NATIONAL ENVIRONMENTAL LABORATORY ACCREDITATION CONFERENCE (NELAC)

ON-SITE LABORATORY ASSESSMENT

AIR SAMPLING AND TESTING CHECKLIST (22 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.

Notes:

The checklist for environmental laboratory **compliance with NELAC Standards** is found on **pages 3-12**

Photocopy pages 5-12 as necessary to assess **each test method** for compliance with the NELAC Standards

To assist the laboratory assessors on-site at the laboratory, **pages 13-22** have been devised to provide a **quick reference**, since the Fields of Testing for Air Sampling and Analysis are not as commonly known relative to testing for waters and solids

The quick reference **does not substitute** for **qualifications & training** required of on-site laboratory assessors under NELAC

Pages 13-17 contain the requirements for **essential equipment, chemical reagents, & analysis conditions** for mandated test methods, in terms of both composition & shelf-life

Holding Times, Containers, & Preservation requirements for samples analyzed for particular contaminants are found on **page 19**

Initial Instrument Calibration & Calibration Verification requirements & acceptance criteria for mandated test methods are found on **pages 18-19**

Requirements for **Initial Demonstration of Capability** in mandated test methods are found on **page 19**

Pages 20-22 contain requirements in mandated test methods for **sample batch acceptability** in terms of **requirements for Quality Control samples** & requirements necessary for the **satisfactory performance** of **mandated test methods**

40 CFR Part 50 mandates use of **Reference Methods** within, or based on procedures within, the **Appendices to this Part**
40 CFR Part 60 mandates the use of **Reference Methods** in the **40 CFR Part 60 App. A**
40 CFR Part 60.13(a) mandates the use of **Performance Specifications** in **40 CFR Part 60 App. B**
40 CFR Part 61 mandates the use of test methods in **40 CFR Part 61 App. B (EPA 100-series)**
40 CFR Part 63 mandates the use of test methods in the **Appendices of 40 CFR Parts 51 (EPA 200-series), 60, 61, & 63 (EPA 300-series)**
40 CFR Part 266.104(e)(1) mandates the use of **EPA 0023A**
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**

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)

AIR TESTING LABORATORY TOUR

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

IP-10A – weighing room temp. 20 +/- 3 C, 38-42% rel. humidity

____ **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 0020 – heated Source Assessment Sampling probe & cyclone/filter holder at 400 +/- 36 F (204 +/- 20 C)
EPA 0040 – sample probe outlet & heated filter holder at 130-140 C
Methylene Chloride Solvent Evaporation – 60-70 degrees Celsius
n-Hexane Solvent Evaporation – 85-90 degrees Celsius

____ **D.5.3(c)** Does the laboratory use the results from **analyzing proficiency test samples** to evaluate its **ability to produce accurate data**

____ **D.5.5** Has the laboratory **documented procedures for data reduction** (e.g. use of linear regression)

____ **D.5.6(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.5.6(b)** Does the laboratory document the purity of each analyte standard & each reagent through **analysis certificates** from the manufacturer, **vendor specifications**, and/or **independent analysis**

____ **D.5.6(c)** Does the laboratory **use analytical reagent grade** materials when the purity of reagents is not specified in the test method

____ **5.5.6.4(d)** Do all containers of prepared reagents & standards have a **unique identifier & expiration date** that **links** these specific containers of reagents & media to their preparation records
Note: See pages 13-17 for the reagents & standards required for each test method, along with the required purities & shelf-life

____ **5.5.6.4(e)** Are procedures in place to ensure that prepared reagents **meet the requirements of the test method** (see the scientific discipline & technology checklists for specific requirements)
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

____ **D.5.7** Has the laboratory **developed & documented** criteria for **test method selectivity** such as absolute & relative retention times, wavelength assignments, mass spectral library quality of match, & mass spectral tuning

____ **D.5.8(a)** Does the laboratory's **test instruments** consistently **operate within the specifications** required of the applications for which they are used

____ **D.5.8(b)** Does the laboratory **document** that all sampling equipment, containers, & media used or supplied by the laboratory **meet required test method criteria**

____ **D.5.8(c)** Has the laboratory developed procedures for **field equipment decontamination** & documented their **use**, if field equipment is supplied or used by the laboratory

____ **D.5.8(d)** Does the laboratory have a documented program for **calibration & verification of sampling equipment** such as pumps, meter boxes, critical orifices, flow measurement devices, & continuous analyzers, if these equipment are used or supplied by the laboratory

____ **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 page 19 for holding times, sample containers allowed, & preservation required for each analyte

COMMENTS: List any test instruments, support equipment, & laboratory work areas that do not meet the above Standards

AIR ANALYSIS SAMPLING & TEST METHODS

TEST METHOD EVALUATED: _____

____ **5.5.4.1.2(a)** Does the laboratory have an **in-house methods manual** for each accredited **analyte** or **method**
Note: This manual may consist of copies of published or referenced test methods

____ **5.5.4.1.2(b)** Does the laboratory **clearly indicate** in its methods manual **any modifications** made to the referenced test method and **describe any changes or clarifications** where the referenced test method is ambiguous or provides insufficient detail

Does each test method in the in-house methods manual include or reference, where applicable:

