Reviewing and Assessing NATTS/PAMS Data

An Overview of Data Review, Verification, and Validation

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National Ambient Air Monitoring Conference

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Topics

1. Data Review Process
    - Define verification and validation
    - Roles and responsibilities

2. Verification and Validation Steps
    - Review of Data Records
    - Examples

3. A Recap from Recent Audits
    - Audit of Data Quality
    - Parameter Specific Tips

Data Verification and Validation Overview

Air Monitoring Quality Paradigm

Plan

- Quality Systems Documents
  - QAPP
  - QMP
  - Monitoring Network Plan

Do

- Monitoring Activities
- SOPs
- Data Generation
- Data Verification & Validation

Check

- Technical Assessments
- Internal/External Audits
- Ongoing Demonstration of Capabilities

Act

- Take action to continually improve performance
- Develop/improve standardized methods for improvement

Two Key Elements of the Data Review Process

Step 1: Verification

Are you collecting the data correctly?

Evaluates the completeness, correctness, and conformance of data against method, procedural, and/or contractual specifications.

Goal is to ensure and document that the reported results reflect the activities performed and measurements acquired

Step 2: Validation

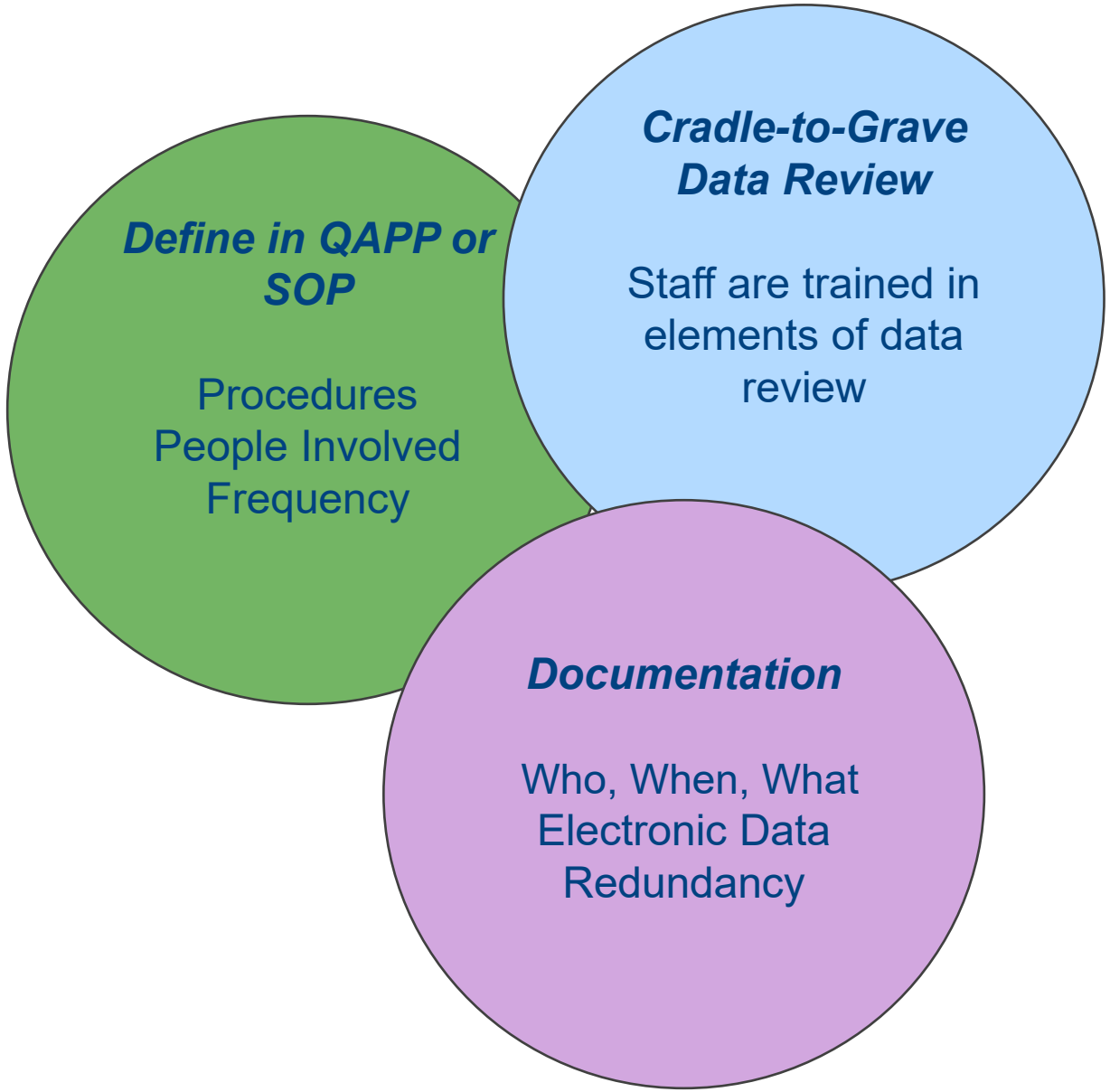
Are you collecting the correct data?

Examination that the particular requirements for a *specific intended use* (i.e., monitoring objectives) are fulfilled.

