11 (immerse electrode in solution FIRST)

Lead – ASTM D3559-95C; SM3130B

Hanging drop Hg electrode & Calomel ref. electrode (-0.8 v to deposit Pb onto Hg, re-oxidized at –0.45 v)
Nitric Acid digestion solution
Ammonium Citrate buffer, to adjust sample pH to 3.0
Hydroxylamine, to reduce interfering Fe(III) to Fe(II)

Mercury – EPA 7472

Gold metal film deposited on glassy carbon electrode (+500 mv applied potential vs. SCE)
Saturated Calomel reference electrode (SCE)
Hydrochloric Acid or Sodium Chloride, to adjust all samples & stds. to 0.1 M chloride

Nitrate – EPA 9210; SM4500NO3- D; Orion 601

Nitrate Specific Electrode with Ammonium Sulfate filling solution
Buffer Solution (Aluminum Sulfate, Silver Sulfate, Boric Acid, Sulfamic Acid, Sodium Hydroxide to pH 3)

Dissolved Oxygen – EPA 360.1; SM4500O G; ASTM D888-92B; USGS I-1576-78

Specific Oxygen Uptake Rate – SM2710B

Oxygen Membrane Electrode

Potassium – SM3500K E (<=19th ed.), SM3500K C (20th ed.)

Potassium Ion Specific Electrode
Sodium Chloride ionic strength adjustment solution & reference electrode filling solution

Salinity – SM2520B

Synthetic Seawater samples of known Salinity, to calibrate Conductivity Meter

Specific Conductance – EPA 120.1, 9050; SM2510B; ASTM D1125-95A; USGS I-1780-85; AOAC 973.40

Sodium Chloride or Potassium Chloride standards (Wheatstone Bridge with platinum electrodes)

Sulfide Distillation – EPA 9030

Sulfuric Acid for Acid-soluble Sulfides (EPA 9030)
Zinc Acetate & Formaldehyde gas washing solutions
Tin(II) Chloride & Hydrochloric Acid for Acid-insoluble Sulfides (EPA 9031)

Sulfide – EPA 9215

Sulfide Specific Electrode
Silver Nitrate & Sodium Chloride, to standardize Sulfide standards
Anti-oxidant Buffer (Sodium Salicylate & Ascorbic Acid)

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, pH (CWA & SDWA)

Analyze Immediately in field or upon arrival at the laboratory, glass bottle & top
Dissolved Oxygen (electrode method), Salinity (6-mo hold if wax seal is used)

8-Hour Holding Time, glass bottle & top, fix on-site & store in the dark
Dissolved Oxygen (Winkler Titration)

24-Hour Holding Time, plastic or glass containers, 4 C
Chromium(VI), pH (RCRA)

48-Hour Holding Time, plastic or glass containers, 4 C, unpreserved
Biochemical Oxygen Demand, Carbonaceous Biochemical Oxygen Demand, Nitrate

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
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 $NaAsO_2$ or Ascorbic Acid if oxidizing agents present (RCRA))

28-Day Holding Time, plastic or glass containers, 4 C
Bromide, Chloride, Specific Conductance

28-Day Holding Time, plastic container (only)
Fluoride

28-Day Holding Time, plastic or glass containers, 4 C, Sulfuric Acid to pH<2
Ammonia, Total Kjeldahl Nitrogen, Organic Nitrogen

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

6-Month Holding Time, plastic or glass containers, Nitric Acid to pH<2
Metals (except Cr(VI) & Hg; add HNO_3 if sample unpreserved & let stand prior to analysis)

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank

SM1020B, 5, applies to all SM methods unless more stringent requirements are found in individual mtds
EPA 350.2, 7.4
EPA 350.3, 7.2
EPA 351.3, 8.4
EPA 351.4, 8
D1426-93B, 21.1, to calibrate NH3 electrode
OIA1677, 10.3, CF (if used) < 10% RSD

4 standards

SM3500K E (<=19th ed.), 4

5 standards + blank

EPA 7063, 7.7
EPA 7472, 7.8

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 90-110%

EPA 7063, 7472, 8.1 & 8.2, also **every 10 samples & end of run**
EPA 9210, 9211, 9212, 9213, 9214, 9215, 8.2-8.3, plus after **every 10 samples & end of run**, calibration blank analysis also required each time

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

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

OIA1677, 9.2

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Matrix Spike Recoveries within 75-125%

EPA 7063, 7472, 9210, 9211, 9212, 9213, 9214, 9215, 8.5, analyzed every 20 samples or batch

Duplicate Precisions within 20%

EPA 7063, 7472, 9210, 9211, 9212, 9213, 9214, 9215, 8.5, MSD or sample dup. analyzed every 20 samples or batch

Analyte Concentrations in Blank

EPA 9210, 8.4, < 1.0 mg/L Nitrate
EPA 9211, 8.4, < 0.3 mg/L Bromide
EPA 9212, 8.4, < 1.0 mg/L Chloride
EPA 9213, 8.4, < 0.03 mg/L Cyanide
EPA 9214, 8.4, < 0.1 mg/L Fluoride
EPA 9215, 8.4, < 0.05 mg/L Sulfide

BOD Results 198 +/- 30.5 mg/L for 300 mg/L Glucose-Glutamic Acid Solution

SM5210B, 6a, can adjust amount of seed added to blanks & samples such that corresponding GGA results will achieve criteria (NOTE: EPA allows CBOD results to be independently evaluated by the laboratory with at least 20 replicate determinations if these control limits are not within 198 +/- 30.5 mg/L; value must be above 150 mg/L & precision must be below +/- 26 mg/L)

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

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

RCRA TOXICITY CHARACTERISTICS

Arsenic	5.0 mg/L
Cadmium	1.0 mg/L
Lead	5.0 mg/L
Mercury	0.2 mg/L

ADDITIONAL REQUIREMENTS

Matrix Spikes, Control Standards, & Duplicates at least 15% of workload for any parameter
USGS Bk. 5, Ch. A1, p.7, applies to all USGS Metals & General Chemistry mtds.

Method of Standard Additions required to quantitate Metal analytes
EPA 7198, 8.3 (Cr(VI) by differential pulse polarography)
ASTM D3557-95C, 32 (Cd by ASV)
ASTM D3559-96C, 33 (Pb by ASV)

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)

Matrix Spike & Matrix Spike Duplicate every 10 samples
OIA1677, 9.3

Matrix Spike & Matrix Spike Duplicate each batch of 20 samples or fewer
EPA 7063, 7472, 8.5, may use sample dup. in place of MSD

Calibration Verification every 10 samples
OIA1677, 9.5

Calibration Verification every 15 samples
EPA 7198, 8.4, second-source std.

Calibration Verification every 3 hours
D1426-93B, 21.2.1, for NH3 by electrode

BOD & CBOD Calculation Criteria
SM5210B, 5, only sample dilutions with at least 2.0 mg/L oxygen depletion after 5 days incubation at 20 C, with at least 1.0 mg/L dissolved oxygen remaining in that dilution, used to calculate results

Duplicate Readings until successive results agree within 0.1 pH units
EPA 9040, 7.2

TITRIMETRIC METHODS
includes Visual & Electrometric Endpoints

REQUIRED REAGANTS & STANDARDS

Acidity – EPA 305.1; SM2310B(4a); ASTM D1067-92
Sodium Hydroxide titrant
Hydrogen Peroxide digestion reagent
Phenolphthalein indicator (colorless to red) or pH meter to detect endpoint
KHP standard

Alkalinity – EPA 310.1; SM2320B; ASTM D1067-92; USGS I-1030-85; AOAC 973.43
Sulfuric Acid or Hydrochloric Acid titrant
Bromcresol Green (green to yellow) or Methyl Orange (yellow to red) indicators,
or pH meter to detect endpoint
Sodium Carbonate or Calcium Carbonate standard

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; SM4500NH3 C (>=19th ed.), SM4500NH3 E (<=18th ed.)
Sulfuric Acid titrant
Mixed Indicator (Methyl Red + Methylene Blue) (green to lavender)

Bromide – EPA 320.1; ASTM D1246-95C; USGS I-1125-85
Phenylarsine Oxide or Sodium Thiosulfate titrant
Calcium Hypochlorite, to convert Bromide+Iodide to bromate & iodate
Bromine Water, to convert Iodide to iodate (Bromide determined by difference)
Calcium Oxide, to remove Fe, Mn, organic matter interferences

Calcium – EPA 215.2; SM3500Ca D (<=19th ed.), SM3500Ca B (20th ed.); ASTM D511-93A
EDTA titrant (Disodium Ethylenediaminetetraacetic acid)
Sodium Hydroxide to adjust sample pH to 12-13 (magnesium precipitates out)
Murexide (pink to purple) or Eriochrome Blue Black R (red to blue) indicators

Chemical Oxygen Demand – EPA 410.1, 410.2, 410.3; SM5220B, SM5220C; USGS I-3560-85, I-3562-85; ASTM D1252-95A; AOAC 973.46; ANSI Photo. Effluents
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)
Ferrous Ammonium Sulfate titrant
Ferroin Indicator (blue-green to red-brown)
Open Reflux digestion system (EPA 410.2, 410.3; SM5220B; USGS I-3562-85)
Closed Reflux digestion system (EPA 410.1; SM5220C; ASTM D1252-88A; USGS I-3560-85; AOAC 973.46; ANSI Photo Effluents)

Chloride – EPA 9253; SM4500Cl- B; ASTM D512-89B; USGS I-1183-85
Silver Nitrate titrant
Alum (Aluminum Hydroxide) to decolorize samples
Sulfuric Acid or Sodium Hydroxide to adjust sample pH to 7-10
Potassium Chromate indicator (white precipitate to yellow precipitate)

Chloride – EPA 325.3, 9252; SM4500Cl- C; ASTM D512-89A; USGS I-1184-85; AOAC 973.51