____ **5.5.4.1.2(b)(1)** **Identification** of the test method
____ **5.5.4.1.2(b)(2)** Applicable **matrix or matrices**
____ **5.5.4.1.2(b)(3)** **Method Detection Limit**
____ **5.5.4.1.2(b)(4)** **Scope & application**, including components to be analyzed
____ **5.5.4.1.2(b)(5)** **Summary** of the test method
____ **5.5.4.1.2(b)(6)** **Definitions**
____ **5.5.4.1.2(b)(7)** **Interferences**
____ **5.5.4.1.2(b)(8)** **Safety**
____ **5.5.4.1.2(b)(9)** **Equipment & supplies**
____ **5.5.4.1.2(b)(10)** **Reagents & standards**
____ **5.5.4.1.2(b)(11)** Sample **collection, preservation, shipment, & storage**
____ **5.5.4.1.2(b)(12)** **Quality control**
____ **5.5.4.1.2(b)(13)** **Calibration & standardization**
____ **5.5.4.1.2(b)(14)** **Procedure**
____ **5.5.4.1.2(b)(15)** **Calculations**
____ **5.5.4.1.2(b)(16)** Method **performance**
____ **5.5.4.1.2(b)(17)** Pollution **prevention**
____ **5.5.4.1.2(b)(18)** **Data assessment & acceptance criteria** for quality control measures
____ **5.5.4.1.2(b)(19)** **Corrective actions** for out-of-control data
____ **5.5.4.1.2(b)(20)** Contingencies for **handling out-of-control or unacceptable data**
____ **5.5.4.1.2(b)(21)** **Waste management**
____ **5.5.4.1.2(b)(22)** **References**
____ **5.5.4.1.2(b)(23)** **Tables, diagrams, flowcharts, validation data**

____ **D** Does the laboratory ensure that the **essential standards** outlined in Appendix D are incorporated into the method manuals and/or Quality Manual

____ **D** Does the laboratory's data indicate that the **quality control protocols** specified in its test method manual **are being followed**

____ **D** Does the laboratory have **procedures** for developing **acceptance/rejection criteria** for each Air Toxics test method

COMMENTS:

TEST METHOD EVALUATED: _____

Note: The following Standards for initial instrument calibration & calibration verification are also referenced in NELAC **D.5.3(b)**

____ **5.5.5.2.2** Do the laboratory's initial & continuing instrument calibration verifications meet the requirements in **mandated test methods & regulations** (see pages 18-19 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

AIR 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

AIR 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:

TEST METHOD EVALUATED: _____

____ **5.5.4.2.2(a)** Has the laboratory performed a **satisfactory demonstration of method capability** prior to the acceptance & institution of this test method
C.1
D.5.3(a)
Note: Demonstrations of capability are done in an applicable & available **clean matrix sample** in a 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 matrix** sufficient to prepare **4 aliquots** at the **specified concentration** or to a concentration approximately **10 times** the method-stated or laboratory-determined **method detection limit**

____ **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 reporting 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)
- Another **demonstration of capability or initial measurement system evaluation**
- Successful performance of a blind performance sample on a **similar test method** using the **same technology** (acceptance criteria must be determined prior to analysis)
- At least **4 consecutive** laboratory **control samples** with **acceptable levels** of precision & accuracy (acceptance criteria must be determined prior to analysis)
- Analysis of **authentic samples** that have been analyzed by **another trained analyst** with **statistically identical 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 all associated supporting data** necessary to **reproduce the analytical results** summarized in the appropriate certification statement

____ **5.5.4.2.2(e)** **C.1** **D.5.3(a)** Does the laboratory **complete a demonstration of capability each time** there is a **change** in **instrument type, personnel, quality system matrix, 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 19 for such Demonstration of Capability procedural requirements & acceptance criteria

TEST METHOD ASSESSED: _____

____ **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**
Note: These Standards do not apply to field activities such as source air emission measurements or the use of continuous analysis devices

____ **D.5.1(a)(1)** Does the laboratory analyze **method blanks** at a frequency of at least **one per sample batch of 20 or less environmental samples per sample preparation** method

____ **D.5.1(a)(1)** Does the laboratory **investigate the source of contamination** & take measures to **correct, minimize, or eliminate the problem** if the method blank result **exceeds the limit of detection & exceeds 10% of total analyte amount** in the sample

____ **D.5.1(a)(1)** Are **all samples** associated with a **contaminated blank reported** with appropriate **data qualifying codes**

____ **D.5.1(a)(1)** Is the method blank used to **evaluate contribution** of laboratory-provided sampling media & analytical sample preparation procedures to the amount of analyte found in each sample

____ **D.5.1(a)(2)** Does the laboratory assess **collection efficiency** if required by the client; by separating sampling trains of multiple sections (e.g., filters, sorbent tubes, & impingers) into front & back sections, with each section processed, analyzed, & results reported separately

____ **D.5.1(b)(1)** Does the laboratory analyze at least **one laboratory control sample** (QC Check Sample) per batch of **20 or less samples per sample preparation** method for **each analyte**

____ **D.5.1(b)(1)** If a spiking solution is not available, does the laboratory use **calibration solution** whose concentration approximates that in samples with each batch & with each lot of media

____ **D.5.1(b)(1)** Does the laboratory **notify the client** prior to the start of the analysis if the calibration solution must be used for the laboratory control sample