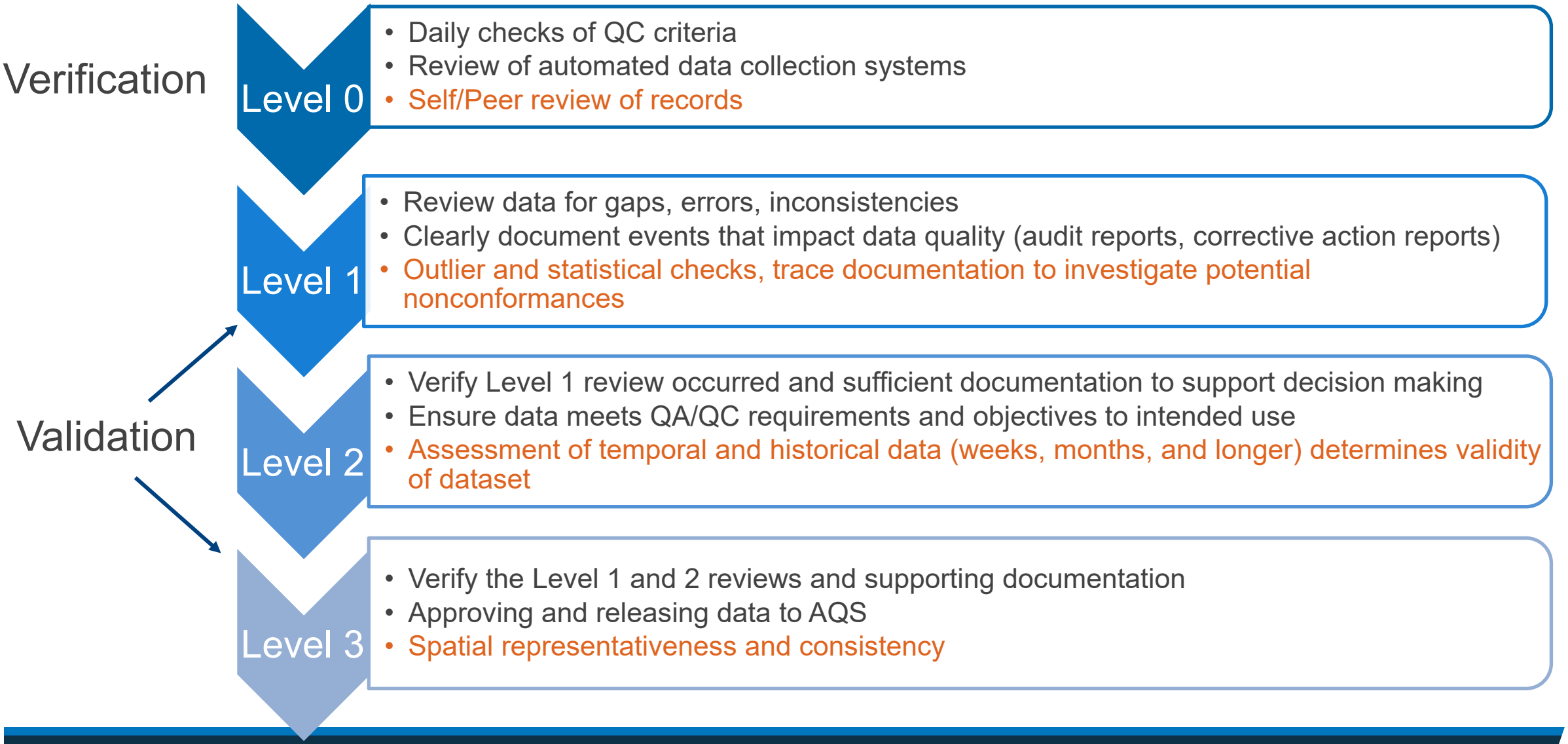
Goal is to determine the quality of the data relative to the end use.

What is your role?

- Analysts and site operators perform self review routinely to verify recorded information
- QA Staff perform technical review to verify data meet acceptance criteria, documentation is complete, flags are appropriately applied
- Data validators perform higher level data analysis to ensure data are comparable, accurate, and defensible

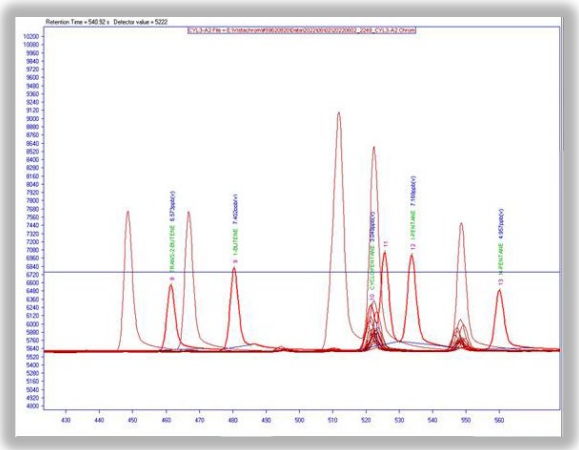


Tiered Data Review Approach



Data Verification

- Data review occurring frequently and comprehensively
  - Data are complete and correct
  - Data were collected according to SOP
  - Data meet acceptance criteria
  - Calibrations and calculations correct
  - QC samples meet criteria
    - Precision
    - Blanks
    - Standards



- Specific verification activities prescribed in SOP(s)
  - Verify these processes are followed and documented
- Incorporates self-review and peer review
- Activities are to be documented
- Opportunity to correct, qualify, or invalidate data

Data Validation

Occurs after data verification

- Incorporates examining many data sources
- Data validators independent from those collecting and verifying data
- Compares data over time for consistency and reasonability
- Record the:
  - Software tools
  - Products prepared
  - Responsible individuals

Validation Tools

More information on validation tools is available in the PAMS Webinar Series
Contact Corey Mocka or Berkley Hillis at EPA OAQPS to access

- AirVision
- DART
- DR DAS (Envidas and Evista)
- Open-Source Data Tools
  - R, Rstudio, and Rmarkdown, R OpenAir
  - R Shiny Dashboards
  - EPA AQS API
  - EPA PAMS Dashboard

Benefits of Data Validation Software

- Customizable to meet data needs
- Automates review process
- Continuous and non-continuous methods

