



Data Validation Templates for NATTS Pollutants

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Quality assurance activities are built into ambient air monitoring regulations

NAAQS:

40 CFR Part 50

Technical Activities:

40 CFR Parts 50 & 53

QA/QC Activities:

40 CFR Part 58, Appendices A-E



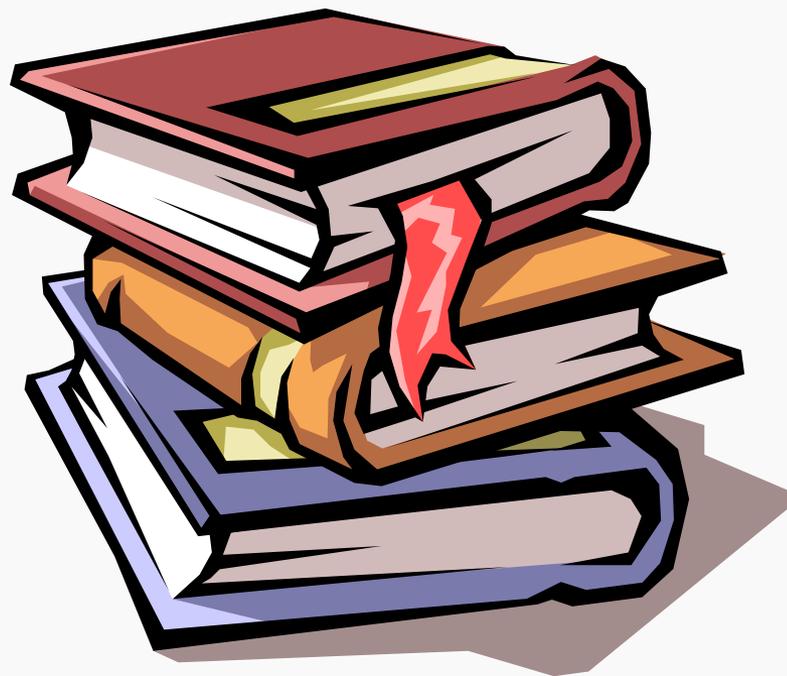
But there are no NAAQS for air toxics...



So how do we verify air toxics data quality?

- NATTS TAD
- Method Compendia
- QAPP & SOP Requirements (MQOs, DQIs, etc)

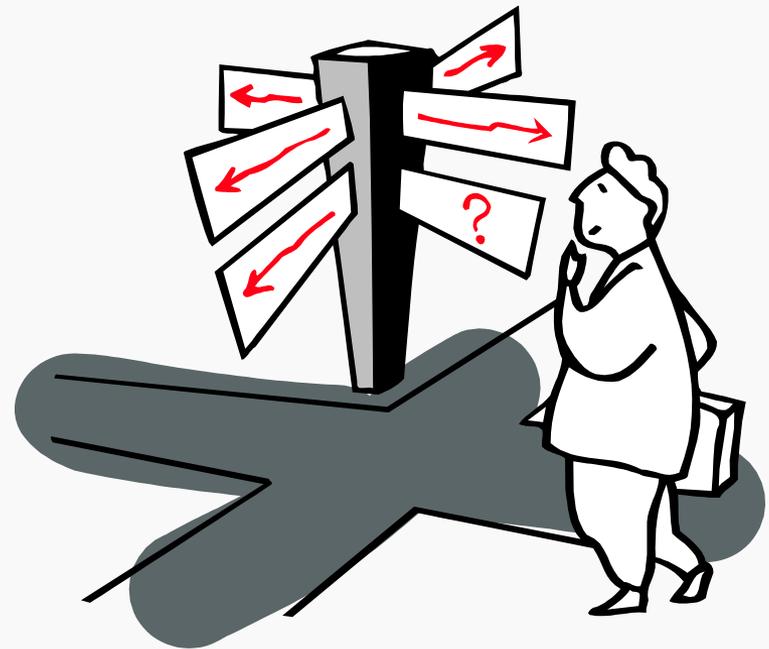
However, after we verify the data, the Methods & TAD don't provide detailed guidance on data coding for AQS...