____ **D.5.1(b)(1)** Is the laboratory control standard concentration **relevant** to the intended use of data & either **at or below a regulatory limit**

____ **D.5.1(c)** Does the laboratory use **surrogates as required by the test method** or if requested by the client

____ **D.5.1(d)** Does the laboratory perform **matrix spikes as required by the test method** or if requested by the client

____ **D.5.2** Does the laboratory analyze **matrix spike duplicates or laboratory duplicates** at a minimum frequency of **one in 20 samples per sample batch**

____ **D.5.2** Does the laboratory **document** its procedure for **selecting** the use of the **appropriate type of spikes & duplicates**

____ **D.5.2** Is the **selected sample** for the **duplicate rotated among client samples** so that various sample matrix problems may be noted and/or addressed

TEST METHOD EVALUATED: _____

____ **D.5.2** Does the laboratory **report poor performance** in a duplicate **to the client whose sample was used** for the duplicate

____ **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 20-22 for acceptance criteria

____ **5.1.1** Does the laboratory fulfill **additional requirements** specified in the **mandated test method** or **regulation**
Note: See pages 20-22 for the additional requirements stated in test methods

____ **D.5.4** **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.5.4** **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.5.4** **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.5.4** **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.5.4** **D.1.2.1(c)** Does the laboratory have **established procedures** to relate **LOD's with Limits of Quantitation (LOQ's)**

____ **D.5.4** **D.1.2.1(d)** Has the laboratory **verified the LOD annually** for each quality system matrix, test method, & analyte
Note: All sample processing steps of the analytical method must be included in the determination of the LOD
Note: Validity of the LOD is confirmed by **qualitative identification** of the analyte(s) in a quality control sample in each quality system matrix containing the analyte at **no more than 2-3x** the LOD for single-analyte tests and **1-4x** the LOD for multiple analyte tests
Note: LOD verification must be performed **on every instrument that is to be used** for analysis of samples & reporting of data
Note: A LOD study is not required for any component for which spiking solutions or quality control samples are not available (e.g., Temperature), or when test results are **not to be reported to the LOD** (versus the Limit of Quantitation or working range of instrument calibration according to Appendices D.1.2, D.4.5, D.5.4, and D.6.6 to NELAC Chapter 5).

COMMENTS:

REQUIRED EQUIPMENT, REAGANTS, & STANDARDS

Source Air Emissions Sampling Train Equipment (in order)

Pitot Tube & Differential Pressure Gauge - **EPA 0010, 0011, 0020, 0023A, 0040, 0050, 0051, & 0060**

Calibrated Sample Probe Nozzle & Heated Probe – **EPA 0010, 0011, 0023A, 0060, & 0061**

Sample Probe with Glass Wool Plug – **EPA 0030, & 0031**

Heated Sample Probe – **EPA 0040**

Calibrated GLASS Probe Nozzle & Heated Probe – **EPA 0050**

Heated GLASS Sample Probe – **EPA 0051 (probe nozzle turned upwards)**

Source Assessment Sampling Probe Nozzle – **EPA 0020**

Recirculatory Sample Train & Aspirator – **EPA 0061**

Glass Sample Probe Liner – **EPA 0010, 0011, 0020, 0023A, 0050, 0051, & 0060**
Note: Liner must be borosilicate or quartz glass; others (metal) require EPA written approval

Cyclone or Cyclone Train – **EPA 0020**

Glass- or Quartz-Fiber Filter & Heated Filter Holder – **EPA 0010, 0011, 0020, 0023A, 0040, 0050, 0051, & 0060**

Teflon Mat Filter & Heated Filter Holder – **EPA 0050 & 0051**

Condensers & Condensate Traps – **EPA 0020, 0030, 0031, 0040**

XAD-2 Adsorbent – **EPA 0010, 0011, 0020, & 0023A**

Tenax Adsorbent – **EPA 0030 & 0031**

Charcoal Adsorbent – **EPA 0030**

Anasorb Adsorbent – **EPA 0031**

Dinitrophenylhydrazine impregnated Silica Gel Adsorbent – **EPA 0100**

Condenser & Impinger Train Solutions – **EPA 0010, 0011, 0020, 0023A, 0050, 0051, 0060, & 0061**

Tedlar or Mylar Bag – **EPA 0040**

Silica Gel Drying Tube – **EPA 0040 (& charcoal) & 0051 (optional)**

Metering System

Rotameter, Vacuum Gauge, & Pump - **EPA 0030, 0031, 0040, & 0100**

Dry Gas Meter, Vacuum Gauge, Pump, & Thermometers –
EPA 0010, 0011, 0020, 0023A, 0030, 0031, 0040, 0050, 0051, 0060, 0061, & 0100

Orifice Plate, Manometer, & Barometer – **EPA 0010, 0011, 0020 (optional), 0023A, 0050, 0051, 0060, & 0061**

Gas Density Measuring Equipment – **EPA 0010, 0011, 0023A, 0050, 0051, 0060, 0061**

(General Chemistry)

EPA 0010, 0020

Impinger Solutions: Impingers 1 & 2: Water
Impinger 3: Empty
Impinger 4: Anhydrous Silica Gel