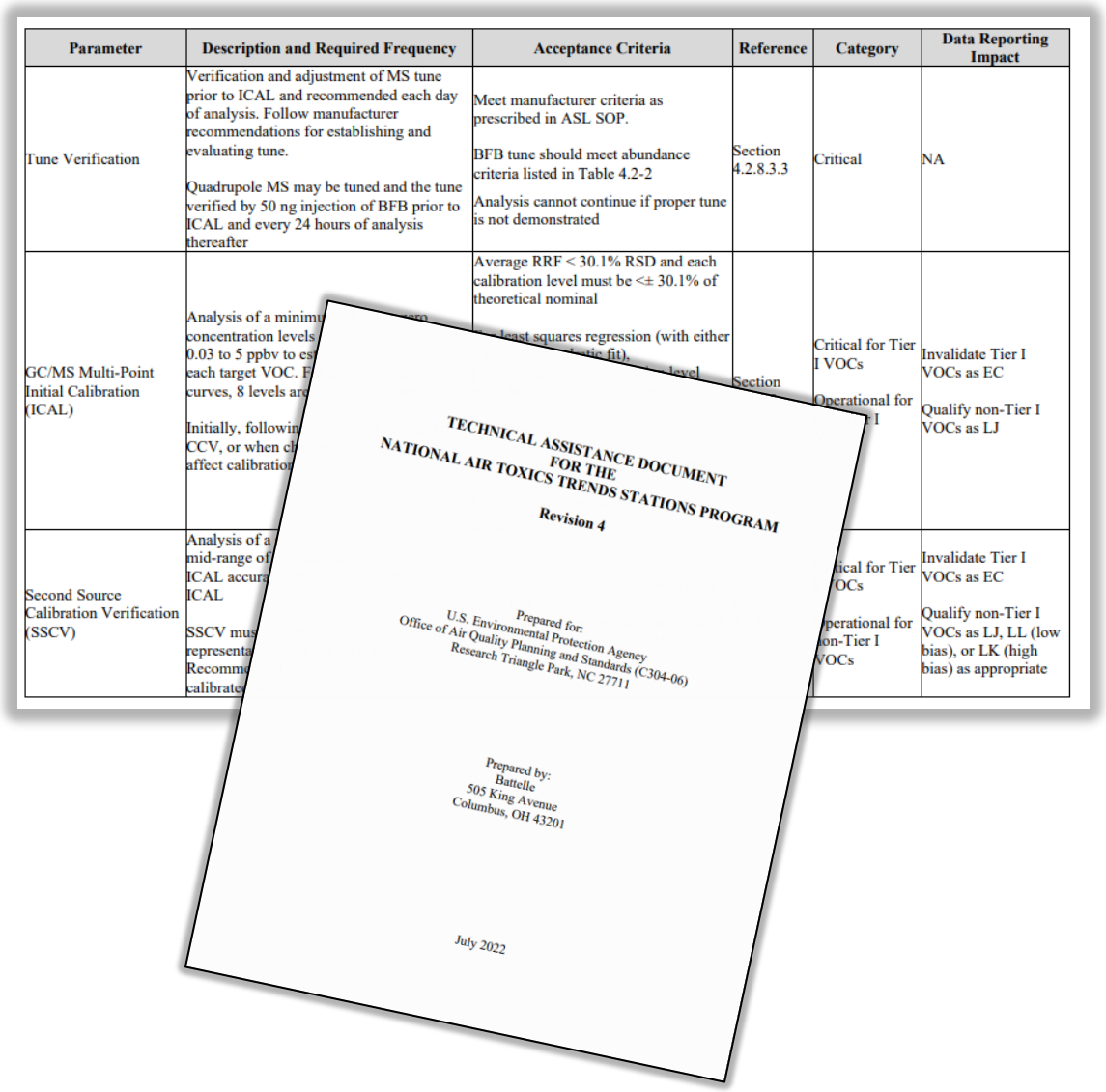
Data Review Checklists

Benefits

- Simplifies complex data reviews
- Can be used as a training tool
- Provides documentation

Developing Checklists

- Reference Documents Contain Validation Tables



Parameter	Description and Required Frequency	Acceptance Criteria	Reference	Category	Data Reporting Impact
Tune Verification	Verification and adjustment of MS tune prior to ICAL and recommended each day of analysis. Follow manufacturer recommendations for establishing and evaluating tune. Quadrupole MS may be tuned and the tune verified by 50 ng injection of BFB prior to ICAL and every 24 hours of analysis thereafter	Meet manufacturer criteria as prescribed in ASL SOP. BFB tune should meet abundance criteria listed in Table 4.2-2 Analysis cannot continue if proper tune is not demonstrated	Section 4.2.8.3.3	Critical	NA
GC/MS Multi-Point Initial Calibration (ICAL)	Analysis of a minim concentration levels 0.03 to 5 ppbv to es each target VOC. F curves, 8 levels ar Initially, followin CCV, or when c affect calibratio	Average RRF < 30.1% RSD and each calibration level must be <± 30.1% of theoretical nominal least squares regression (with either fit), level	Section	Critical for Tier I VOCs Operational for I	Invalidate Tier I VOCs as EC Qualify non-Tier I VOCs as LJ
Second Source Calibration Verification (SSCV)	Analysis of a mid-range of ICAL accura ICAL SSCV mus representa Recomm calibrate			ical for Tier OCs perational for on-Tier I OCs	Invalidate Tier I VOCs as EC Qualify non-Tier I VOCs as LJ, LL (low bias), or LK (high bias) as appropriate

TECHNICAL ASSISTANCE DOCUMENT
FOR THE
NATIONAL AIR TOXICS TRENDS STATIONS PROGRAM
Revision 4

Prepared for:
U.S. Environmental Protection Agency
Office of Air Quality Planning and Standards (C304-06)
Research Triangle Park, NC 27711