Mercuric Nitrate titrant
Mixed Indicator (Diphenylcarbazone, Nitric Acid, Xylene Cyanol FF for low-Cl- samples) (blue-green to purple)
Mixed Indicator (Diphenylcarbazone, Bromphenol Blue for high-Cl- samples; add Nitric Acid to pH 2.5 (purple to yellow), then titrate (yellow to purple))

Chloride – SM4500Cl- D

Silver Nitrate titrant
Glass & Ag/AgCl Electrodes for potentiometric endpoint

Chlorine – EPA 330.1; SM4500CL D, SM4500CL E (low-level free chlorine); ASTM D1253-92

Phenylarsine Oxide titrant
Phosphate Buffer to adjust sample pH to 6.5-7.5
Platinum Electrode & Amperometric Detection System

Chlorine – EPA 330.2; SM4500CL C

Iodine or Potassium Iodate titrant
Phenylarsine Oxide or Sodium Thiosulfate
Potassium Iodide & Starch Indicator (colorless to blue), or amperometric detection

Chlorine – EPA 330.3; SM4500CL B

Phenylarsine Oxide or Sodium Thiosulfate titrant
Potassium Iodide reagent (iodine liberated upon reaction with chlorine)
Potassium Bi-iodate or Potassium Dichromate primary standard
Starch indicator (blue to colorless)

Chlorine – EPA 330.4; SM4500CL F

Ferrous Ammonium Sulfate titrant
DPD Indicator (N,N-Diethyl-p-phenylenediamine) (red to colorless)
Potassium Dichromate primary standard
Potassium Iodide, to convert monochloramine & dichloramine to chlorine
Glycine, to determine bromine+iodine & subtract from the total halogen result

Total Chlorine – EPA 9076

Silver Coulometric Titration Cell
Chlorobenzene standard

Total Chlorine – EPA 9077

Metallic Sodium, Naphthalene, & Diglyme (converts organic halogens to sodium halides)
Mercuric Nitrate titrant
Diphenylcarbazone indicator (yellow to violet)

Chlorine Dioxide – SM4500ClO2 D

Ferrous Ammonium Sulfate titrant
DPD Indicator
Glycine, to suppress the response due to free chlorine

Chlorine Dioxide – SM4500ClO2 E

Sodium Thiosulfate or Phenylarsine Oxide titrant
Platinum Electrode & Amperometric detection system
Potassium Iodide, to release iodine upon reaction with chlorine
Phosphate Buffer, to adjust sample to pH 7 for residual chlorine determination
Hydrochloric Acid, for chlorite determination (chlorine dioxide by difference)

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 9014; SM4500CN- D; ANSI Photo. Effluents

Silver Nitrate titrant
p-Dimethylaminobenzalrhodanine Indicator (yellow to salmon)
Sodium Chloride, to standardize titrant

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

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

Hardness – EPA 130.2; SM2340C; ASTM D1126-86(92); USGS I-1338-85; AOAC 973.52B

EDTA titrant (Disodium salt of Ethylenediaminetetraacetic Acid)
Calcium Carbonate standard
Eriochrome Black T or Calmagite indicators (red to blue)
Sodium Cyanide, Sodium Sulfide, or CDTA inhibitors (to sharpen titration endpoints if necessary)
Ammonia Buffer to adjust sample pH to 10.0-10.1

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; SM4500NH3 C (>=19th ed.), SM4500NH3 E (<=18th ed.); ASTM D3590-89A; AOAC 973.48; PAI-DK01

Sodium Hydroxide distillation reagent
Indicating Boric Acid receiver solution
Sulfuric Acid titrant
Mixed Indicator (Methyl Red + Methylene Blue) (green to lavender)

Organic Halogens – EPA 1650, 9020, 9021, 9022, 9023; SM5320B

Granular Activated Column adsorbent for TOX (EPA 1650, 9020, 9022)
Trichlorophenol or Chloroform standards (instrument calibration, adsorption efficiency checks)
Potassium Nitrate acid wash solution
Sodium Chloride, for coulometric titration cell testing & inorganic halide std.
Ethyl Acetate extraction solvent (EPA 9023)

Dissolved Oxygen – EPA 360.2; SM4500O C; ASTM D888-92A; USGS I-1575-78; AOAC 973.45B

Manganous Sulfate
Alkali-Iodide-Azide reagent (Sodium or Potassium Hydroxide, Sodium Iodide, Sodium Azide)
Sodium Thiosulfate titrant (Winkler titration)
Starch indicator (blue to colorless)
Potassium Bi-iodate primary standard

Sulfide Distillation – EPA 9030

Sulfuric Acid for Acid-soluble Sulfides (EPA 9030)
Zinc Acetate & Formaldehyde gas washing solutions
Tin(II) Chloride & Hydrochloric Acid for Acid-insoluble Sulfides (EPA 9031)

Sulfide – EPA 376.1, 9031, 9034; SM4500S= E (<=18th ed.), SM4500S= F (>=19th ed.); USGS I-3840-85

Sodium Thiosulfate or Phenylarsine Oxide titrant
Iodine reagent & Starch indicator (blue to colorless)
Potassium Bi-iodate primary standard

Sulfite – EPA 377.1; SMSM4500SO3= B

Potassium Iodide & Potassium Iodate titrant
Starch Indicator (colorless to blue)
EDTA fixing solution
Sulfamic Acid, to eliminate nitrite interference

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

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, Sulfite, Chlorine Dioxide

8-Hour Holding Time, glass bottle & top, fix on-site & store in the dark

Dissolved Oxygen (Winkler Titration)

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

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

Bromide, Chloride

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

Ammonia, Chemical Oxygen Demand, Total Kjeldahl Nitrogen, Organic Nitrogen

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

TOX (must have Teflon-lined cap)

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

Hardness

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

Cyanide	0.2 mg/L
Chlorine	4.0 mg/L as Cl2
Chloramine	4.0 mg/L as Cl2
Chlorine Dioxide	0.8 mg/L as ClO2

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample Recoveries & Matrix Spike Recoveries within 80-120%
PAI-DK01

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

External QC Check Sample Recoveries within 90-110%
PAI-DK01

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 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)

Matrix Spikes analyzed every 10 samples
EPA 9020, 8.5 & **EPA 9021**, 8.4
EPA 1650, 9.3

Matrix Spike & Sample Duplicate every 20 samples
EPA 9253, 8.4, or **batch**
EPA 9010/9014, 8.4 & 8.5

Matrix Spike & Matrix Spike Duplicate
EPA 5050, 9023, 9076, 8.3, for oxidative combustion & bomb preparations of solid waste for EOX & Cl2

Independent Quality Control Check Samples analyzed every 15 samples
EPA 9021, 8.3

Method Blank analyzed every 20 samples or each 8 or 12-hour work shift, per matrix
EPA 9021, 8.2
EPA 9253, 8.3

Spike Duplicate analyzed every 10 samples
EPA 9022, 8.6

All Samples analyzed in Duplicate
EPA 9020, 8.2 & **9021**, 8.5

Calibration Verification & Method Blank analyzed every 5 samples
EPA 9020, 7.2.2-7.2.3, since Nitrate-wash blanks & CCV repeated **every 10 pyrolysis determinations**

Calibration Verification every 15 samples
EPA 9022, 8.5, second-source std.

Reagent Blank analyzed every 8 samples
EPA 9020, 8.3

GRAVIMETRIC (PRECIPITATION) METHODS

REQUIRED REAGANTS & STANDARDS

Magnesium – SM3500Mg D (<=19th ed.); ASTM D511-77A

Diammonium Hydrogen Phosphate precipitating reagent (as MgNH4PO4, ignited to Mg2P2O7)

Oil & Grease – EPA 413.1, 9070, 9071; SM5520B, SM5520D, SM5520E, SM5520F

Trichlorotrifluoroethane Extraction Solvent (1,1,2-Trichloro-1,2,2-trifluoroethane)
Sodium Sulfate drying agent
Corn Oil, or Isooctane/Hexadecane/Benzene mixture, as Oil & Grease standard
EPA 413.1, 9070, SM5520B, SM5520F: Separatory funnel extraction
EPA 9071, SM5520D, SM5520E: Soxhlet extraction
SM5520E: Magnesium Sulfate Monohydrate, to dehydrate sludge
SM5520F: Silica Gel cleanup sorbent, to quantitate Oil & Grease as "Hydrocarbons"

Oil & Grease; Total Petroleum Hydrocarbons – EPA 1664

n-Hexane extraction solvent
Sodium Sulfate drying agent
Silica Gel, to remove aromatics & polars & vegetative matter (to determine Total Petroleum Hydrocarbons)
Stearic Acid & Hexadecane standard
2-mg and 1000-mg Class S weights to calibrate analytical balance

Potassium – SM317B (14th ed.)

Cobaltinitrite precipitating reagent (potassium precipitates as NaK2Co(NO2)6)
Potassium Dichromate oxidizing agent (excess measured at 425 nm)

Total Residue (TS) – EPA 160.3; SM2540B; USGS I-3750-85
Filterable Residue (TDS) – EPA 160.1; SM2540C; USGS I-1750-85
Nonfilterable Residue (TSS) – EPA 160.2; SM2540D; USGS I-3765-85

Volatile Residue – EPA 160.4; SM2540E; USGS I-3753-85

Glass fiber filter, ignition of volatile matter at 500-600 degrees Celsius

Settleable Residue – EPA 160.5; SM2540F

Volumetric (Imhoff Cone) or gravimetric method

Total, Fixed, & Volatile Solids – SM2540G

Ignition of voltatile residue from sludge 450-550 degrees Celsius

Silica as SiO2 – SM4500Si C (<=19th ed.)