EPA 0011 - Formaldehyde

Impinger Solutions: Impingers 1, 2, & 3: 2,4-Dinitrophenylhydrazine
Impinger 4: Empty
Impinger 5: Anhydrous Silica Gel

EPA 0060 – Metals

Impinger Solutions: Impinger 1: Empty (moisture trap)
Impingers 2 & 3: 5% Nitric Acid & 10% Hydrogen Peroxide
Impinger 4: Empty
Impingers 5 & 6: 10% Sulfuric Acid & 4% Potassium Permanganate
Impinger 7: Anhydrous Silica Gel

EPA 0061 – Chromium(VI)

Impinger Solutions: Impingers 1, 2, & 3: 0.1 N Potassium Hydroxide
Impinger 4: Empty
Impinger 5: Anhydrous Silica Gel

TO-12 – Nonmethane Hydrocarbons (FID)

Stainless Steel, Summa-polished canister
Cryogenic focusing with liquid argon
Propane calibration std. in Air

TO-16 – FTIR Spectrometry

IP-3A (NDIR Spectrometry), IP-3B (Gas Filter Correlation Spectrometry), & IP-3C (Electrochemical Oxidation)

Carbon Monoxide calibration gas in Nitrogen

IP-5A – Nitrogen Dioxide (Chemiluminescence)

Luminol (produces chemiluminescence upon oxidation with NO2) (5-Amino-2,3-dihydro-1,4-phthalazine dione)
NO2 gas standard

IP-5B – Nitrogen Dioxide (UV-VIS)

Palmes Tube (NO2 adsorbed onto surface coated with Triethanolamine)
N-1-Naphthylethylenediamine Dihydrochloride color reagent (540 nm)
Sodium Nitrite standards

IP-5C – Nitrogen Dioxide (Ion Chromatography)

Glass Fiber Filter coated with Triethanolamine
Sodium Carbonate & Sodium Bicarbonate mobile phase & Anion Exchange Column
Potassium Nitrate standard

IP-6B – Formaldehyde (UV-VIS Spectrophotometry)

Scrubbing Solution: Sodium Tetrachloromercurate & fixed amount of Sodium Sulfite
Acid-bleached Pararosaniline color reagent (550 nm)

IP-9 – Gaseous SO2, HNO2, HNO3, NH3 & Particulate Mass, Sulfate, Nitrate, Ammonium, & Hydrogen Ion

Elutriator-accelerator jet assembly, impactor frit, & coupler assembly (& Cyclone if there are coarse particles)
Denuder #1 coated with Sodium Chloride (traps SO2 & HNO3)
Denuder #2 coated with Sodium Carbonate (traps HCl, HNO2, HNO3, & SO2)
Denuder #3 coated with Sodium Carbonate (traps NO2, HNO2, & SO2)
Denuder #4 coated with Citric Acid (traps NH3)
Teflon Filter (traps Ammonium & Sulfate particulates)
Nylon Filter (traps Hydrogen-ion & Nitrate particulates)
Ion Chromatography with Anion Exchange Resin column & Sodium Carbonate / Sodium Bicarbonate mobile phase
(determines Sulfate & Nitrate particulates and gaseous HNO3, SO2, HNO2), Sulfuric Acid regenerant;
Sodium Sulfate, Sodium Nitrate, & Sodium Nitrite stds.
Ion Chromatography with Cation Exchange Resin column & HCl / Histidine / Diaminopropionic Acid mobile phase
(determines particulate Hydrogen-ion & Ammonium), Tetrabutylammonium Hydroxide regenerant
pH Glass Electrode & Meter to determine particulate Hydrogen-ion
(Sulfuric Acid stds., pH 4 & 7 buffers; HClO4, KCl, Ethanol for check std. pH 4.09 +/- 0.04)
UV-VIS Spectrophotometer or Autoanalyzer to determine gaseous NH3 & particulate Ammonium
(EDTA buffer, Phenol, Sodium Hydroxide, Sodium Nitroprusside, Sodium Hypochlorite, & heating bath)
(Ammonium Chloride, citrate buffer, & glycerol for stds.)

IP-10A – Respirable Particulates (Size-Specific Impaction)

Microenvironmental Exposure Monitor (fixed sampler location):
Inlet Section – 4 large circumferential slots for aerosols to enter (keep large 100-um particles out)
3-Piece Inertial Impaction Section – Nozzle, impaction plate, & mount
Nozzle – converging inlet & cylindrical throat;
8-mm throat diameter removes particles with > 10 um aerodynamic diameter (1^{st} stage);
3-mm throat diameter removes particles with > 2.5 um aerodynamic diameter (2^{nd} stage)
Impaction Plate – stainless steel sintered disk permanently mounted; pores filled with mineral oil
(to prevent bounce of particles upon impact)
(air with the smaller particles flow around the impaction plate through 3 annular slots)
Air-flow straightening zone above filter holder
2-um pore size Teflon filter with cellulose backing material
Personnel Exposure Monitor:
Inlet & Nozzle Section – aerosol enters through 6 nozzles;
1.8 mm throat diameter for particulate matter cut size < 10 um
1.3 mm throat diameter for particulate matter cut size < 2.5 um
Annular Impactor Plate – stainless steel sintered annulus permanently mounted; pores filled with oil;
(air with smaller particles flows through circular opening in center of plate)
2-um pore size Teflon filter with cellulose backing material
Exit Section & Calibrated Rotameter with pump
Microbalance (weighs up to 3.5 g with accuracy +/- 0.5 ug)
Filters conditioned at least 24 hr in weighing room before initial and final weighings