Prepared by:
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505 King Avenue
Columbus, OH 43201

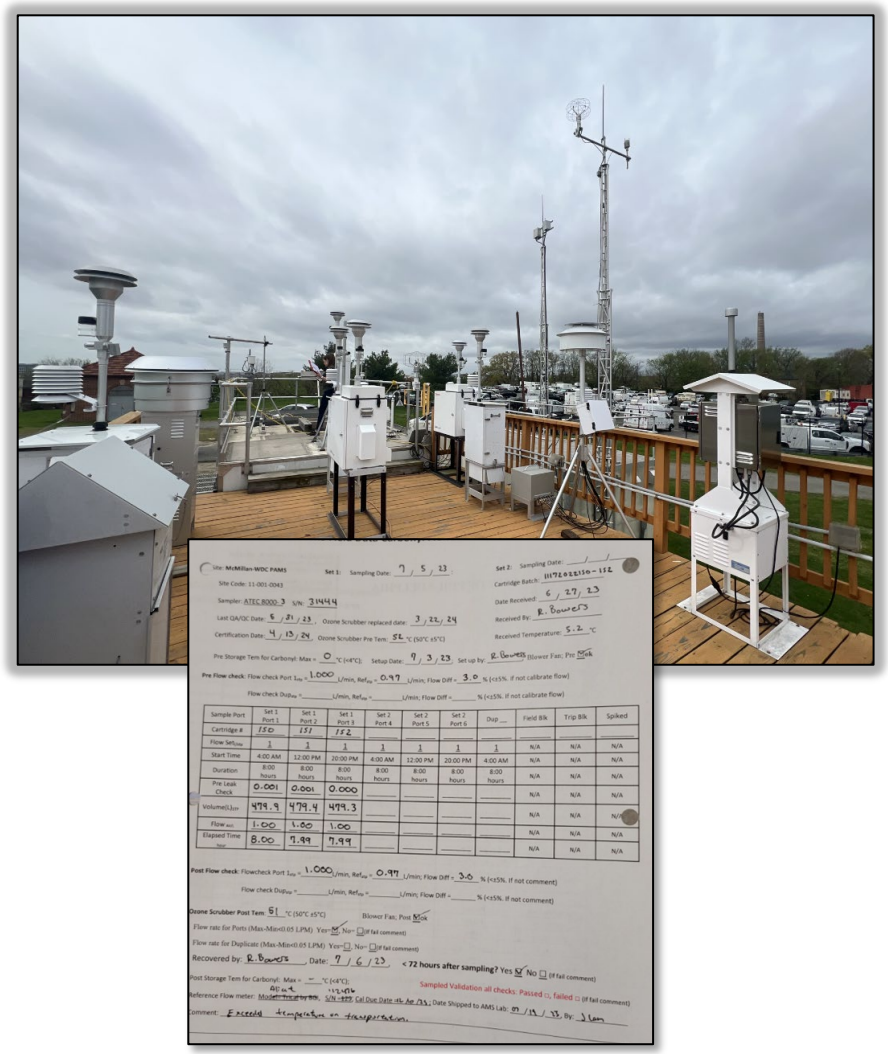
July 2022

Verification Steps

Step 1: Verify Sample Information

Monitoring Site

- Sample collection forms
  - Ensure the proper information is recorded (not overlooked)
  - Dates match sampling calendar
  - Sample volumes are correct
  - Routine maintenance and unusual conditions are recorded
  - Refrigerated storage
- Sampler operation
  - Reasonability checks for running samplers
  - Error codes