Consistency is an important QA function!

In order to perform sampling & analysis methods consistently, standardized procedures are developed.

How comparable are data sets that have not been handled & reported in the same manner?





Criteria Pollutant Data Validation Templates

QA Handbook Volume II, Appendix D
 Revision No. 1
 Date 12/08
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PM_{2.5} Filter Based Local Conditions Validation Template

Criteria	Frequency	Acceptable Range	Information (CFR or Method 2.12)
CRITICAL CRITERIA- PM_{2.5} Filter Based Local Conditions			
Filter Holding Times	all filters	± 7 days 9 hours from sample end date	Part 50 App L Sec 10.10
Sample Recovery	all filters	± 10 days from sample end date if shipped at ambient temp. or ± 30 days if shipped below ave ambient (or 4° C or below for ave sampling temps < 4° C.) from sample end date	Part 50 App L Sec 8.3.6
Post-sampling Weighing	all filters	± 1380-1500 minutes, or value if < 1380 and exceedance of NAAQS ^{1/} midnight to midnight	Part 50 App L Sec 3.3 Part 50, App L Sec 7.4.15
Sampling Period (including multiple power failures)	all filters	1380-1500 minutes, or value if < 1380 and exceedance of NAAQS ^{1/} midnight to midnight	Part 50 App L Sec 3.3 Part 50, App L Sec 7.4.15
Sampling Instrument	every 24 hours of op	average within 5% of 16.67 liters/minute	Part 50 App L Sec 7.4
Average Flow Rate	every 24 hours of op	CV ≤ 2%	Part 50, App L Sec 7.4.3.2
Variability in Flow Rate	every 24 hours of op		
Filter			
Visual Defect Check (unexposed)	all filters	see reference	Part 50, App L Sec 10.2
Filter Conditioning Environment			
Equilibration	all filters	24 hours minimum	Part 50, App L Sec 8.2
Temp. Range	all filters	24-hr mean 20-23° C	Part 50, App L Sec 8.2
Temp. Control	all filters	± 2° C SD ^{2/} over 24 hr	Part 50, App L Sec 8.2
Humidity Range	all filters	24-hr mean 30% - 40% RH or ± 5% sampling RH but > 20%RH	Part 50, App L Sec 8.2
Humidity Control	all filters	± 5% SD ^{2/} over 24 hr	Part 50, App L Sec 8.2
Pre-post Sampling RH	all filters	difference in 24-hr means ± 5% RH	Part 50, App L Sec 8.3.3
Balance	all filters	located in filter conditioning environment	Part 50, App L Sec 8.3.2
Verification Calibration			
One-point Flow Rate Verification	1/4 weeks	± 4% of transfer standard	Part 50, App L Sec 9.2.5 Part 58, Appendix A Sec 3.2.3 & 3.3.2
OPERATIONAL EVALUATIONS TABLE PM_{2.5} Filter Based Local Conditions			
Filter Checks			
Lot Blanks	9 filters per lot	less than 15 µg change between weighings	Method 2.12 Sec 7.7
Exposure Lot Blanks	3 filters per lot	less than 15 µg change between weighings	Method 2.12 Sec 7.7
Filter Integrity (exposed)	each filter	no visual defects	Method 2.12 Sec 8.2
Filter Holding Times			
Pre-sampling	all filters	< 30 days before sampling	Part 50, App L Sec 8.3
Lab QC Checks			
Field Filter Blank	10% or 1 per weighing session	± 30 µg change between weighings	Part 50, App L Sec 8.3
Lab Filter Blank	10% or 1 per weighing session	± 15 µg change between weighings	Part 50, App L Sec 8.3
Balance Check	beginning, 10th sample, end	± 3 µg	Method Sec 7.9
Duplicate Filter Weighing	1 per weighing session	± 15 µg change between weighings	Method Sec 7.11
Sampling Instrument			

The QA Handbook is part of the quality management system designed to ensure that the Air Program is implemented consistently across the nation.