IP-10B – Respirable Particulate Matter (Continuous Monitor)

Heated air inlet (equipped with optional sampling heads to pre-separate particles at 2.5 um or 10 um diameter)
Exchangeable filter cartridge
Sensor unit for filtered air, with microbalance system & automatic flow controller

(Volatile Organics)

TOXIC ORGANICS & INDOOR AIR POLLUTION METHODS

Zero-humidified Air for method blanks
TO-1, IP-1B: Tenax sorbent, cryogenic focusing (GC-MS)
TO-2: Carbon Molecular Sieve sorbent (GC-MS)
TO-3: Cryogenic focusing (GC-FID or GC-ECD)
TO-14A, IP-1A: SUMMA canister sample collection, cryogenic focusing (GC-MS or GC with multiple detectors)
TO-15A: Stainless Steel canister sample collection, multisorbent concentrator, reduced-temperature trap (GC-MS)
TO-17: Multiple Sorbents used in sampling, focusing in secondary sorbent trap (GC-MS)

IP-4A – Air Exchange Rate (GC-ECD)

Perfluorocarbon Tracer Gases: Perfluorodimethylcyclobutane, Perfluoromethylcyclopentane, Perfluorocyclohexane, or Perfluorodimethylcyclohexane
Capillary Adsorption Tubes with Activated Charcoal spherules
Nafion column (remove water vapor) & catalytic columns (to oxidize interfering chlorofluorocarbons)

IP-4B – Air Exchange Rate (GC-ECD)

Sulfur Hexafluoride Tracer Gas
Air Samples collected in bags, syringes with needle caps, or plastic bottles

(Extractable Organics & Pesticides)

TO-4A & IP-8 – Organochlorine Pesticides (GC-ECD, GC-NPD, GC-FPD, GC-ELCD, GC-MS, or HPLC-UV)

Polyurethane Foam (PUF) sorbent with quartz fiber filters
10% Ethyl Ether in Hexane eluting solvent (5% Ethyl Ether in Hexane for **IP-8**)

TO-5 – Aldehydes & Ketones (HPLC-UV)

Impinger Solution: 0.05% Dinitrophenylhydrazine in 2 N HCl
Isooctane & 30% Methylene Chloride in Hexane extraction solvents
Methanol exchange solvent
4:1 v/v Methanol-water mobile phase (reverse-phase HPLC)

TO-6 – Phosgene (HPLC-UV)

2% Aniline in Toluene (converts Phosgene to Carbanilide)
Acetonitrile exchange solvent
1:1 acetonitrile-water mobile phase (reverse-phase HPLC)

TO-7 – N-Nitrosodimethylamine (GC-MS)

Thermosorb/N sorbent
Acetone elution solvent

TO-8 – Phenol & Cresols (HPLC-UV)

Impinger solution: 0.1 N Sodium Hydroxide
Sulfuric Acid to adjust sample pH < 4
30% Acetonitrile / 0.1M pH 4.8 Acetate Buffer mobile phase (reverse-phase HPLC)

TO-9A – Polychlorinated & Polybrominated Dibenzo-p-dioxins & Dibenzofurans (HRGC-HRMS)

Quartz Fiber filter & Polyurethane Foam sorbent
Soxhlet Extractions with Toluene, Hexane exchange solvent
Acid-Base, Silica (optional), Alumina, & activated Carbon clean-up sorbents
9 Carbon-13 labeled polychlorinated Dibenzo-p-dioxin & Dibenzofuran internal stds. (added before extractions)
3 Carbon-13 labeled polybrominated Dibenzo-p-dioxin & Dibenzofuran internal stds.
Carbon-13 labeled 1234-TCDD injection std.
4 Carbon-13 labeled polychlorinated Dibenzo-p-dioxin & Dibenzofuran surrogates (added to PUF before sampling)
Tetradecane final exchange solvent
Perfluorokerosene for mass calibration & resolution calibration (>10000) of HRMS

TO-10A – Organochlorine Pesticides (GC-ECD)

Polyurethane Foam sorbent
5% Ethyl Ether in Hexane extraction solvent

TO-11A, IP-6A, & IP-6C – Aldehydes & Ketones (HPLC-UV)

Silica Gel sorbent impregnated with 2,4-Dinitrophenylhydrazine (DNPH)
Glass Fiber Filter coated with DNPH (**IP-6C**)
Acetonitrile elution or extraction solvent
Gradient elution mobile-phase 60% CH3CN/water to 100% CH3CN (reverse-phase HPLC)

TO-13A (GC-MS) & IP-7 (GC-FID, GC-MS, or HPLC) – Polynuclear Aromatic Hydrocarbons (PAH's)

Quartz Fiber filter & Polyurethane Foam (or XAD-2) sorbents
Soxhlet extraction (Methylene Chloride for filter & XAD-2, 10% Ethyl Ether in Hexane for PUF sorbent)
Silica Gel clean-up sorbent (optional)
Dibromobiphenyl (GC-FID or GC-MS), Decachlorobiphenyl (HPLC), or Deuterated PAH (GC-MS) internal stds.
Deuterated PAH Surrogates (added prior to extraction) & Field Surrogates (added prior to sampling)
Decafluorotriphenylphosphine mass spectrometer tuning solution (MS tuned each 12-hour shift)

IP-2A – Nicotine (GC-NPD)

XAD-4 sorbent
Ethyl Acetate elution solvent with 0.01% Triethylamine (prevents Nicotine adsorption onto glass)
Quinoline Internal Std.