Step 1: Verify Sample Information

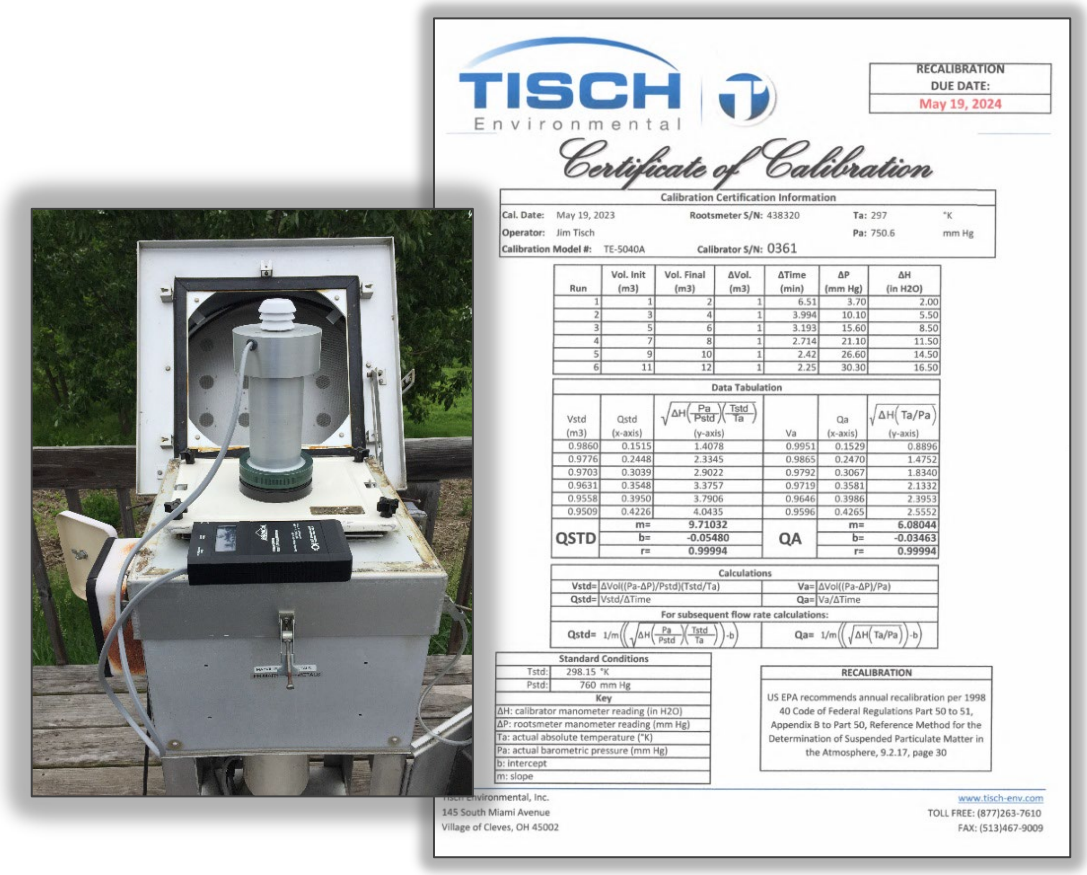
Laboratory

- Sample receipt forms
  - Ensure the proper information is recorded
  - Receipt temperature, pressure, and hold times
- Sample preparation records
  - Digestion and extraction batches
- Support equipment calibration forms
  - Thermometers
  - Mechanical pipettes
  - Analytical balances
  - Mass flow controllers

Step 2: Review Calibrations

Monitoring Site

- Verify calibration of equipment
  - Ensure proper information recorded and criteria met
  - Orifice Certifications and Calibration
  - NIST traceable flow standard Certification and Flowrate Verification
- Verify calculations
  - Manual check of automated functions and spreadsheets



TISCH Environmental

RECALIBRATION DUE DATE: May 19, 2024

Certificate of Calibration

Calibration Certification Information

Cal. Date: May 19, 2023 Rootsmeter S/N: 438320 Ta: 297 °K
Operator: Jim Tisch Pa: 750.6 mm Hg
Calibration Model #: TE-5040A Calibrator S/N: 0361

Run	Vol. Init (m3)	Vol. Final (m3)	ΔVol. (m3)	ΔTime (min)	ΔP (mm Hg)	ΔH (in H2O)
1	1	2	1	6.51	3.70	2.00
2	3	4	1	3.994	10.10	5.50
3	5	6	1	3.193	15.60	8.50
4	7	8	1	2.714	21.10	11.50
5	9	10	1	2.42	26.60	14.50
6	11	12	1	2.25	30.30	16.50

Data Tabulation

Vstd (m3)	Qstd (x-axis)	$\sqrt{\Delta H\left(\frac{Pa}{Pstd}\right)\left(\frac{Tstd}{Ta}\right)}$ (y-axis)	Va	Qa (x-axis)	$\sqrt{\Delta H\left(\frac{Ta}{Pa}\right)}$ (y-axis)
0.9860	0.1515	1.4078	0.9951	0.1529	0.8896
0.9776	0.2448	2.3345	0.9865	0.2470	1.4752
0.9703	0.3039	2.9022	0.9792	0.3067	1.8340
0.9631	0.3548	3.3757	0.9719	0.3581	2.1332
0.9558	0.3950	3.7906	0.9646	0.3986	2.3953
0.9509	0.4226	4.0435	0.9596	0.4265	2.5552
QSTD	m=	9.71032	QA	m=	6.08044
	b=	-0.05480		b=	-0.03463
	r=	0.99994		r=	0.99994

Calculations

Vstd= $\Delta Vol(Pa-\Delta P)/Pstd)(Tstd/Ta)$ Va= $\Delta Vol((Pa-\Delta P)/Pa)$
Qstd= Vstd/ΔTime Qa= Va/ΔTime

For subsequent flow rate calculations:

Qstd= $1/m\left(\left(\sqrt{\Delta H\left(\frac{Pa}{Pstd}\right)\left(\frac{Tstd}{Ta}\right)}\right)-b\right)$ Qa= $1/m\left(\left(\sqrt{\Delta H\left(\frac{Ta}{Pa}\right)}\right)-b\right)$

Standard Conditions
Tstd: 298.15 °K
Pstd: 760 mm Hg

Key
ΔH: calibrator manometer reading (in H2O)
ΔP: rootsmeter manometer reading (mm Hg)
Ta: actual absolute temperature (°K)
Pa: actual barometric pressure (mm Hg)
b: intercept
m: slope

RECALIBRATION

US EPA recommends annual recalibration per 1998 40 Code of Federal Regulations Part 50 to 51, Appendix B to Part 50, Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere, 9.2.17, page 30