Perchloric Acid or Hydrochloric Acid as dehydrating agents, sample evaporated to dryness
Hydrofluoric & Sulfuric Acids, to volatilize SiO2 as SiF4
Platinum crucibles, to hold SiO2 residue when dried at 110 C and ignited at 1200 C

Sulfate – EPA 375.3; SM4500SO4= C, SM4500SO4= D; AOAC 925.54

Barium Chloride precipitating agent (digest at 80-90 C for 2 hours)
Precipitate dried in 105 C oven (EPA 375.3; SM4500SO4= D)
Precipitate ignited at 800 C for 1 hour (SM4500SO4= C)

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

7-Day Holding Time, plastic or glass containers, 4 C
Total Residue, Filterable Residue, Nonfilterable Residue, Settleable Residue, Volatile Residue

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

28-Day Holding Time, glass container (only), 4 C, HCl or H2SO4 to pH<2
Oil & Grease

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 prior to analysis)

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

Average Recovery & Standard Deviation of Recovery compared to Acceptance Criteria in Table of Test Method
EPA 1664, MDL also required each analyst, <1.4 mg/L for Oil & Grease and <1.6 mg/L for TPH

QUALITY 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 1664

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 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)

Matrix Spike & Sample Duplicate every 20 samples
EPA 9071, 8.3 or **batch**

Matrix Spike every 20 samples per site
EPA 1664, 9.3

Method Blank analyzed every 20 samples or each 8 or 12-hour work shift, per matrix
EPA 9071, 8.2

Residues weighed to constant weight (repeat cycle of drying, cooling, desiccating, & weighing)
EPA 160.1, 160.2

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)

VOLATILE ORGANICS GAS CHROMATOGRAPHY

REQUIRED REAGANTS & STANDARDS

EPA 502.2, 8021 (GC-PID & ELCD series); 601 (GC-ELCD); 602 (GC-PID); 603, 8015 (GC-FID); SM6220B (<=19th ed.) (GC-PID), SM6230B (<=19th ed.) (GC-ELCD) (both packed-column); SM6200C (20th ed.), SM6230D (<=19th ed.) (both capillary-column GC-PID & ELCD series)

Tenax/Silica Gel/Charcoal solid sorbent trap, or synthetic polymer, conditioned per manufacturer's instructions **(includes EPA 5030 prior to 8021)**

Note: Direct sample injection or other sample introduction techniques available for **EPA 8015**

EPA 504, 504.1, 505, 8011 (GC-ECD); EPA 8031 (GC-NPD); SM6231B, SM6232B (GC-ECD)

Sodium Chloride to salt aqueous phase (dissolve before hexane is added)
n-Hexane micro-extraction solvent (**EPA 504, 504.1, 505, 8011; SM6231B**), to extract EDB & DBCP
MTBE micro-extraction solvent (**EPA 8031**)
Pentane, Hexane, Isooctane, or Methylcyclohexane extraction solvents (**SM6232B**), to extract THM's

EPA 1671 (GC-FID)

Internal Standards

EPA 3585 with EPA 8021, 8031, 8033, 8260

Waste Dilution Solvent: n-Hexadecane

EPA 5035 with EPA 8021, 8260

Methanol (or other water-miscible solvent)
Sodium Bisulfate, as solid preservative

EPA 8032 (GC-ECD)

KBr & HBr, to brominate Acrylamide
Ethyl Acetate extraction solvent
Sodium Sulfate to salt the aqueous phase
Sodium Thiosulfate, to quench excess bromine

Gasoline-Range Organics

Methanol dissolution of Solids
Gasoline commercial std. (**EPA 8015, NWTPH-Gx, CA LUFT, AK-101, OA-1** + BTEX, **TN-GRO**, as LCS)
Component std. of BTEX, MTBE, i-C5, n-C5, i-C8, n-C9, C10H8, 124-Trimethylbenzene (**MA-VPH**)
Component std. of BTEX, MTBE, C10H8, 124- & 135-Trimethylbenzenes (**WI-GRO, OK-GRO, ME 4.2.17**)
Component std. of BTEX, i-C5, i-C8, n-C7, 124-Trimethylbenzene (**MT-GRO, TN-GRO**)
2,5-Dibromotoluene Surrogate (**MA-VPH**)
Bromofluorobenzene or Trifluorotoluene Surrogate (**AK-101**)
Isopropyltoluene Surrogate (**TN-GRO**)

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

14-Day Holding Time, glass vial w/ Teflon-lined septum, 4 C, 0.008% Na2S2O3

Purgeable Halocarbons (NO HEADSPACE)

14-Day Holding Time, glass vial with Teflon-lined septum, 4 C, 0.008% Sodium Thiosulfate, HCl to pH<2 (NO HEADSPACE)

Purgeable Aromatic Hydrocarbons (7-Day Holding Time if pH adjustment not made)

14-Day Holding Time, glass vial with Teflon-lined septum, 4 C, 0.008% Sodium Thiosulfate, pH 4-5

Acrolein & Acrylonitrile (3-Day Holding Time for Acrolein & 7-Day Holding Time for Acrylonitrile if pH adjustment not made)

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank

SM6020B, 1a & 1b, applies to all SM Organics mtds., int. std. response factor (if used) < 20% RSD
EPA 502.2, 10.2, calibration factor or response factor (if used) < 10% RSD
EPA 504.1, 10.1, calibration factor (if used) < 20% RSD
EPA 601, 602, 603, 7.3, calibration factor or response factor (if used) < 10% RSD
OK-GRO, correlation coefficient > 0.990
AK-101, MT-GRO, calibration factor (if used) < 25% RSD
OA-1, ME 4.2.17, calibration factor (if used) < 20% RSD or correlation coefficient > 0.990

5 standards + blank

EPA 1671, 10.2, calibration factor (if usd) < 10% 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 8011, 8015, 8021, 8031, 8032, 8033** (GC Volatile Organics)
Requires **client notification of analytes** quantitated from CF or RF when mtd. criteria not met (and mean RSD < 20%)
WI-GRO, NWTPH-Gx, correlation coefficient > 0.990
TN-GRO, calibration factor (if used) < 25% RSD

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 60-140%

EPA 8011, 8.3, also analyzed every 20 samples

Recovery 75-125%

MA-VPH, AK-101, MT-GRO, TN-GRO

Recovery 80-120%

EPA 502.2, 10.3.4, PLUS (**40 CFR 141**) 50-150% recovery for MRL verification std. at 1.0 ug/L for THM's
EPA 8021, 7.4.8, for target analytes that boil below 30 C
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-1, WI-GRO, ME 4.2.17

Recovery 85-115%

EPA 8000, 7.7

Recovery 90-110%

CA-LUFT

Recovery within the Test Method QC Acceptance Criteria

EPA 601, 602, 603, 7.5
EPA 1671, 13.1

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

Mean Accuracy 60-140% for each analyte

EPA 8011, 8.2, 0.030 ug/L used for spike concentration & 7 replicates used
OK-GRO, precision < 30% RSD

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

EPA 504.1, 9.2, MDL study also required
MA-VPH, precision RSD < 25% RSD

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

EPA 502.2, 9.3, concentrations 0.1-5.0 ug/L for each analyte, **MDL** also required

Average Recovery & Std. Dev. of Recovery compared to Acceptance Criteria in Table of Test Method

EPA 601, 602, 603, 8.2, concentration 20 ug/L or less for each target analyte
EPA 8000, 8.6, applicable to all **EPA 8000-series** methods

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Control Standard Recoveries within 50-150%

TN-GRO, LCSD Precisions < 20% RPD, at 1.0 mg/L spike concentration

QC Check Sample (or LCS or LFB) Recoveries within 60-140%

EPA 8011, 8.3.2, each target analyte concentration must be 0.25 ug/L

QC Check Sample Recoveries within 70-130%

EPA 502.2, SDW Technical Notes for **EPA 524.2**
EPA 504.1, 9.3, Recommended criteria, but required every **10 samples**, each analyte must be 0.25 ug/L

Control Standard Recoveries 75-125%

AK-101, analyzed every **10 samples** & end of run, LCSD Precisions < 20% RPD

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

EPA 601, 602, 603, 8.4
EPA 8000, 8.8, applicable to **EPA 8011, 8015, 8021**

External QC Check Sample Analyzed Quarterly

EPA 502.2, 9.10

External QC Check Sample Recoveries within 60-140%

EPA 8011, 8.4, analyzed weekly, analyte concentrations at 0.10 ug/L

Matrix Spike Recoveries 60-140%, MSD Precision < 20% RPD

ME 4.2.17

Matrix Spike Recoveries 65-135%

EPA 504.1, 9.5, analyzed every 20 samples

Matrix Spike Recoveries 70-130%, MSD Precision < 50% RPD

MA-VPH

Matrix Spike Recoveries 80-120%

WI-GRO (MSD Precision < 20% RPD), OK-GRO aqueous phase (60-140% for soils)

Surrogate Recoveries 80-120%

EPA 502.2, 9.7 & **551.1**, 9.8

Internal Standards Responses

EPA 502.2, 9.6, 80-120% of mean response from last Initial Calibration

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

1,2-Dibromomethane (EDB)	0.05 ug/L
1,2-Dibromo-3-chloropropane (DBCP)	0.2 ug/L
Each Regulated VOC (MDL requirement)	0.5 ug/L
Total Trihalomethanes	80 ug/L

RCRA TOXICITY CHARACTERISTICS

Benzene	0.5 mg/L	Carbon Tetrachloride	0.5 mg/L
Chlorobenzene	100.0 mg/L	Chloroform	6.0 mg/L
1,4-Dichlorobenzene	7.5 mg/L	1,2-Dichloroethane	0.5 mg/L
1,1-Dichloroethene	0.7 mg/L	Methyl Ethyl Ketone	200.0 mg/L
Tetrachloroethene	0.7 mg/L	Trichloroethene	0.5 mg/L
Vinyl Chloride	0.2 mg/L		