IP-2B – Nicotine (GC-NPD)

Teflon Filter to collect Particulates
Teflon Filter treated with Sodium Bisulfate, to collect Nicotine
Particulates extracted with Methylene Chloride, exchanged into Ammonia-saturated Heptane
Treated filter extracted with 5% Ethanol, Nicotine deprotonated with Sodium Hydroxide & concentrated in Ammonia-saturated Heptane (prevents adsorption of Nicotine onto glass)

INSTRUMENT CALIBRATION REQUIREMENTS FOR MANDATED TEST METHODS

ROTAMETERS & FLOWMETERS

IP-2A, 13.2 & **IP-2B**, 12.2 (calibrate sampling pumps beginning and end of study)
IP-10A, 16.2 (Sampling pump flow rate accurate to within 5%)
TO-1, TO-2, 10.2.2 (Flow rate calibration w/ soap-bubble flowmeter, prior to sampling)
TO-5, TO-6, 10.2 (Flow rate calibration w/ soap-bubble flowmeter, prior to sampling)
TO-7, 9.3 & **TO-8**, 9.2 (Flow rate calibration w/ soap-bubble flowmeter, prior to sampling)
TO-4A, TO-9A, TO-13A, 11.2.3 (Flow rate calibration w/ soap-bubble flowmeter, prior to sampling)
TO-11A, 13.6.1 (Flow rate calibration w/ soap-bubble flowmeter, before & after sampling)

ANALYTICAL BALANCE or WOOD HEATER PLATFORM SCALE

IP-10A, 16.2 – Balance Zero 0.000 +/- 0.004 mg, Balance Calib. +/- 0.002 mg of std.; balance audited monthly

NON-DISPERSIVE INFRARED SPECTROMETER

IP-3A, 9.2.3 & 11.2.1 – 5 stds.+blank, each pt. +/- 1 ppm of expected value, precision check at 8-10 ppm every 2 wks
IP-3C, 10.3 – span gas concentrations at 0, 20, 40, 60, & 100% of full-scale (after electrochemical oxidation)

UV-VIS SPECTROPHOTOMETER

IP-5B, 10.2.1 – 6 stds. + Blank
IP-9, 18.1 – 8 Stds. + Blank in DDI water, and 8 Stds. + Blank in KCl solutions

CHEMILUMINESCENCE ANALYZER

IP-5A, 10.2.3 & 12.2.1 – span gases at 10, 20, 40, 60, & 80% of full-scale, precision check at 8-10 ppm every 2 wks
IP-5A, 11.2 – leak check weekly, potentiometer settings daily (zero & span settings within 5% of stated values)

ION CHROMATOGRAPH

IP-9, 17.4.3.2 – 3 Stds. + Blank

ION SPECIFIC ELECTRODE POTENTIOMETRY

IP-9, 19.5 – 6 Stds. + Blank

FLAME IONIZATION DETECTOR ANALYZER

TO-12, 11.3.4 – each std. analyzed in triplicate

GAS CHROMATOGRAPH – FLAME IONIZATION DETECTION

TO-3, 12 – Calib. Verification Std. every 4-6 hr (also applies to GC-ECD)
IP-7, 13.4.1 & 13.4.2 – 5 Stds., CF or RF (if used) < 20% RSD; CCV recovery 80-120%

GAS CHROMATOGRAPH – NITROGEN-PHOSPHORUS DETECTOR

IP-2A, 11.1.2 – 5 Stds., r2 > 0.998 (linear or quadratic fit)
IP-2B, 10.3.3.2 & 11.4 – 3 high-level Stds. or 5 low-level Stds., r2 > 0.998 (linear or quadratic fit)

GAS CHROMATOGRAPH (detector not specified or multiple detectors)

IP-1A, 10.3.3 – 3 Stds. + Zero humid air
IP-8, 13.2 & 13.3 – 3 Stds., CF or RF (if used) < 10% RSD; CCV recovery 90-110%
TO-10A, 12.1.6 – 3 Stds., CF or RF (if used) < 20% RSD; CCV recovery 85-115%
TO-14A, 10.3.3.1 & 10.3.3.2 – 3 Stds. + Zero humid air, CF or RF (if used) < 30% RSD: CCV recovery 70-130%

GAS CHROMATOGRAPH – MASS SPECTROMETER

IP-1A, 10.2.3 – 3 Stds. + Zero humid air
IP-1B, 12.5.2.2 – 5 Stds., CF or RF (if used) < 10% RSD
IP-7, 14.3.1 & 14.3.2 – 5 Stds., CF or RF (if used) < 20% RSD; CCV recovery 80-120%, done each 12-hr shift
TO-13A, 13.3.4 & 13.3.5 – 5 Stds., RF (if used) < 30% RSD; CCV recovery 70-130%, done each 12-hr shift
TO-14A, 10.2.3.1 & 10.2.3.2 – 3 Stds. + Zero humid air, CF or RF (if used) < 30% RSD; CCV recovery 70-130%
TO-15, 10.5 & 10.6 – 5 Stds., RF (if used) < 30% RSD (2 analytes may be < 40% RSD); CCV recovery 70-130%