The QA Handbook contains “validation templates” designed for consistent data validation!

Critical, Operational, & Systematic Criteria...



Templates for Toxics?

- The uniqueness of air toxics requires better communication and understanding between laboratories and air monitoring agencies to achieve the highest quality of data.
- On March 29, 2013, a small workgroup in R4 formed. First meeting was held in Athens, immediately following the annual R4 Air Monitoring Workshop.
- Workgroup included R4 air monitoring managers, laboratory managers, and EPA auditors.
- First conference call on April 25, 2013.



R4 Air Toxics Workgroup Members

- **SESD:** Tim Slagle, Ray Terhune, Richard Guillot, Doug Jager, Stephanie McCarthy*
- **States:** Kentucky* (Stacie Masters); South Carolina (Scott Reynolds, Robert Schilling)
- **Locals:** Hillsborough County (Alain Watson, Joe Barron); Pinellas County (Tom Stringfellow, Michael Liadis)





Goals & Objectives

- To provide a mechanism for Region 4 S/L/T agencies that would facilitate more consistent data handling procedures for air toxics pollutants
 - Identify the critical QC elements for each pollutant
 - Prioritize the QC elements for each pollutant
 - Incorporate requirements for both field sample collection and laboratory analysis, based on current methodology
 - Provide guidance on use of AQS flags, based upon results of lab analysis and field issues



- Build data validation templates for air toxics pollutants similar to the templates used for the criteria pollutants in the QA Handbook
- Focus on NATTS pollutants and guidance
 - Serve as a useful reference for organizations sampling for toxics at non-NATTS sites
- Regional acceptance and use of templates
 - Feedback from other R4 personnel
 - EPA approval



Workgroup Activities

- Between April 2013 – February 2014, the workgroup participated in twelve conference calls to discuss and build the templates
- Initial template for each pollutant built using NATTS TAD & Method Compendia





Workgroup Activities

- Lengthy discussions about each template and QC criteria
 - Current methods are not interpreted the same from agency to agency
 - Critical, operational, or systematic criteria?
 - Logistical issues based upon the knowledge and experience of workgroup members
- Extensive review by individual members for accurate citations & references



Air Toxics Data Validation Templates



PM10 Metals NATTS Data Validation Template			
Requirement	Frequency	Acceptance Criteria	Reference
CRITICAL CRITERIA - PM10 Metals Data for NATTS participating agencies			
Filter media	All samples	Quartz for hi-volume; Teflon for low-volume	NATTS TAD Table 3.2-7
Sampling Period (including any power failures)*	All samples	1380-1500 minutes (24 hrs ± 1 hr)	NATTS TAD Table 3.2-7
Field Sampler Performance Checks			
Field Sampler Flow Rate Verification – Hi-volume	Quarterly	± 7% of transfer standard; ±10% of design flow rate.	40 CFR 58, Appendix A, Section 3.3.2 Method 2.10, Table 3-1
Field Sampler Flow Rate Verification – Low-volume	Quarterly	± 4% of transfer standard; ±5% of design flow rate.	40 CFR 58, Appendix A, Section 3.3.2 40 CFR 50, Appendix L, Section 9.2.5 ³
Sample Pick-Up Time	All samples	As soon as practicable within 6 days	As per agency requirements
Sample Holding Time	All samples	Filters maintained in climate-controlled storage until extraction. Filters extracted within 180 calendar days post-sampling.	NATTS TAD Table 3.2-7
Chain-of-custody	All samples	Each filter must have a unique identification number. Filter has a valid and legible COC with complete sample documentation.	
MS Multi-Point Calibration	Daily	At least 4 calibration points; correlation coefficient ≥ 0.995	IO-3.5 Section 10 NATTS TAD Table 3.2-7
Tuning Standard	Daily - Before Calibration	Resolution at low mass - Magnesium isotopes 24, 25, & 26; high mass indicated by lead isotopes 206, 207, & 208. Resolution = 0.75amu peak width at 5% peak height. RSD less than 5% for 5 replicates.	IO-3.5 Section 10.1.1 NATTS TAD Section 4.3.3.4
Internal Standards	Every sample	60-125% of the intensity of the daily multi-point calibration standard	IO-3.5 Section 11.5
Initial Calibration Verification (ICV) – QC Standard prepared from 2nd Source	Immediately after daily multi-point calibration	90 to 110% of the actual concentration	IO-3.5 Section 11.3.2 NATTS TAD Tables 3.2-7 and 4.3-4
Initial Calibration Blank (ICB)	Immediately after initial calibration verification	<MDL	IO-3.5 Section 11.3.3 NATTS TAD Tables 3.2-7 and 4.3-4
High Standard Verification	Following the initial calibration blank analysis	95 to 105% of the actual concentration	IO-3.5 Section 11.3.4 NATTS TAD Table 3.2-7