ADDITIONAL REQUIREMENTS

Matrix Spikes analyzed every 10 samples

EPA 601, 602, 603, 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 1671, 9.3

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

Quality Control Check Samples analyzed every 10 samples

EPA 504.1, 9.3, concentration must be 0.25 ug/L for each target analyte

EPA 601 & 602, 8.1.5, frequency may be reduced if Matrix Spike recoveries meet all specified QC criteria

Method Detection Limit Requirements

WI-GRO, 0.1 mg/L for waters & 10 ppm for soils

ME 4.2.17, 10 ug/L for waters

CA-LUFT, 5 ppm headspace

GC Retention Time Definitions for Gasoline Range Organics

EPA 8015, TN-GRO: 2-Methylpentane to 124-Trimethylbenzene, valley-valley integration

MA-VPH: C5 to C8 Aliphatics, C9 to C12 Aliphatics, C9 to C10 Aromatics

WI-GRO, ME 4.2.17: MTBE to Naphthalene, forced baseline integration

MT-GRO: Iso-octane to 124-Trimethylbenzene, forced baseline integration

NWTPH-Gx: Toluene to Naphthalene, forced baseline integration

AK-101: C6 to C10, forced baseline integration

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

40 CFR Part 141, applies to Trihalomethanes

VOLATILE ORGANICS GAS CHROMATOGRAPHY / MASS SPECTROMETRY

REQUIRED REAGANTS & STANDARDS

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

Internal Standards (isotopically labeled for **EPA 1624 & 1666**)
Surrogate solutions
Bromofluorobenzene (BFB) Tuning Solution for MS (**daily**)
Tenax/Silica Gel/Charcoal sorbent trap, or synthetic polymer, conditioned per manufacturer's instructions **(includes EPA 5030 prior to 8260)**
Note: Direct sample injection or other sample introduction techniques available for **EPA 1666**

EPA 3585 with EPA 8021, 8031, 8033, 8260

Waste Dilution Solvent: n-Hexadecane

EPA 5035 with EPA 8021, 8260

Methanol (or other water-miscible solvent)
Sodium Bisulfate, as solid preservative

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

14-Day Holding Time, glass vial w/ Teflon-lined septum, 4 C, 0.008% Na2S2O3

Purgeable Halocarbons (NO HEADSPACE)

14-Day Holding Time, glass vial with Teflon-lined septum, 4 C, 0.008% Sodium Thiosulfate, HCl to pH<2 (NO HEADSPACE)

Purgeable Aromatic Hydrocarbons (7-Day Holding Time if pH adjustment not made)

14-Day Holding Time, glass vial with Teflon-lined septum, 4 C, 0.008% Sodium Thiosulfate, pH 4-5

Acrolein & Acrylonitrile (3-Day Holding Time for Acrolein & 7-Day Holding Time for Acrylonitrile if pH adjustment not made)

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank

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

5 standards + blank

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

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 70-130%

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

Recovery 80-120%

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

Recovery within the Test Method QC Acceptance Criteria

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

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

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

EPA 524.2, 9.3, concentrations 2.0-5.0 ug/L for each analyte, MDL also required

Average Recovery & Std. Dev. of Recovery compared to Acceptance Criteria in Table of Test Method

EPA 624, 1624, 8.2, concentration 20 ug/L or less for each target analyte
EPA 1666, 9.2
EPA 8000, 8.6, applicable to all **EPA 8000-series** methods

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample Recoveries within 70-130%

EPA 524.2, 9.6

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

EPA 624, 8.4
EPA 8000, 8.8, applicable to **EPA 8260**
EPA 1666, 9.3

External QC Check Sample Analyzed Quarterly

EPA 524.2, 9.9

Internal Standards Responses

EPA 524.2, 10.3, 70-130% from last Calib. Verification & 50-150% from last Initial Calibration
EPA 1624, 8.3, serves as Matrix Spike since labeled stds. added to each sample, acceptance criteria in Table

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

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

RCRA TOXICITY CHARACTERISTICS

Benzene	0.5 mg/L	1,1-Dichloroethene	0.7 mg/L
Carbon Tetrachloride	0.5 mg/L	Methyl Ethyl Ketone	200.0 mg/L
Chlorobenzene	100.0 mg/L	Tetrachloroethene	0.7 mg/L
Chloroform	6.0 mg/L	Trichloroethene	0.5 mg/L
1,4-Dichlorobenzene	7.5 mg/L	Vinyl Chloride	0.2 mg/L
1,2-Dichloroethane	0.5 mg/L		

ADDITIONAL REQUIREMENTS

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

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

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

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

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

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

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

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

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

mass ion (m/z)	abundance criteria
50	15-40% of m/z 95
75	30-60% of m/z 95 (30-80% for **EPA 524.2**)
95	100% (base peak)
96	5-9% of m/z 95
173	<2% of m/z 174
174	>50% of m/z 95
175	5-9% of m/z 174
176	95-101% of m/z 174
177	5-9% of m/z 176

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 & Dibenzofurans 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

QUALITY 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

EXTRACTABLE ORGANICS GAS CHROMATOGRAPHY MASS SPECTROMETRY (GC/MS);

REQUIRED REAGANTS & STANDARDS

EPA 608.1, 627 (GC-ECD); 613 (GC-MS or HRMS); 619, 622 (GC-NPD or FPD)

Methylene Chloride extraction solvent
Sodium Sulfate drying reagent
n-Hexane exchange solvent
TCDD Internal Standard, isotopically labeled (**EPA 613**)
Note: GC-MS may also be used for **EPA 608.1, 619, 622, 627**

EPA 525.2, 526, 528 (GC-MS)

Reverse-phase C-18 solid-phase disks or cartridges
Ethyl Acetate, Methylene Chloride, Methanol solid-phase conditioning reagents
Ethyl Acetate, then Methylene Chloride, as eluting solvents (**EPA 525.2**)
Methylene Chloride eluting solvent (**EPA 528**)
Sodium Sulfate drying reagent
Endrin & DDT for GC degradation check (**EPA 525.2**)
Internal Standards (required for **EPA 525.2**)
DFTPP (Decafluorotriphenylphosphine) for tuning MS (**EPA 525.2**) (**daily**)

EPA 615 (GC-MS)

Ethyl Ether extraction solvent
Potassium Hydroxide as phenoxyacid ester hydrolysis reagent
Sulfuric Acid to adjust aqueous-phase pH
Acidified Sodium Sulfate dehydrating agent
n-Hexane exchange solvent
Diazald to generate diazomethane derivatizing agent (if diazomethane solution made, usable within 48 hr)
Silicic Acid to quench excess diazomethane from Diazald or diazomethane solution
Surrogate Standards

EPA 548.1 (GC-FID or MS)

Anion Exchange solid-phase disks or cartridges
Methanol, water, 10% Sulfuric Acid/Methanol, water, 1 N NaOH, water to condition the solid phase
10% Sulfuric Acid/Methanol eluting solvent, derivatizes Endothall to its dimethyl ester
Methylene Chloride (**EPA 548.1**)
Sodium Sulfate drying agent
Internal Standards

EPA 613, 8280 (low-resolution GC-MS)

Isotopically labeled C-13 surrogate stds. & internal std., plus Cl-37 cleanup stds.
Perfluorokerosene, for calibrating the resolution of the MS & for use as the lock-mass
Methylene Chloride extraction solvent
Sodium Chloride drying agent
Toluene solvent for Soxhlet Dean-Stark extractions of solids & nonaqueous liquids
1:1 Methylene Chloride / Hexane for extracting tissue samples
Tetradecane (EPA 613) or Tridecane (EPA 8280) as final exchange solvents

EPA 614, 617 (GC-MS alternate detector)

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

EPA 625, 1625 (GC-MS); SM6410B (GC-MS); USGS O-3116-87 (GC-MS); EPA 634 (GC-MS alternate detector); EPA 3510, 3520, 3540, 3541 with EPA 8270 (GC-MS); SM6630D (GC-MS pesticides & PCB's)

Methylene Chloride extraction solvent
Sulfuric Acid & Sodium Hydroxide to adjust aqueous-phase pH
Sodium Sulfate drying reagent
Internal Standards (isotopically labeled for **EPA 1625**)
Surrogate Compounds
DFTPP Tuning Solution for MS
Soxhlet extraction thimble (if solids are extracted)

EPA 633 (GC-NPD or MS)

Methylene Chloride extraction solvent
Sodium Sulfate drying agent
Acetone exchange solvent

EPA 1653 (GC-MS)

Isotopically labeled internal standards
Carbonate Buffer, to raise sample pH to 9.0-11.5
Acetic Anhydride, to derivatize chlorophenolics to acetates
Hexane extraction solvent
Ascorbic Acid, to stabilize chlorocatechols (must calibrate GC-MS with & without ascorbic acid present)

EPA 3545 with EPA 8270

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 8270

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 3561 with EPA 8270

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 8270

Waste Dilution Solvents: Methylene Chloride or Hexane

EPA 613; EPA 3610, 3611 prior to EPA 8270

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 & Dibenzofurans 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 613; EPA 3630 prior to EPA 8270

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 613, 625, 1613, 1625, 1668, 8280, 8290; EPA 3650 prior to EPA 8270

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 3620 prior to EPA 8270

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 3640 prior to EPA 8270

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

EPA 3660 prior to EPA 8270

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

EPA 1613, 1668, 8280, 8290

Activated Carbon Clean-up Sorbent
Conditioning Reagents (5% Toluene & 20% CH3OH in CH2Cl2, 1:1 CH2Cl2/Cyclohexane, & Hexane)
Hexane, 1:1 CH2Cl2/Cyclohexane, & 5% Toluene/20 % CH3OH in CH2Cl2, to elute impurities
Toluene, reverse-flow, to elute Dibenzo-p-dioxins & Dibenzofurans