GAS CHROMATOGRAPH – HIGH-RESOLUTION MASS SPECTROMETER

TO-9A, 13.5 – 5 Stds. each analyte, Relative Response Factors constant within 25%
TO-9A, 13.7 – Verification with Mid-Range Std., results agree within 25%

HIGH PERFORMANCE LIQUID CHROMATOGRAPH

TO-6, 12.5 & 12.6 – 5 Stds., triplicate injections each conc., r2 > 0.999; CCV recovery 90-110%
TO-8, 11.5 & 11.6 – 5 Stds., triplicate injections each conc., r2 > 0.999, RRT < 2%; CCV recovery 90-110%
IP-6A, 11.4.2 & **IP-6C**, 9.3.2 – 5 Stds., triplicate injections each conc., r2 > 0.999
IP-6A, 11.4.3 & **IP-6C**, 9.3.3 – CCV recovery 90-110% for HCHO > 1 mg/L or 80-120% for HCHO < 1 mg/L
IP-7, 15.4 – d
TO-11A, 11.4.3 & 11.4.5 – 5 Stds., triplicate injections each conc., r2 > 0.999, every 6 mo.; CCV recovery 85-115%

CERTIFICATION OF CANISTERS & SAMPLING SYSTEMS

TO-12, 10.1.6 (<0.02 ppm C)
TO-14A, IP-1A, 9.1.3.6 & 11.1 (<0.2 ppbv each analyte)
TO-15, 8.2.3.6 & 8.4.1 (<0.2 ppbv each analyte)
Zero humid air certification of each canister prior to use,
then 100% of canisters tested with zero humid air after each cleaning
(percentage of canisters tested may be reduced if cleanup system & canisters are proven reliable)

TO-14A, IP-1A, 9.1.3.6 & 11.2.2 (<0.2 ppbv each analyte)
TO-15, 8.2.3.6 & 8.4.3 (<0.2 ppbv each analyte)
Zero humid air certification of sampling system (one time prior to use, without an evacuated canister)

TO-14A, 9.2.2 & 11.2.3 (90-110% recovery each analyte in ppbv range)
TO-15, 8.4.4 (90-110% recovery each analyte in ppbv range)
IP-1A, 9.1.3.6 & 11.2 (90-110% recovery each analyte at 10 ppbv)
Certification of sampling system with humid-air calibration gas standards
(one time prior to use with certified evacuated canister)

HOLDING TIME, SAMPLE CONTAINER, & SAMPLE PRESERVATION REQUIREMENTS

7 Days to Extract Sample, 40 Days to Analyze Extract

TO-4A, 11.3.4.10
TO-9A, 11.3.4.9
TO-10A, 10.3

14 Days

EPA 0061, 6.3 – Chromium(VI)

28 Days

EPA 0050, 0051, 6.2 – Hydrogen Chloride & Chlorine

QUALITY CONTROL ACCEPTANCE CRITERIA FOR MANDATED TEST METHODS

EPA Audit Sample Analysis with each sample set

TO-11A, 13.6.1 – analyzed before & after sample analyses

Leak Check of Sampling System after each sampling run (& with each major component change during run)

Plug probe nozzle & pull vacuum to 380 mm Hg (15 inches)
Acceptable leakage rate < 0.00057 m3/min (0.02 cfm) or 4% of average sampling rate (whichever is less)

EPA 0010, 0011, 0023A, 6.5.2-6.5.3
EPA 0020, 4.4.3.11-4.4.4 – acceptable leakage rate < 0.0014 m3/min (0.05 cfm)
EPA 0031, 6.5.3 & 7.3.2 – acceptable leakage rate < 0.02 Lpm at 1.0 Lpm flow rate, < 0.01 Lpm for lower flows
EPA 0050, 7.4.2-7.4.3
EPA 0060, 6.1.5
EPA 0061, 7.1.5

Plug probe nozzle & pull vacuum to at least 250 mm Hg (10 inches)
Acceptable leakage rate < 2% of average sampling rate

EPA 0030, 4.6 & 4.7.2 – Acceptable leakage rate < 2.5 mm Hg after 1 min
EPA 0051, 7.2.2 & 7.2.4 – vacuum remains stable for 30 sec

Seal end of probe & pull vacuum to 5 inches Hg
Acceptable leakage rate < 0.1 inch Hg per minute

EPA 0040, 7.3.3

Leak Check of Sampling System prior to sampling

EPA 0010, 0011, 0023A, 0031, 6.5.1 – same procedure as for the post-test leak check
EPA 0030, 4.6 – same procedure as for the post-test leak check
EPA 0040, 7.3.2 – same procedure as for the post-test leak check
EPA 0051, 7.2.2 – same procedure as for the post-test leak check