Air Toxics Data Validation Templates



OPERATIONAL CRITERIA - Carbonyls Data for NATTS participating agencies

Media Handling	All samples	Sample collection recommended within 72 hours post-sampling. Collected sample maintained at $\leq 4^{\circ}\text{C}$ until extraction. If a cartridge leaks silica gel, it must be voided.	TO-11A Sections 3.2, 10.12, & 11.1.1 NATTS TAD Section 4.2.3.3 As per agency SOP requirements
HPLC Column Efficiency	Determined at instrument setup and once per sample batch	Resolution between Acetone hydrazone and Propionaldehyde hydrazone ≥ 1.0 .	NATTS TAD Tables 3.2-9 and 4.2-4
Field Sampler Clock/Timer Check	All samples	Clock/timer accurate, set to local standard time; sample collection midnight to midnight.	Clock's accuracy specification is equipment dependent and EPA-approved QAPP and/or SOP specific.
Field Sampler Performance Checks			
Field Sampler Single-Point Flow Check	Before and after each sample collection	Equipment dependent. Minimum sample volume required determined by individual agency/laboratory in EPA-approved QAPP or SOP.	TO-11A Section 10.5
Field Sampler Leak Check	Before and after each sample collection	Equipment dependent.	TO-11A Sections 10.2 & 13.6
Sampler Zero Certification	Annually. Performed prior to field deployment and after any major component repair/replacement.	Humidified zero air or Nitrogen; < 0.2 ppbv for each analyte	NATTS TAD Table 3.2-9 NATTS TAD Section 4.2.2.2
Ozone Scrubber ² Performance	At least once annually	Effectively remove O ₃ from the air stream for up to 100,000 ppb-hours. Scrubber is expected to last for 12 months of 24-hour sampling on every sixth day.	NATTS TAD Section 4.2.1.2
Sampling Field Blank	6 per year	< 0.30 μg /cartridge Formaldehyde; < 0.4 μg /cartridge Acetaldehyde; < 0.75 μg /cartridge Acetone; < 7.0 μg /cartridge sum of all others	NATTS TAD Tables 3.2-9 and 4.2-4 US EPA NATTS Grant Workplan
Precision	Duplicate or collocated samples. Duplicate samples are taken simultaneously through the same collection system. Collocated samples are taken simultaneously through 2 separate collection systems at the same location. 10 % of total samples (6 per year for 1-in-6 day sampling.)	$< 15\%$ CV	NATTS TAD Section 4.2.2.3 NATTS TAD Table 3.2-4