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

7 Days to Extract Sample, Analyze Extract Immediately; glass container with Teflon-lined cap; 4 C
Dibenzo-p-dioxins & Dibenzofurans (solids, mixed-phase, & tissues for CWA)

7 Days to Extract Sample, 7 Days to Analyze Extract; Teflon-lined cap; 4 C; 0.008% Sodium Thiosulfate
Benzidines (analyze extract immediately if not stored in oxygen-free system)

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)

7 Days to Extract Sample, 40 Days to Analyze Extract; glass containers with Teflon-lined cap; 4 C; pH 5-9
Pesticides (CWA), Organophosphorus Pesticides (RCRA)

14 Days to Analyze Sample, 4 C, Sodium Sulfite & HCl to pH<2
SDWA Chlorinated Phenoxy Acids (Herbicides)

14 Days to Extract Sample, 14 Days to Analyze Extract; 4 C
SDWA Chlorinated Solvents & Disinfection By-Products

14 Days to Extract or Analyze Sample, 4 C
Other SDWA Pesticides & PCB's

30 days to Extract Sample, 45 Days to Analyze Extract; glass container with Teflon-lined cap; 4 C; 0.008% Sodium Thiosulfate
Dibenzo-p-dioxins & Dibenzofurans (RCRA aqueous-phase samples)

1-Year Holding Time, glass container with Teflon-lined cap, 4 C, 0.008% Na2S2O3
Dibenzo-p-dioxins & Dibenzofurans (aqueous-phase samples for CWA)

1-Year Holding Time, glass container with Teflon-lined cap, frozen <-10 C
Dibenzo-p-dioxins & Dibenzofurans (solids, mixed-phases, & tissues for CWA)

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 608.1, 614, 617, 619, 622, 627, 633, 7.2 & 7.3, calib. factors or response factors (if used) < 10% RSD
EPA 613, 7.1, response factors (if used) < 10% RSD
EPA 615, 7.2, calibration factors (if used) < 10% RSD
EPA 625, 7.2.2, response factors (if used) < 35% RSD

4 standards + blank

EPA 548.1, 10.3, response factor < 30% RSD, stds. contain all derivatization reagents

5 standards + blank

EPA 1625, 7.4-7.5, relative response factor (if used) < 20% RSD relative to the isotope-labeled analog, < 35% RSD relative to sample matrix internal standard
EPA 1653, 10.4-10.5, relative response factor (if used) < 20% RSD relative to the isotope-labeled analog, < 35% RSD relative to sample matrix internal standard (**standards acylated** along with samples)
EPA 8270, 7.3, response factor (if used) < 15% RSD for each analyte, response factor < 30%RSD for Calibration Check Compounds
EPA 8275, 7.2.5
EPA 8280, 7.13.3.1-7.13.3.4, relative response factors < 15% RSD
EPA 8000, 7.4-7.5, requires **client notification of analytes** quantitated from CF or RF when mtd. criteria not met (and mean RSD < 20%)
EPA 526, 528, 10.2, relative response factors (if used) < 30% RSD
EPA 680, 7.14 & 9.2.8, relative response factors < 20% RSD

6 standards + blank

EPA 525.2, 7.10 & 10.2, response factor (if used) < 30% RSD

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 70-130%

EPA 525.2, 526, 528, 10.3, absolute peak areas for Internal Standards & Surrogates must also be checked
EPA 526, 528, 10.3, 50-150% allowed for low-level stds.; CCV also required every 10 samples & end of run
EPA 548.1, 10.4, absolute peak area for Internal Standard within 30% of area from most recent calibration verification & within 50% of area from most recent initial instrument calibration
EPA 8280, 7.13.3.6

Recovery 80-120%

EPA 625, 7.3
EPA 680, 9.3.7
EPA 8270, 7.4, for Calibration Check Compounds
EPA 8275, 7.3.2
SM6020B, 1b (applies to SM Organics methods)
40 CFR 141.40, App. A, 3, mid-range std. **plus** 60-140% recovery for std. at or below Minimum Reporting Level (applies to SDWA Unregulated Contaminants)

Recovery 85-115%

EPA 613, 7.1.3

Recovery 90-110%

EPA 608.1, 614, 617, 619, 622, 627, 633, 7.2 & 7.3
EPA 615, 7.2

Recovery within the Test Method QC Acceptance Criteria

EPA 1625, 12.5
EPA 1653, 9.6

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

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

EPA 608.1, 614, 615, 617, 619, 622, 625, 627, 633, 8.2
EPA 613, 8.1.1
EPA 1653, 9.3
EPA 8000, 8.6, applicable to all **EPA 8000-series** methods

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample Recoveries within 70-130%

EPA 526, 9.6 & **528**, 9.8, 50-160% allowed for low-level concentrations

QC Check Sample Recoveries within 80-120%

EPA 548.1, 9.6.2

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

EPA 608.1, 625, 8.4
EPA 8270, 8.7

External QC Check Sample Recoveries within 70-130%

EPA 525.2, 9.9, analyzed quarterly

Matrix Spike Recoveries within 50-150% or 50-160%

EPA 526, 9.10, 70-130% recommended for mid- & high-level spikes, analyzed each extraction batch

Surrogate Recoveries within 70-130%

EPA 526, 9.9
EPA 528, 9.10

Internal Standards Responses

EPA 525.2, 526, 10.3, 70-130% from last Calib. Verification & 50-150% from last Initial Calibration
EPA 528, 9.9, 70-130% from last Calib. Verification & 50-150% from last Initial Calibration
EPA 680, 9.3.6, decrease < 30% from last Calib. Verification & < 50% from last Initial Calibration
EPA 1625, 8.3 & **1653**, 9.4, serves as Matrix Spike since labeled stds. added to each sample, acceptance criteria in Table

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

Alachlor	2.0 ug/L	Atrazine	3.0 ug/L
Benzo(a)pyrene	0.2 ug/L	Chlordane	2.0 ug/L
Bis(2-ethylhexyl) Adipate	400 ug/L	Bis(2-ethylhexyl) Phthalate	6.0 ug/L
Endrin	2.0 ug/L	Heptachlor	0.4 ug/L
Heptachlor Epoxide	0.2 ug/L	Hexachlorobenzene	1.0 ug/L
Hexachlorocyclopentadiene	50 ug/L	Lindane	0.2 ug/L
Methoxychlor	40 ug/L	Simazine	4.0 ug/L
Toxaphene	3.0 ug/L	Pentachlorophenol	1.0 ug/L

RCRA TOXICITY CHARACTERISTICS

o-Cresol	200.0	mg/L	Endrin	0.02	mg/L
m-Cresol	200.0	mg/L	Heptachlor & its Epoxide	0.008	mg/L
p-Cresol	200.0	mg/L	Lindane	0.4	mg/L
Total Cresols	200.0	mg/L	Methoxychlor	10.0	mg/L
2,4-Dinitrotoluene	0.13	mg/L	Chlordane	0.03	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			

ADDITIONAL REQUIREMENTS

All samples spiked with Isotopically Labeled Compounds

EPA 1625, 8.3
EPA 1653, 9.4

Matrix Spikes analyzed every 10 samples

EPA 608.1, 614, 615, 617, 619, 622, 627, 633, 8.4, or Monthly
EPA 613, 8.1.4
SM6020B, 3c, or Monthly (whichever is more frequent) (applies to all SM Organics methods)
USGS Bk.5, Ch.A3, p.5, applies to all USGS Organics mtds., not required if **Surrogates** analyzed **each sample**

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

40 CFR 141.40, App. A, 6, must also **alternate between mid- & low-level** concentrations for spikes (applies to SDWA Unregulated Contaminants)
EPA 680, 10.7
EPA 8000, 8.5, applies to all 8000-series Organics mtds., may use sample dup. in place of MSD

Matrix Spike every 20 samples per site

EPA 625, 8.3

Matrix Spike every 20 samples

EPA 528, 9.11, or each batch whichever more frequent

Quality Control Check Samples analyzed every 10 samples

EPA 613, 8.4

Field Duplicates analyzed

EPA 526, 9.11, each extraction batch unless MSD analyzed

Method Blank analyzed every 20 samples or each 8 or 12-hour work shift, per matrix

EPA 1653, 9.5
EPA 1625, 8.5
EPA 8280, 7.1-7.2

Peak Tailing Factors evaluated each analytical batch
EPA 528, 10.2.3

GC Injector Port Degradation < 20% for Endrin & DDT (evaluated each initial calibration)
EPA 525.2, 10.2

GC Injector Port Degradation < 10% for Endrin & < 5% for DDT
EPA 680, 9.2.5.1 & 9.2.5.3, when Pesticides are determined

GC Retention Time Windows established for each analyte
EPA 680, 9.2.9, within 10 sec between analyses
EPA 1625, 12.3
EPA 1653, 9.6.1.1
EPA 8000, 7.6

Chromatographic Resolution Checks
EPA 525.2, 10.2.4, Anthracene & Phenanthrene, Benz(a)anthracene & Chrysene every 12 hours
EPA 1625, 12.4, Anthracene & Phenanthrene
EPA 1653, 9.6.1.2; 4,6-Dichloroguaiacol & 3,4-Dichloroguaiacol
EPA 8280, 7.12; 2,3,7,8-TCDD & 1,2,3,4-TCDD

Response Factors > 0.050 for System Performance Check Compounds
EPA 8270, 7.4, during Initial Instrument Calibrations & Calibration Verifications