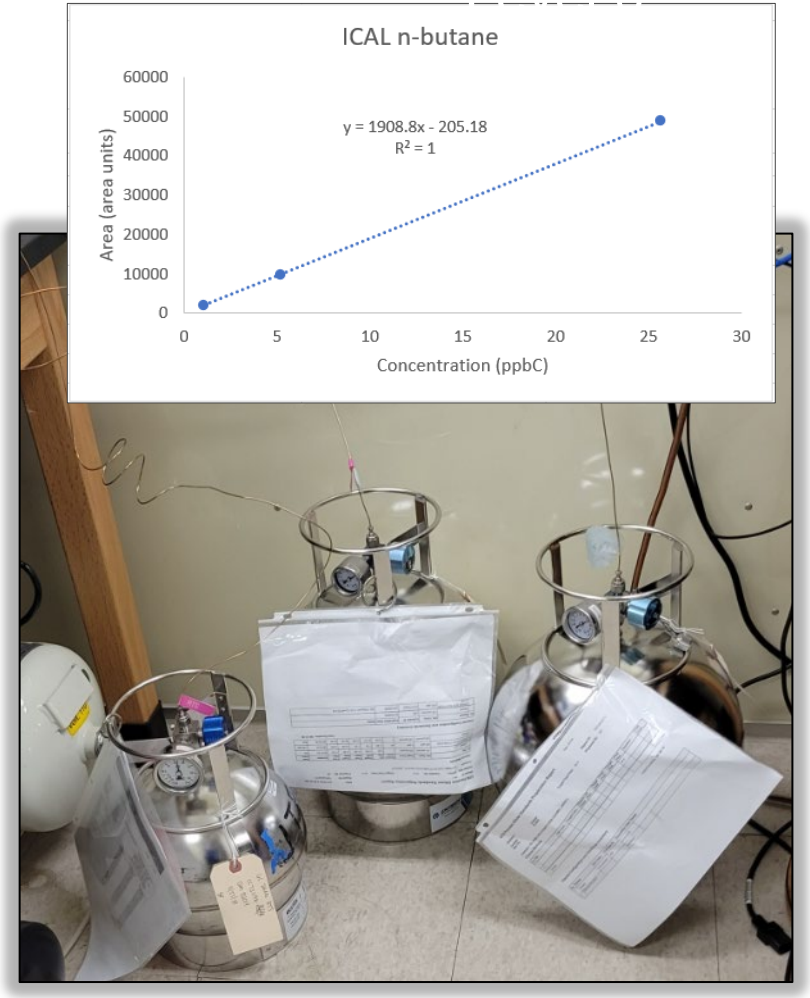
Tisch Environmental, Inc.
145 South Miami Avenue
Village of Cleves, OH 45002

www.tisch-env.com
TOLL FREE: (877)263-7610
FAX: (513)467-9009

Step 2: Review Calibrations

Laboratory

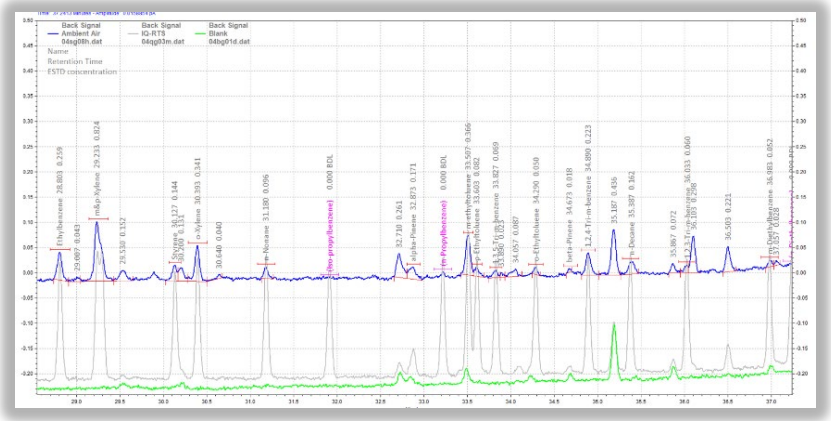
- Verify calibration inputs from the COA
- Review records for standard preparation
- Review calibration curves
- Verify acceptance criteria
  - Response factors, retention times
- Manual calculation to confirm spreadsheet or automated calculations



Step 3: Review Raw and QC Data

Raw Data

- LIMS outputs
  - Manual calculation verification
- Manual integrations
- MDLs
- Compound identification based on retention time



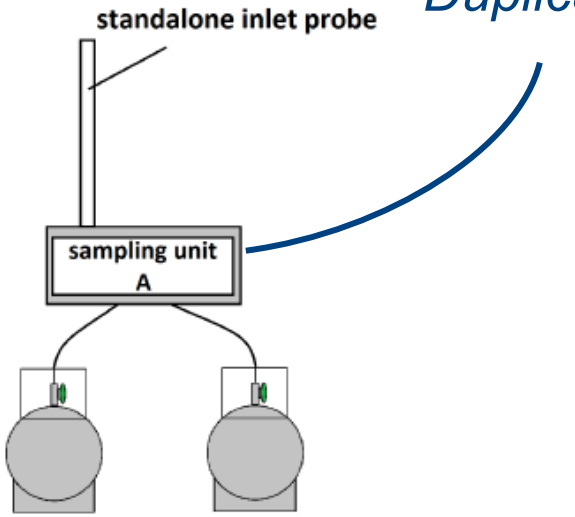
QC Data

- QA Flow Audits
  - Separate flow transfer standards, equipment
  - Independent auditor performs routine flow check
- Blanks
  - Field blanks, lot blanks, laboratory blanks
- Precision Data

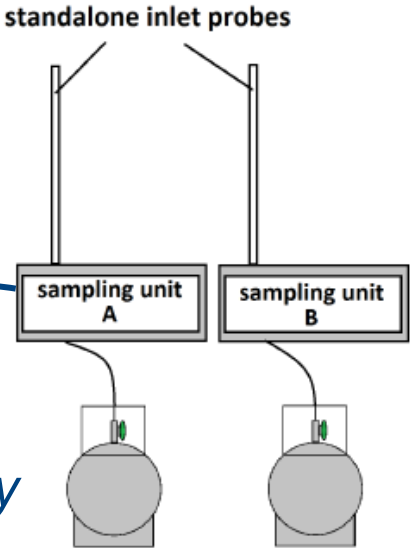
Step 3: Review QC Data

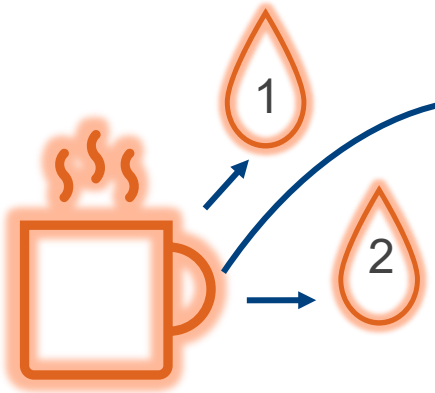
Precision

Duplicate: Single Pathway



Collocated: Separate Pathway





Replicate:
Multiple Injections from Same Sample

Step 4: Apply Qualifiers

Laboratory & monitoring site agency work together to ensure all qualifiers are applied