Air Toxics Data Validation Templates



SYSTEMATIC CRITERIA - VOC Data for NATTS participating agencies

SYSTEMATIC CRITERIA - VOC Data for NATTS participating agencies			
Field Sampler Performance Checks			
Sampler Calibration Verification	Annually	Per manufacturer's specifications	Instrument user manual
Total system leak check ²	Quarterly	Equipment dependent. If targeting 3.5 sccm flow, recommend <0.1 sccm leak rate; or, equivalent loss of vacuum.	Instrument user manual. NATTS TAD Section 4.5.1.2
Field Sampler Flow Rate Audit ²	Quarterly	± 10% of target flow rate	TO-15 Section 8.3.5 As per agency requirements
Siting	Annually	270° unobstructed probe inlet, placed 2-4 meters above-ground level, at least 10 meters from the drip line of trees	NATTS TAD Section 4.1.4.1
Sample Probe and Inlet		Chromatographic grade stainless steel or Borosilicate Glass	NATTS TAD Sections 4.1.4.1 and 4.1.4.2
Filter and Sample Line Cleaning*	Quarterly	Cleaned with distilled water	NATTS TAD Section 4.1.4.1
Data Reporting to AQS*	Quarterly	Reported at Standard Conditions. All data are reported, including data below MDL.	NATTS TAD Sections 4.0, 5.3.1.1, and 5.3.1.4
AQS Reporting Units*	Quarterly	ppbv or ug/m3	NATTS TAD Section 5.3.1.4
Data Completeness*	Quarterly	>85%	NATTS TAD Table 3.2-1



Laboratory Manager Perspective

- Many laboratory personnel may have limited experience or understanding regarding the field elements involved in sample collection
 - The templates are all-inclusive and provide information laboratory personnel can utilize to determine sample validity upon receipt at the laboratory
- There are discrepancies between the TAD and method compendia, in regards to laboratory requirements
 - The templates clarify these requirements so all NATTS laboratories report data using the same guidelines
- Templates provide guidance for laboratories to ensure the data generated meets the objective of the project
- Will help laboratory personnel provide useful comments to air agencies in data packages, to assist S/L/T with data validation procedures



Air Agency Manager Perspective

- Air agency staff may or may not be chemists!
- NATTS TAD currently does not categorize QC elements as “critical, ” “operational,” or “systematic”
- Without a keen understanding of analytical methods & flags, an air agency data reviewer may miss important qualifiers
 - Templates would clearly define the critical elements for sample collection and analysis to provide data assessors more guidance on how to properly interpret final data reports



Air Agency Manager Perspective

- Lab personnel verify data and submit reports to the client based upon their own QMP.
- Responsibility of the State/Local to review & validate the data, ensuring it meets the requirements of its intended use – **the QAPP and AQS.**
 - Templates would foster consistency in data handling in regards to determining validity & application of AQS flags.

The screenshot shows the AQS software interface with a table of Monitor / Geographic Criteria. The table has columns for State Code, County Code, Site Id, Parameter Code, POC, City Code, AGCR Code, UAR Code, CBSA Code, CSA Code, and EPA Region Code. Below the table, there are sections for Protocol C17 and Pollutant Type. A tablet in the foreground displays the AQS Data Coding Manual, which includes the EPA logo and the title 'AQS Data Coding Manual'.

Qualifier Code	Qualifier Desc	Qualifier Type Desc
1	Deviation from a CFR/Critical Criteria Requirement	Quality Assurance Qualifier
2	Operational Deviation	Quality Assurance Qualifier
3	Field Issue	Quality Assurance Qualifier
4	Lab Issue	Quality Assurance Qualifier
5	Outlier	Quality Assurance Qualifier
6	QAPP Issue	Quality Assurance Qualifier
7	Below Lowest Calibration Level	Quality Assurance Qualifier
8	Negative value detected - zero reported	Quality Assurance Qualifier
9	Values have been Blank Corrected	Quality Assurance Qualifier
CC	Clean Canister Residue	Quality Assurance Qualifier
CL	Surrogate Recoveries Outside Control Limits	Quality Assurance Qualifier
DI	Sample was diluted for analysis	Quality Assurance Qualifier
EH	Estimated; Exceeds Upper Range	Quality Assurance Qualifier
FB	Field Blank Value Above Acceptable Limit	Quality Assurance Qualifier
	hold time exceeded	Quality Assurance Qualifier
	above acceptable limit	Quality Assurance Qualifier
	Analyte Is Acceptable; Reported Value Is An Estimate	Quality Assurance Qualifier
	Reported Value May Be Biased High	Quality Assurance Qualifier
	Reported Value May Be Biased Low	Quality Assurance Qualifier
	DL	Quality Assurance Qualifier
	1/2 MDL substituted.	Quality Assurance Qualifier
		Quality Assurance Qualifier
	by source	Quality Assurance Qualifier
	Criteria	Quality Assurance Qualifier
	QL and MDL	Quality Assurance Qualifier
	from secondary monitor	Quality Assurance Qualifier
	ing Criteria	Quality Assurance Qualifier
	Above Acceptable Limit	Quality Assurance Qualifier
	ature Is Out of Specs.	Quality Assurance Qualifier
	at; no reason to invalidate	Quality Assurance Qualifier
	re out of Spec.	Quality Assurance Qualifier
	re Difference out of Spec.	Quality Assurance Qualifier
	me out of Spec.	Quality Assurance Qualifier