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

Criteria for Qualitative Identification of Dibenzo-p-dioxins & Dibenzofurans (EPA 1613, 1668, 8280, 8290)
GC retention times for target analytes agree match the retention times for the isotopically labeled analogs
GC retention times for target analytes in samples match the retention times in the CCV std.
GC retention times for target analytes in calibration verification std. match the retention times in the GC column performance check solution
MS ion current responses for both quantitation mass ions of target analyte reach maximum simultaneously
MS ion current responses for both quantitation mass ions of each isotopically labeled std. reach maximum simultaneously
Integrated peak areas for both quantitation mass ions meet isotope abundance ratio criteria
Signal-to-noise ratio for GC peaks exceeds 2.5 (10 for EPA 1613)
No GC peaks due to polychlorinated diphenyl ethers are observed

GC/MS Tune Criteria with DFTPP (EPA 525.2, 625, 8270)

mass ion (m/z)	abundance criteria
51	30-60% of m/z 198 (10-80% for **EPA 525.2**)
68	<2% of m/z 69
70	<2% of m/z 69
127	40-60% of m/z 198 (10-80% for **EPA 525.2**)
197	<1% of m/z 198 (<2% for **EPA 525.2**)
198	100% (base peak)
199	5-9% of m/z 198
275	10-30% of m/z 198 (10-60% for **EPA 525.2**)
365	>1% of m/z 198
441	present but less than the intensity of m/z 443
442	>40% of m/z 198 (>50% of m/z 198 or base peak for **EPA 525.2**)
443	17-23% of m/z 442 (15-24% for **EPA 525.2**)

HIGH-RESOLUTION GAS CHROMATOGRAPHY HIGH-RESOLUTION MASS SPECTROMETRY (HRMS)

REQUIRED REAGANTS & STANDARDS

EPA 613 (GC-MS or HRMS)

Methylene Chloride extraction solvent
Sodium Sulfate drying reagent
n-Hexane exchange solvent
TCDD Internal Standard, isotopically labeled (**EPA 613**)

EPA 613, 1613, 1668, 8280, 8290 (GC-MS)

Isotopically labeled C-13 surrogate stds. & internal std., plus Cl-37 cleanup stds.
Perfluorokerosene, for calibrating the resolution of the MS & for use as the lock-mass
Methylene Chloride extraction solvent
Sodium Chloride drying agent
Toluene solvent for Soxhlet Dean-Stark extractions of solids & nonaqueous liquids
1:1 Methylene Chloride / Hexane for extracting tissue samples
Nonane (EPA 1613, 1668, 8290), Tetradecane (EPA 613), or Tridecane (EPA 8280) as final exchange solvents

EPA 613, 1613, 8280, 8290; EPA 3610, 3611 prior to EPA 8270

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 & Dibenzofurans 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 1613, 1668

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 613, 1613, 1668, 8280, 8290

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 613, 625, 1613, 1625, 1668, 8280, 8290; EPA 3650 prior to EPA 8270

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 1613, 1668; EPA 3640 prior to EPA 8270

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

EPA 1613, 1668, 8280, 8290

Activated Carbon Clean-up Sorbent
Conditioning Reagents (5% Toluene & 20% CH3OH in CH2Cl2, 1:1 CH2Cl2/Cyclohexane, & Hexane)
Hexane, 1:1 CH2Cl2/Cyclohexane, & 5% Toluene/20 % CH3OH in CH2Cl2, to elute impurities
Toluene, reverse-flow, to elute Dibenzo-p-dioxins & Dibenzofurans

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

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

Dibenzo-p-dioxins & Dibenzofurans (solids, mixed-phase, & tissues for CWA)

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

PCB's

30 days to Extract Sample, 45 Days to Analyze Extract; glass container with Teflon-lined cap; 4 C; 0.008% Sodium Thiosulfate

Dibenzo-p-dioxins & Dibenzofurans (RCRA aqueous-phase samples)

1-Year Holding Time, glass container with Teflon-lined cap, 4 C, 0.008% Na2S2O3

Dibenzo-p-dioxins & Dibenzofurans (aqueous-phase samples for CWA)

1-Year Holding Time, glass container with Teflon-lined cap, frozen <-10 C

Dibenzo-p-dioxins & Dibenzofurans (solids, mixed-phases, & tissues for CWA)

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

2,3,7,8-TCDD (Dioxin)	30 pg/L

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank

EPA 613, 7.1, response factors (if used) < 10% RSD

5 standards + blank

EPA 8280, 7.13.3.1-7.13.3.4, relative response factors < 15% RSD

EPA 8290, 7.7, response factors < 20% RSD relative to internal stds., internal stds. < 30% RSD relative to Surrogate stds.

EPA 1613, 10.5-10.6, relative response factor (if used) < 20% RSD relative to the isotope-labeled analog, < 35% RSD relative to sample matrix internal standard

EPA 1668, 10.4, labeled compound calibration factor (if used) < 20% RSD

EPA 680, 7.14 & 9.2.8, relative response factors < 20% RSD

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 70-130%

EPA 8280, 7.13.3.6

Recovery 80-120%

EPA 680, 9.3.7

EPA 8290, 7.7, plus 70-130% recovery for internal standards

Recovery 85-115%

EPA 613, 7.1.3

Recovery within the Test Method QC Acceptance Criteria

EPA 1613, 1668, 15.3

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

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

EPA 613, 8.1.1

EPA 1613, 1668, 9.2

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

QUALITY 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 1613, 15.5

EPA 1668, 9.3

Duplicate Precisions within 20%

EPA 8290, 8.3.6, precision within 25% for sample duplicate rather than Matrix Spike duplicate

Internal Standards Responses

EPA 680, 9.3.6, decrease < 30% from last Calib. Verification & < 50% from last Initial Calibration

EPA 1613, 9.3, serves as Matrix Spike since labeled stds. added to each sample, acceptance criteria in Table

ADDITIONAL REQUIREMENTS

All samples spiked with Isotopically Labeled Compounds
EPA 1613, 1668, 9.3

Matrix Spike & Matrix Spike Duplicate each batch of 20 samples or fewer
EPA 680, 10.7
EPA 8000, 8.5, applies to all 8000-series Organics mtds., may use sample dup. in place of MSD

Matrix Spike & Quality Control Check Samples analyzed every 10 samples
EPA 613, 8.1.4 & 8.4

Both Matrix Spike Duplicates & Sample Duplicates analyzed each sample batch
EPA 8290, 8.3.5-8.3.6

Method Blank analyzed every 20 samples or each 8 or 12-hour work shift, per matrix
EPA 1613, 9.5
EPA 8280, 7.1-7.2
EPA 8290, 8.2

GC Injector Port Degradation < 10% for Endrin & < 5% for DDT
EPA 680, 9.2.5.1 & 9.2.5.3, when Pesticides are determined

GC Retention Time Windows established for each analyte
EPA 680, 9.2.9, within 10 sec between analyses
EPA 1613, 15.4.1, acceptance limits for each target analyte given in Table
EPA 1668, 10.1.2
EPA 8000, 7.6

Chromatographic Resolution Checks
EPA 1613, 15.4.2, Tetra-dioxin & Tetra-furan isomers
EPA 8280, 7.12; 2,3,7,8-TCDD & 1,2,3,4-TCDD
EPA 8290, 8.2.1; 2,3,7,8-TCDD & all other TCDD's

Mass Spectrometer Resolution > 10000
EPA 1613, 10.1.2 & **EPA 8290**, 8.2.2
EPA 1668, 10.2

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

Criteria for Qualitative Identification of Dibenzo-p-dioxins & Dibenzofurans (EPA 1613, 1668, 8280, 8290)
GC retention times for target analytes agree match the retention times for the isotopically labeled analogs
GC retention times for target analytes in samples match the retention times in the CCV std.
GC retention times for target analytes in calibration verification std. match the retention times in the GC column performance check solution
MS ion current responses for both quantitation mass ions of target analyte reach maximum simultaneously
MS ion current responses for both quantitation mass ions of each isotopically labeled std. reach maximum simultaneously
Integrated peak areas for both quantitation mass ions meet isotope abundance ratio criteria
Signal-to-noise ratio for GC peaks exceeds 2.5 (10 for EPA 1613)
No GC peaks due to polychlorinated diphenyl ethers are observed

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)
LIQUID CHROMATOGRAPHY / MASS SPECTROMETRY (LC/MS)

REQUIRED REAGANTS & STANDARDS

EPA 532 (HPLC-UV) (all HPLC stationary phases are nonpolar reverse-phase, unless otherwise indicated)

Reverse-phase C-18 solid-phase disks or cartridges
Ethyl Acetate, Methylene Chloride, Methanol solid-phase conditioning reagents
Methanol eluting solvent
Sodium Sulfate drying reagent
Reverse-phase C18 primary column and Cyano-C18 confirmation column; PO4 buffer mobile phase

EPA 531.1, 531.2 (HPLC-Fluorescence)

o-Phthalaldehyde & 2-Mercaptoethanol post-column derivatizing agents (prepared fresh daily)
Sodium Hydroxide post-column hydrolysis reagent
Monochloroacetic Acid to adjust sample pH to 2.8-3.2 prior to analysis (EPA 531.1)
Potassium Dihydrogen Citrate & Sodium Thiosulfate to adjust sample pH around 3.8 (EPA 531.2)
Methanol & water mobile phases, gradient elution (15% to 100% or 10% to 80% Methanol, depending on column (EPA 531.1); Methanol-Acetonitrile-Water 1:0:7 to 1:1:6 to 2:2:4 (EPA 531.2))
Excitation wavelength 330 or 340 nm, fluorescence emission wavelength 418 or 465 nm

EPA 547, SM6651B (HPLC-Fluorescence)

Cation or anion exchange resin stationary phase
o-Phthalaldehyde & 2-Mercaptoethanol post-column derivatizing agents
Calcium Hypochlorite post-column oxidizing agent
4% Methanol / 5 mM pH 1.9 Phosphate buffer mobile phase, degassed with helium

EPA 549.2 (HPLC-UV, 308 nm for Diquat)