Leak Test ALL Cannisters prior to Sample Collection

TO-12, 10.1.2 (30 +/- 2 psig over 24 hr)
TO-14A, 11.1.2 (30 +/- 2 psig over 24 hr)
TO-15, 8.4.1.2 (30 +/- 2 psig over 24 hr)

Leak Test of Analytical System

TO-12, 11.1 & **TO-17**, 11.2

Mass Flow Meter Calibrations

IP-7, 10.2.2, 10.3.7, 10.4.6 – voltage vs. flow rate before & after each test series
TO-10A, 8.2 – calibrate air samplers before & after each collection period
TO-11A, 13.6.1 – calibration factor determined quarterly

Matrix Spike every 20 Samples

TO-13A, 14.1.3

QC Sample results

TO-9A, 15.9 – Recoveries 70-130% each analyte at the 0.25-2.0 pg/m3 conc. level
TO-14A, 12.2 – Recoveries 90-110% each analyte at 8 ppbv level
TO-15, 11.1.1 & **TO-17**, 14.1 – Recoveries 70-130% each analyte

All Samples analyzed in Duplicate

TO-12, 11.4.19

One Sample in 10 prepared & analyzed (or counted) in Duplicate

IP-10A, 16.2 – 10% of filters weighed by 2nd analyst, results agree within +/- 0.010 mg

TO-11A, 13.6.1

Field Duplicates Collected & Analyzed 10% of Samples

TO-11A, 13.6.1

Duplicate Precision Results

TO-9A, 15.9 – Precisions < 30% RPD

TO-15, 11.1.1 & **TO-17**, 14.1 – Precisions < 25% RPD

Backup Cartridge Collected on 10% of Samples to Test for Breakthrough

TO-11A, 13.6.1

Zero Gas Blank Results

TO-14A, 10.2.3.1 – <0.2 ppbv each analyte in zero humid air, repeat analysis after each GC/MS initial calibration

TO-15, 10.7 – method blank with zero humid air

Sorbent Blank Results

IP-7, 10.2-10.4 – <10 ng each analyte on quartz filter or XAD-2 & PUF cartridges

TO-9A, 15.9 – <0.25 pg/m3 each isomer

TO-10A, 9.5 – <10 ng per cartridge each single-component analyte, <100 ng each multicomponent analyte

TO-11A, 13.6.1 – <0.15 ug HCHO, <0.30 ug Acetone, <0.10 ug other analytes per cartridge; at least 3 cartridges analyzed per lot or prepared batch

Solvent Blank Analysis Each Batch

TO-11A, 13.6.1

Baseline Drift

IP-10A, 16.2 – balance zero checked every 10 weighings & end of batch (+/- 0.007 mg of 1st zero check in batch & +/- 0.005 mg of previous balance zero check)

Sample Collection Efficiency

IP-8, 17.3.1 & **TO-10A**, 15.3.1 – prior to first use of method

TO-9A, 15.9 – recoveries 50-120% each spiked labeled compound

TO-13A, 13.4.6.3 – recoveries 60-120% each spiked deuterated PAH

Internal Standard Recoveries (Carbon-13 Labeled Cmpds. added prior to extraction)

TO-9A, 15.9 – recoveries 50-120% for tetra-, penta-, & hexa-chloro Dibenzo-p-dioxins & Dibenzofurans; Recoveries 40-120% for hepta- & octa-chloro Dibenzo-p-dioxins & Dibenzofurans

TO-13A, 13.3.7.4 – recoveries 60-120% for deuterated PAH's added prior to extraction; Recoveries of int. stds. added prior to analysis 50-200% of value from last CCV

TO-15, 10.5 – Recoveries 60-140% for int. stds. added prior to GC/MS analysis (compared with last CCV)

Surrogate Recoveries

IP-7, 16.3.5 & 16.4.7 – 80-120% (GC/FID & GC/MS)

GC Retention Time Windows

TO-13A, 13.3.4 & 13.3.6.4 & 13.3.7.4 – within 20 sec of mean RT during initial calib, within 20 sec of RT during last CCV for QC samples

TO-15, 10.5 & 10.7.5 & 10.8.6.2 – within 20 sec of mean RT during initial calib, within 20 sec of RT during last CCV for QC samples

Identification Criteria for Chlorinated Dibenzo-p-dioxins & Dibenzofurans

TO-9A, 14.2 – M/M+2 & M+2/M+4 within 15% of theoretical values;
Analyte retention times within 3 sec of corresponding carbon-13 labeled std.;
Mass ions for given analyte reach maximum response within 2 sec of each other;
Signal-to-noise ratio for all mass ions > 2.5;
No signal observed for Pentachlorodiphenyl Ethers at same retention times as PCDF's

Method Detection Limit Determined According to 40 CFR Part 136 Appendix B

TO-11A, 13.5-13.6.1 – also verified annually or with each instrument change
TO-15, 11.1.1 & **TO-17**, 14.1 – MDL < 0.5 ppbv each analyte

Fourier Transform IR Operations (after obtaining sample spectrum) – TO-16

Subtract out background FTIR spectrum
Subtract out FTIR spectrum due to water vapor
Subtract out FTIR spectrum due to stray light or blackbody radiation
Correct for any spectral shifts in wavenumbers
Measure return beam intensity with each change of IR source or detector
Relate any absorbances at specific wavenumbers with compound concentrations if calibrations are available