EPA Auditor Perspective



- The TAD/Methods may be guidance, but they define the limits of acceptable data
- Samples should be analyzed by similar methods and QC requirements
- Some flexibility is allowed, but should be backed by an **EPA-approved** QAPP/SOP
- The quality of the data must be documented and verified
- Consistency is the goal!
- Templates may foster improved communications between laboratories and air agencies



OAQPS Joins the Team

- In the summer 2013, OAQPS was notified of the R4 workgroup formation & objective
- First draft of completed templates were submitted to OAQPS for initial review in late February 2014
- On March 18, 2014, the workgroup conducted a conference call with OAQPS to discuss the templates & specific line items
 - New language proposed for some criteria that needed OAQPS approval
 - Overall response from OAQPS was positive!
- During the call, OAQPS expressed the need for consistent air toxics data handling on a national level & that these templates could spark that effort!



Additional Objectives Emerged

- The workgroup identified discrepancies within the NATTS TAD, as well as inconsistencies between the TAD and method compendia
- Developed a list of recommendations for OAQPS to consider regarding potential changes within the TAD
 - Clarification of stated procedures or language the group found confusing
 - Review of requirements the group found logistically challenging, such as annual certification of VOC samplers
 - Recommended updates based on current instrumentation
 - Best practices, such as improving documentation of chain-of-custody



Region 4 Premiere

- On March 27, 2014, the workgroup presented the draft templates to R4 agencies during the annual Air Monitoring Workshop
- Templates were submitted to all attendees via email for closer inspection
 - Asked to share the templates with field and laboratory personnel
 - Solicited comments
- Workgroup received comments and suggestions from both air agencies & laboratories





Template Revision

- Workgroup reconvened to discuss all comments received
- Revised templates & TAD discussion items were shared with OAQPS in June 2014
- Final conference call with OAQPS & Battelle in July 2014
 - Discussed both templates & TAD
- Templates approved for R4 use!
 - Will be distributed in the coming weeks





National News!

- OAQPS has stated that the templates will be adopted into the revised NATTS TAD
- Templates will be modified for national use
- The templates will be available for review and comment with the draft version of the new NATTS TAD

Requirement	Frequency	Acceptance Criteria	Reference
CRITICAL CRITERIA - PAHs Data for NATTS participating agencies			
Filter media	All samples	Quartz fiber filter with PUF/XAD-2* sorbent cartridge.	TO-13A Section 2.1 NATTS TAD 4.5.1 ASTM D6209-98 (2004) Section 7.4
Sampling Period (including any power failures)*	All samples	1380-1500 minutes (24 hrs ± 1 hr)	NATTS TAD Table 3.2-10
Sample Holding Time	All samples	Extraction: Within 7 days from sample collection (at 4°C). Analysis: Within 40 days from extraction (4°C).	NATTS TAD Section 4.5.3 NATTS TAD Tables 3.2-10 and 4.5-6 ASTM D6209-98 (2004) Sections 12.3.15 & 13.1.14
Chain-of-custody	All samples	Each sample should have a unique identification number. Sample has a valid and legible COC, with complete sample documentation.	TO-13A Section 11.3.4.8
GC/MS Multi-Point Calibration	After failure to meet daily acceptance criteria. Or, after major repair, maintenance, or change in instrumentation. At a minimum, annually.	5 points over the range of 0.1 to 2.0 ng/ul.	TO-13A Section 13.3.4 NATTS TAD Section 4.5.4 ASTM D6209-98 (2004) Section 14.2.1