- Review corrective action reports
  - Unresolved CARs that impact data
- Review maintenance records
- Generate AQS strings and perform independent review for accuracy
  - Automatically or manually generated?
    - Independent review to confirm manual
    - Routine verification of automatic process

Validation Examples

Data validation activities build on the data verification process

- Should only be conducted on data which have gone through data verification
- May identify data which require further investigation which may include repeating some steps of the data verification process
- Examination of the data set for internal, historical, and spatial consistency

NATTS Data Validation Tables

Refer to Section 7.0 of the NATTS TAD

Field

Parameter	Description and Required Frequency	Acceptance Criteria	Reference	Category	Data Reporting Impact
Sampling Unit Clock/Timer Check	Verified with each sample collection event	Clock/timer accurate to ±5 minute of reference for digital timers and ±15 minutes for mechanical timers, set to local standard time Sample collection period verified to be midnight to midnight	Section 4.3.8.1.1	Operational	NA
Sampling Unit Leak Check	Pressurization or evacuation of internal sampler flow paths to demonstrate as leak-free At setup of each sampling event Sampling cannot commence if leak check fails. (*strongly recommended*)	No indicated flow or manufacturer criteria for passing leak check	Section 4.3.8.1.3	Critical if sampler is so equipped	Invalidate data collected with failing leak check as AQ
Sampling Frequency	One sample every six days according to the EPA National Monitoring Schedule	Sample must be valid or a make-up sample should be scheduled (refer to Section 2.1.2.1)	Section 4.3.8.1.1	MQO	NA
Sampling Period	All routine primary, duplicate, and collocated field-collected samples	1380-1500 minutes (24 ± 1 hr) starting and ending at midnight	Sections 2.1.1 and 4.3.8.1.1	Critical and MQO	Invalidate all measurement for sample as AG
Pre-Sample Collection Purge	*Strongly recommended* with each sampling event	Minimum of ten air changes just prior to sample collection	Section 4.3.8.1.2	Practical	NA
Sample Receipt					
Chain-of-custody	All field-collected samples	Each cartridge must be uniquely identified and accompanied by a valid and legible COC with complete sample documentation	Sections 3.3.1.3.7 and 4.3.8.1.4	Critical	Invalidate measurement data as EC
Sample Holding Time	All field-collected samples, laboratory QC samples, and standards	Extraction: 14 days from sample collection (cartridge storage ≤ 4 °C) Analysis: 30 days from extraction (extract storage ≤ 4 °C)	Section 4.3.9.3	Operational	Qualify affected data exceeding holding time or storage conditions as LJ

Lab

Parameter	Description and Required Frequency	Acceptance Criteria	Reference	Category	Data Reporting Impact
Sample Receipt Temperature Check	Field collected cartridges placed in refrigerated storage at retrieval, within 72 hours of end of sample collection. Shipment temperature measured upon receipt at the laboratory	Cartridge temperature ≤ 4°C or Short term shipments (shipments < 4 hours from retrieval) < 10°C	Section 4.3.8.1.4	Operational	Qualify affected data exceeding shipment temperature conditions as LJ
HPLC Analysis					
Solvent Blank (SB)	Prior to ICAL and daily beginning CCV Analysis must not continue if criteria are not met	All target compounds < MDL_{sp} (refer to Section 4.1.3.1) or $s \cdot K$ (refer to Section 4.1.3.2)	Sections 4.3.9.5 and 4.3.9.5.2	Critical for Tier I carbonyls Operational for non-Tier I carbonyls	Invalidate Tier I carbonyls as EC Qualify non-Tier I carbonyls as LJ
HPLC Initial Multi-Point Calibration (ICAL)	Initially, following failed CCV, or when changes to the instrument affect calibration response Calibration modeled as linear least-squares regression Injection of a minimum of 5 points covering approximately 0.01 to 3.0 µg/mL (concentration range prescribed by ASL)	Correlation coefficient (r) ≥ 0.999; relative error for each level against calibration curve < ±20.1% of theoretical nominal. Absolute value of intercept divided by slope must not exceed MDL_{sp} (MDLs determined by Section 4.1.3.1) or $s \cdot K$ (MDLs determined by Section 4.1.3.2)	Sections 4.3.9.5 and 4.3.9.5.2	Critical for Tier I carbonyls Operational for non-Tier I carbonyls	Invalidate Tier I carbonyls as EC Qualify non-Tier I carbonyls as LJ
Second Source Calibration Verification (SSCV)	Second source standard prepared at the mid-range of the calibration curve, analyzed immediately after each ICAL	<±15.1% difference from theoretical nominal	Sections 4.3.9.5 and 4.3.9.5.3	Critical	Invalidate Tier I carbonyls as EC Qualify non-Tier I carbonyls as LJ
Continuing Calibration Verification (CCV)	Once ICAL is established, prior to sample analysis on days when an ICAL is not performed and minimally every 12 hours of analysis; recommended following analysis of every 10 field-collected samples and at the conclusion of each analytical sequence	<±15.1% difference from theoretical nominal	Sections 4.3.9.5 and 4.3.9.5.4	Critical	Invalidate Tier I carbonyls as EC Qualify non-Tier I carbonyls as LJ
Extraction Solvent Method Blank (ESMB)	An aliquot of extraction solvent delivered to a volumetric flask. One with each extraction batch of 20 or fewer field-collected samples.	Each target carbonyl's concentration < MDL_{sp} (refer to Section 4.1.3.1) or $s \cdot K$ (refer to Section 4.1.3.2)	Section 4.3.9.4.1	Operational	Qualify affected data as LB and QX

NATTS Data Validation Tables

TAD Revision 4, Section 7.0

Critical

Criteria must be met for reported results to be valid. If not met, samples are invalidated.