C-8 solid-phase extraction cartridge or disk
Methanol, water Cetyl Trimethylammonium Bromide, water, Methanol, water, 1-Hexanesulfonate (Na salt) to condition cartridge
Methanol, water, Cetyl Trimethylammonium Bromide, water, 1-Hexanesulfonate (Na salt) to condition disk
Phosphoric Acid / Diethylamine eluting solution
1-Hexanesulfonic Acid ion-pair concentrate
Phosphoric Acid / Diethylamine / 1-Hexanesulfonic Acid mobile phase

EPA 550 (HPLC-UV & Fluorescence)

Methylene Chloride extraction solvent
Sodium Sulfate drying agent
Acetonitrile exchange solvent
Acetonitrile & water mobile phases, gradient elution (35% to 100% Acetonitrile)

EPA 550.1 (HPLC-UV & Fluorescence)

Reverse-phase C-18 solid-phase extraction disks or cartridges
Methylene Chloride & Methanol to condition disks or cartridges
Methylene Chloride eluting solvent (cartridges), or Acetonitrile then Methylene Chloride (disks)
Sodium Sulfate drying agent
Acetonitrile exchange solvent
Acetonitrile & water mobile phases, gradient elution (35% to 100% Acetonitrile)

EPA 555 (HPLC-UV diode array)

Sodium Hydroxide hydrolysis reagent
Phosphoric Acid to adjust sample pH<2 after hydrolysis
C-18/silica concentrator cartridge + analytical column (solid-phase extraction may also be used)
Acetonitrile / Phosphate buffer mobile phase, gradient elution (10% to 90% Acetonitrile)

EPA 605 (HPLC-electrochemical detection)

Chloroform extraction solvent
Sulfuric Acid for back-extraction clean-up
Methanol exchange solvent
1:1 Acetonitrile/Acetate buffer mobile phase

EPA 629, 631 (HPLC-UV)
EPA 3510, 3520, 3540, 3541 with EPA 8321 & 8325 (LC-MS)

Sulfuric Acid hydrolysis reagent (**EPA 631**, hydrolyzes Benomyl to Carbendazim at pH<1)
Sodium Hydroxide (**EPA 631**, neutralize sample pH to 6-8, must determine Benomyl by difference)
Methylene Chloride extraction solvent
Sodium Sulfate drying agent
Methanol exchange solvent
Surrogate standards (**EPA 8000's**)

EPA 610, SM6440B (GC-FID or HPLC-UV & Fluorescence); 632, 637, 639 (HPLC-UV)
EPA 3510, 3520, 3540, 3541 with EPA 8100 (GC-FID) or EPA 8310 (HPLC-UV & Fluorescence)

Methylene Chloride extraction solvent
Sodium Sulfate drying reagent
Acetonitrile exchange solvent (HPLC only)
Surrogate standards (EPA 8100, 8310)

EPA 632.1 (HPLC-UV)

Methylene Chloride extraction solvent
Sodium Sulfate drying agent
1:1 Acetonitrile/water exchange solvent

EPA 1660 (HPLC-UV)

Acetonitrile extraction solvent
Mobile Phase gradient elution (30% Acetonitrile in water to 100% Acetonitrile)

EPA 8315 (HPLC-UV)

Acid to adjust sample pH to 3
Dinitrophenylhydrazine (DNPH) pre-column derivatizing agent
Methylene Chloride extraction solvent
Sodium Sulfate drying agent
Acetonitrile exchange solvent
Mobile Phase Gradient elution (60 or 70% Acetonitrile in water to 100% Acetonitrile)

EPA 8318 (HPLC-Fluorescence)

o-Phthalaldehyde & 2-Mercaptoethanol post-column derivatizing agent
Methylene Chloride extraction solvent
Sodium Sulfate drying agent
Methanol exchange solvent
Reverse-phase C-18 cleanup cartridge
Mobile Phase Gradient Elution (10% CH3OH/CH3CN in H3PO4/H2O to 100% Methanol/Acetonitrile)

EPA 8330 (HPLC-UV)

Acetonitrile extraction solvent
Sodium Sulfate drying agent
1:1 Acetonitrile/water exchange solvent
Cyano-C18 confirmation HPLC column required along with the reverse-phase column
1:1 Methanol/water mobile phase

EPA 8331, 8332 (HPLC-UV)

EPA 8331 Soil Extraction Solvent & Mobile Phase: 1-Decanesulfonic Acid in Acetic Acid, Methanol, & H2O
EPA 8332 Mobile Phase: 60% Acetonitrile in water

EPA 8321 (LC-MS thermospray) & EPA 8325 (LC-MS particle beam)

Ethyl Ether extraction solvent (EPA 8321)
Sodium Sulfate drying agent (EPA 8321)
Acetonitrile exchange solvent (EPA 8321)
MS Tuning Solution (Decafluorotriphenylphosphine Oxide) (EPA 8325) (**daily**)
Surrogate standards
Mobile Phase Gradient Elution
(50% Methanol in water to 100% Methanol, for Organophosphorus Pesticides)
(50% Acetonitrile in water to 100% Acetonitrile, for Azo Dyes)
(25% to 60% Methanol in Ammonium Acetate Buffer, for Chlorinated Phenoxyacids)
(5% Methanol in Ammonium Acetate Buffer to 100% Methanol, for Carbamates)
(25% to 70% Acetonitrile in Ammonium Acetate Buffer, for Benzidines & Organonitrogen Pest.)

EPA 3545 with EPA 8310, 8325

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 8310, 8325

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 3561 with EPA 8310

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 8310, 8325

Waste Dilution Solvents: Methylene Chloride or Hexane

EPA 3610, 3611 prior to EPA 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 & Dibenzofurans 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 610; EPA 3630 prior to EPA 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 3640 prior to EPA 8310

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

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

7 Days to Extract Sample, 7 Days to Analyze Extract; Teflon-lined cap; 4 C; 0.008% Sodium Thiosulfate
Benzidines (analyze extract immediately if not stored in oxygen-free system)

7 Days to Extract Sample, 21 Days to Analyze Extract; Amber PVC or Silanized Amber Glass container; 4 C
SDWA Diquat

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)

7 Days to Extract Sample, 40 Days to Analyze Extract; glass containers with Teflon-lined cap; 4 C; pH 5-9
Pesticides (CWA), Organophosphorus Pesticides (RCRA)

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

14 Days to Extract Sample, 14 Days to Analyze Extract; 4 C
SDWA Chlorinated Solvents & Disinfection By-Products

14 Days to Extract or Analyze Sample, 4 C
Other SDWA Pesticides & PCB's

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 531.1, 10.2 & 10.3, calibration factor or response factor (if used) < 20% RSD
EPA 547, 9.2
EPA 549.2, 10.3
EPA 550 & 550.1, 9.2 & 9.3, calibration factor or response factor (if used) < 10% RSD
EPA 555, 10.2, calibration factor (if used) < 20% RSD
EPA 605, 610, 7.2 & 7.3, calibration or response factors (if used) < 10% RSD
EPA 629, 631, 632, 637, 639, 7.2 & 7.3, calibration factors or response factors (if used) < 10% RSD
EPA 632.1, 8.2, calibration factors (if used) < 10% RSD
EPA 1660, 7.3, calibration factor (if used) < 20% RSD

5 standards + blank

EPA 8000, 7.4-7.5, calibration factor or response factor (if used) < 20% RSD, correlation coefficient >0.990 for non-linear calibration
Applies to **EPA 8310, 8315, 8316, 8318, 8321, 8325, 8330, 8331, 8332** (HPLC Organics)
Requires **client notification of analytes** quantitated from CF or RF when mtd. criteria not met (and mean RSD < 20%)
EPA 531.2, 532, 10.2, calibration factor (if used) < 30% RSD

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 70-130%

EPA 531.2, 532, 10.3, 50-150% allowed for low-level stds.; CCV also required every 10 samples & end of run

Recovery 75-125%

EPA 555, 10.2.3

Recovery 80-120%

EPA 531.1, 10.2.4
EPA 547, 550, & 550.1, 9.4
EPA 549.2, 10.4
EPA 8325, 7.4
SM6020B, 1b (applies to SM Organics methods)
40 CFR 141.40, App. A, 3, mid-range std. **plus** 60-140% recovery for std. at or below Minimum Reporting Level (applies to SDWA Unregulated Contaminants)

Recovery 85-115%

EPA 8000, 7.7

Recovery 90-110%

EPA 629, 631, 632, 637, 639, 7.2 & 7.3
EPA 632.1, 8.2
EPA 8331, 7.3.2

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

Method Detection Limit required for each analyte

EPA 549.2, 9.3

Mean Accuracy 70-130% for each analyte

EPA 555, 9.3
EPA 547, 10.3.2, Precision RSD < 30% from mean

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

EPA 531.1, 9.3, MDL determination also required
EPA 531.2, 532, 9.2, MDL study also required & at least 3 days required (not all aliquots extracted same day)

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

EPA 605, 610, 8.2
EPA 629, 631, 632, 637, 639, 8.2
EPA 1660, 8.2
EPA 8000, 8.6, applicable to all **EPA 8000-series** methods

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample Recoveries within 70-130%

EPA 532, 9.6, 50-160% allowed for low-level concentrations
EPA 550 & 550.1, 10.5.1

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

EPA 605, 610, 8.4
EPA 1660, 8.3

External QC Check Sample Analyzed Quarterly

EPA 531.2, 9.11, recoveries 70-130%
EPA 555, 9.5

Matrix Spike Recoveries within 50-150% or 50-160%

EPA 531.2, 9.8, 70-130% recommended for mid- & high-level spikes, analyzed each analytical batch
EPA 532, 9.9, 70-130% recommended for mid- & high-level spikes, analyzed each extraction batch