OPERATIONAL CRITERIA - PAHs Data for NATTS participating agencies			
Media Handling	All samples	Protected from light prior to & after use/sample collection. Sample retrieved within 24 hours post-sampling. Media maintained at 4°C storage until extraction.	TO-13A Sections 6.2.7 and 11.3.4 NATTS TAD Section 4.5.2.1
Field Sampler Clock/Timer Check	All samples	Clock/timer accurate, set to local standard time; sample collection midnight to midnight.	TO-13A Table 8 NATTS TAD Section 4.5.1.2. Clock's accuracy specification is equipment dependent and EPA-approved QAPP and/or SDP specific.
Field Sampler Performance Checks	Before and after each sample collection	Equipment dependent. Minimum sample volume required determined by individual agency/laboratory in EPA-approved QAPP or SDP.	TO-13A Section 11.2.3 ASTM D6209-98 (2004) Section 12.3.7
Field Sampler Flow Multi-point Calibration	Prior to system deployment, after major repairs or maintenance, and annually thereafter.	Calibration using annually-certified orifice transfer standard. ± 7% of transfer standard; ±10% of design flow rate.	TO-13A Section 11.2.2 NATTS TAD Section 4.5.1.2 NATTS Table 3.2-10
Field Sampler Flow Rate Audit	Quarterly	± 7% of transfer standard; ±10% of design flow rate	TO-13A Section 11.2.2.1 NATTS TAD Table 3.2-10
Sampling Field Blank	Monthly	<3 x MDL	ASTM D6209-98 (2004) Section 12.4.1 US EPA NATTS Grant Workplan
Precision	Collocated samples. Collocated samples are taken simultaneously through 2 separate collection systems at the same location. 10% of total samples (6 per year for 1-in-6 day sampling.)	<15% CV	NATTS TAD Table 3.2-5
Bias (Proficiency Testing)*	Two per calendar year ¹	±25% for each analyte/sample	NATTS TAD Table 3.2-5
Sensitivity (Method Detection Limits)	1/year. Or, after a major instrument change.	Minimum of 7 low level cartridge standards analyzed over a 2-day period (minimum). Naphthalene: 0.029 µg/m ³ Benzo(a)pyrene: 0.00091 µg/m ³	40 CFR 136, Appendix B. NATTS TAD Section 4.5.6 NATTS TAD Table 3.2-5
Laboratory Stock Calibration Standards	Each standard	Must be replaced within one year or within expiration date of standard, whichever comes sooner	TO-13A Section 13.2.1.7 NATTS TAD Section 4.5.2.5 ASTM D6209-98 (2004) Section 16.1
SYSTEMATIC CRITERIA - PAHs Data for NATTS participating agencies			
Siting	Annually	270° unobstructed probe inlet, placed 1.2-3 meters above ground level, at least 10 meters from drip line of trees. Collocated samplers spaced at least 2 meters distance apart.	NATTS TAD Section 4.5.1.2
Data Reporting to AQS*	Quarterly	Reported at Standard Conditions. All data are reported, including data below MDL.	NATTS TAD Sections 5.3.1.1 and 5.3.1.4
AQS Reporting Units*	Quarterly	ng/m ³	NATTS TAD Section 5.3.1.4
Data Completeness*	Quarterly	>85%	NATTS TAD Table 3.2-5



Questions?

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OR... Ask these guys!

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