Matrix Spike Recoveries 65-135%

EPA 531.1, 9.7, analyzed every 20 samples or batch

Surrogate Recoveries within 70-130%

EPA 531.2, 532, 9.7

Internal Standards Responses

EPA 531.1, 9.5, 70-130% from last Calibration Verification
EPA 550, 550.1, 10.4, 70-130% from last Calib. verification

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

SDWA MAXIMUM CONTAMINANT LEVELS

Benzo(a)pyrene	0.2 ug/L
2,4-D	70 ug/L
Pentachlorophenol	1.0 ug/L
2,4,5-TP (Silvex)	50 ug/L
Dalapon	200 ug/L
Dinoseb	7.0 ug/L
Picloram	500 ug/L
Diquat	20 ug/L
Endothall	100 ug/L
Glyphosate	700 ug/L
Carbofuran	400 ug/L
Oxamyl (Vydate)	200 ug/L

RCRA TOXICITY CHARACTERISTICS

2,4-D	10.0 mg/L
2,4,5-TP (Silvex)	1.0 mg/L

ADDITIONAL REQUIREMENTS

Matrix Spikes analyzed every 10 samples

EPA 549.2, 9.6
EPA 547, 550, 550.1, 10.6
EPA 605, 610, 8.3
EPA 629, **631, 632, 637, 639**, 8.4, or Monthly
EPA 1660, 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

Quality Control Check Samples analyzed every 10 samples

EPA 605, 610, 8.1.5, frequency may be reduced if Matrix Spike recoveries meet all specified QC criteria
EPA 632.1, 9.2

Field Duplicates analyzed

EPA 531.2, 9.9, each batch unless MSD analyzed, <50% RSD for low-level, <30% recommended for mid & high level
EPA 532, 9.10, each extraction batch unless MSD analyzed
EPA 632.1, 9.3, ALL samples are collected in duplicate; 10% are analyzed

Peak Gaussian Factor evaluated each analytical batch

EPA 532, 10.2.3, for Flumeturon

GC Retention Time Windows established for each analyte

EPA 8000, 7.6

Chromatographic Resolution Checks

EPA 531.2, 9.10
EPA 8325, 7.3.2; 3,3'-Dimethylbenzidine & 3,3'-Dimethoxybenzidine

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

PESTICIDES GAS CHROMATOGRAPHY (GC)

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

REQUIRED REAGANTS & STANDARDS

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

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

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

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

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

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

EPA 508A (GC-ECD Methylene Chloride extraction solvent
Sodium Sulfate & Sodium Bicarbonate drying agents
Antimony Pentachloride perchlorination reagent & Iron catalyst (converts PCB's to decachlorobiphenyl)
1:1 Hydrochloric Acid quenching reagent
n-Hexane final extraction solvent

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

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

EPA 515.2 (GC-ECD)

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

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

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

EPA 551.1 (GC-ECD)

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

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

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

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

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

EPA 622.1, 633.1, 634 (GC-NPD)

Methylene Chloride extraction solvent
Sodium Sulfate drying reagent

EPA 630.1 (GC-ELCD)

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

EPA 633 (GC-NPD or MS)

Methylene Chloride extraction solvent
Sodium Sulfate drying agent
Acetone exchange solvent

EPA 3545 with EPA 8081, 8082, 8141

Pressurized Extraction Fluids: 1:1 Methylene Chloride/Acetone, 1:1 Hexane/Acetone, Hexane, or CH2Cl2
Note: Exchange solvents are based on clean-up method & determinative methods employed

EPA 3550 with EPA 8081, 8082, 8141

Ultrasonic Extraction Fluids: 1:1 Methylene Chloride/Acetone, 1:1 Hexane/Acetone, or Hexane
Note: Exchange solvents are based on clean-up method & determinative methods employed

EPA 3580 with EPA 8081, 8082, 8141, 8151

Waste Dilution Solvents: Methylene Chloride or Hexane

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

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

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

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

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

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

EPA 3630 prior to EPA 8082

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

EPA 3640 prior to EPA 8081

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

EPA 608; EPA 3660 prior to EPA 8081

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

EPA 3665 prior to EPA 8082

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

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

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

Organochlorine Pesticides (RCRA), PCB's

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

Pesticides (CWA), Organophosphorus Pesticides (RCRA)

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

SDWA Chlorinated Phenoxy Acids (Herbicides)

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

SDWA Chlorinated Solvents & Disinfection By-Products

14 Days to Extract or Analyze Sample, 4 C

Other SDWA Pesticides & PCB's

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

SDWA Haloacetic Acids

INITIAL INSTRUMENT CALIBRATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

3 standards + blank

SM6020B, 1a & 1b, applies to all SM Organics mtds., int. std. response factor (if used) < 20% RSD
EPA 505, 10.2, calibration factor (if used) < 20% RSD
EPA 507, 508, 10.2 & 10.3, calibration factor or response factor (if used) < 20% RSD
EPA 515.1, 9.2, calibration factor or response factor (if used) < 20% RSD
EPA 515.2, 10.2, response factor (if used) < 30% RSD
EPA 552.1, 10.2, each standard processed & analyzed in triplicate, internal standard technique required
EPA 608, 608.1, 614, 617, 619, 622, 622.1, 627, 629, 631, 632, 633, 633.1, 634, 637, 7.2 & 7.3, calibration factors or response factors (if used) < 10% RSD
EPA 608.2, 614.1, 645, 8.1, calibration factors (if used) < 10% RSD
EPA 615, 7.2, calibration factors (if used) < 10% RSD
EPA 630.1, 7.3, calibration factors (if used) < 10% RSD
EPA 632.1, 8.2, calibration factors (if used) < 10% RSD
EPA 1656, 1657, 1658, 1659, 7.3, calibration factor (if used) < 20% RSD
EPA 1661, 7.3, calibration factor (if used) < 15% RSD
D5317-93, 11.2 & 11.3, calibration factor or response factor (if used) < 20% RSD
3M0222, 10.2 & 10.3, calibration factor or response factor (if used) < 10% RSD
O-3106-93, 6.15, performed **daily**

4 standards + blank

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

5 standards + blank

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

6 standards + blank

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

CALIBRATION VERIFICATION ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

Recovery 70-130%

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

Recovery 80-120%

EPA 507, 508, 515.1, 10.2.4
EPA 508A, 9.2, calibration verification performed at end of sample batch despite int. std. technique used
EPA 1656, 1657, 1658, 7.4, for resolved components on the GC
SM6020B, 1b (applies to SM Organics methods)
D5317-93, 11.2 & 11.3
40 CFR 141.40, App. A, 3, mid-range std. **plus** 60-140% recovery for std. at or below Minimum Reporting Level (applies to SDWA Unregulated Contaminants)

Recovery 75-125%

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

Recovery 85-115%

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

Recovery 90-110%

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

Recovery within the Test Method QC Acceptance Criteria

EPA 1656, 13.5

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

Method Detection Limit required for each analyte

EPA 508.1, 9.3

Mean Accuracy 60-140% for each analyte

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

Mean Accuracy 70-130% for each analyte

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

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

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

Mean Accuracy 80-120% for each analyte

EPA 515.3, 9.3

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

EPA 548.1, 9.3.2

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

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

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

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

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

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

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

EPA 608, 608.1, 614, 615, 617, 619, 622, 622.1, 627, 630, 630.1, 633, 633.1, 634, 637, 8.2
3M0222, 9.2
EPA 1656, 1657, 1658, 1659, 1661, 8.2
EPA 8000, 8.6, applicable to all **EPA 8000-series** methods

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

QC Check Sample Recoveries within 70-130%

EPA 508A, 10.6
EPA 508.1, 9.6
EPA 552.1, 9.6

QC Check Sample Recoveries within 80-120%

EPA 548.1, 9.6.2

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

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

External QC Check Sample Analyzed Quarterly

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

External QC Check Sample Recoveries within 70-130%

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

External QC Check Sample Recoveries within 80-120%

EPA 508A, 10.8, analyzed quarterly

Matrix Spike Recoveries 50-150%

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

Matrix Spike Recoveries 65-135%

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

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

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

Matrix Spike Recoveries within 75-125%

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

Matrix Spike Recoveries within 80-120%

EPA 548.1, 9.7.1 (Endothall)

Surrogate Recoveries within 40-120%

EPA 1656, 1657, 1658, 8.3

Surrogate Recoveries within 60-140%

EPA 515.2, 9.5

Surrogate Recoveries within 70-130%

EPA 507, 508, 9.5
EPA 515.1, 10.5
EPA 515.3, 515.4, 552.2, 9.8
EPA 552.1, 9.4
EPA 552.3, 9.7
D5317-93, 19.5

Surrogate Recoveries 80-120%

EPA 551.1, 9.8

Analyte Concentrations in Blank

EPA 508A, 10.1, <0.025 ug/L for Decachlorobiphenyl

Internal Standards Responses

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

EPA REGULATORY LEVELS REQUIRING SPECIFIC DETECTION LIMITS

RCRA TOXICITY CHARACTERISTICS

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

SDWA MAXIMUM CONTAMINANT LEVELS

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

ADDITIONAL REQUIREMENTS

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

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

Matrix Spikes analyzed every 10 samples

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

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

40 CFR 141.40, App. A, 6, must also **alternate between mid- & low-level** concentrations for spikes (applies to SDWA Unregulated Contaminants)
EPA 515.4, 9.9
EPA 8000, 8.5, applies to all 8000-series Organics mtds., may use sample dup. in place of MSD

Matrix Spike every 20 samples

EPA 508A, 10.7

Quality Control Check Samples analyzed every 10 samples

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

Field Duplicates analyzed

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

Reagent Blank analyzed every 10 samples

EPA 508A, 10.1

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

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

GC Injector Port Degradation < 15% for Endrin & DDT

EPA 8081, 8.3

GC Retention Time Windows established for each analyte

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

Chromatographic Resolution Checks

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

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

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

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

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