Parameter	Description and Required Frequency	Acceptance Criteria	Reference	Category	Data Reporting Impact
Cartridge Lot Blank Check	Analysis of a minimum of 3 cartridges or 1% of the total lot, whichever is greater, for each new lot Report the lot blank value to AQS for each new lot of cartridge media	Formaldehyde < 0.15 µg/cartridge, Acetaldehyde < 0.10 µg/cartridge, Acetone < 0.30 µg/cartridge, all other individual target carbonyls < 0.10 µg/cartridge	Section 4.3.5.1 and Table 4.3-2	Critical	Invalidate affected compounds as EC

NATTS Data Validation Tables

TAD Revision 4, Section 7.0

MQO

(Measurement Quality Objective)

Required NATTS Measurement Quality Objective which must be attained.

If not met, does not necessarily invalidate data, but may compromise data and result in exclusion from trends analysis.

Parameter	Description and Required Frequency	Acceptance Criteria	Reference	Category	Data Reporting Impact
Sampling Frequency	One sample every six days according to the EPA National Monitoring Schedule	Sample is to be valid or a make-up sample should be scheduled (refer to Section 2.1.2.1)	Section 4.2.3.4.1	MQO	NA

NATTS Data Validation Tables

TAD Revision 4, Section 7.0

Operational

Failure to meet criteria does not invalidate reported results; results are compromised and may require qualification on a case-by-case basis; guidance on qualifiers is provided in the tables.

Parameter	Description and Required Frequency	Acceptance Criteria	Reference	Category	Data Reporting Impact
Sample Receipt Temperature Check	Field collected cartridges placed in refrigerated storage at retrieval, within 72 hours of end of sample collection. Shipment temperature measured upon receipt at the laboratory	Cartridge temperature ≤ 4°C or Short term shipments (shipments < 4 hours from retrieval) < 10°C	Section 4.3.8.1.4	Operational	Qualify affected data exceeding shipment temperature conditions as LJ

NATTS Data Validation Tables

TAD Revision 4, Section 7.0

Practical

If not met, does not invalidate reported results. Results may be compromised but do not require qualification.

Parameter	Description and Required Frequency	Acceptance Criteria	Reference	Category	Data Reporting Impact
ICP/MS Warm Up	Warm up of ICP torch and MS detector Each day of analysis	Minimum of 30 minutes (or according to manufacturer specifications) prior to performing initial calibration	Section 4.4.11.6	Practical	NA

Create Visualizations to Assess Trends

Internal Consistency

- Validation of outlier data
- Investigate one-point maximum outliers
- Co-contaminant trends
  - e.g., things that trend together, things that trend opposites

Geographical Trends

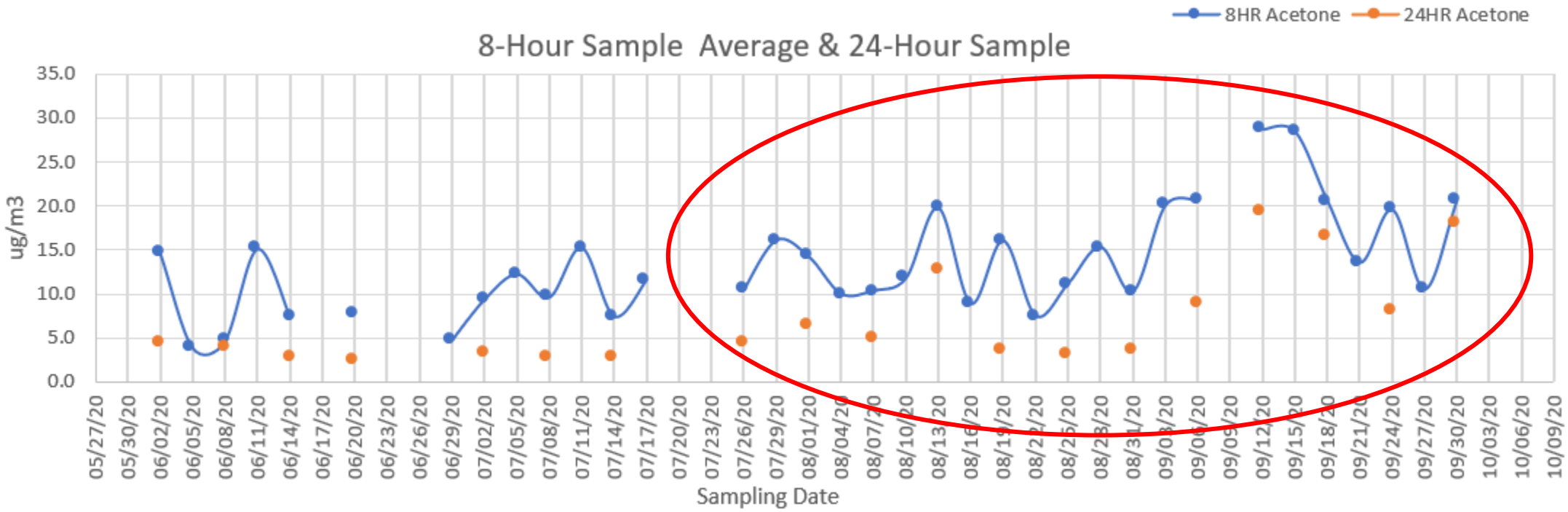
- Identical or similar technologies
- Local or regional trends
- Validation of outlier data

Historical Trends

- Weekly, Monthly, and Longer
- Seasonal trends
- Annual comparisons

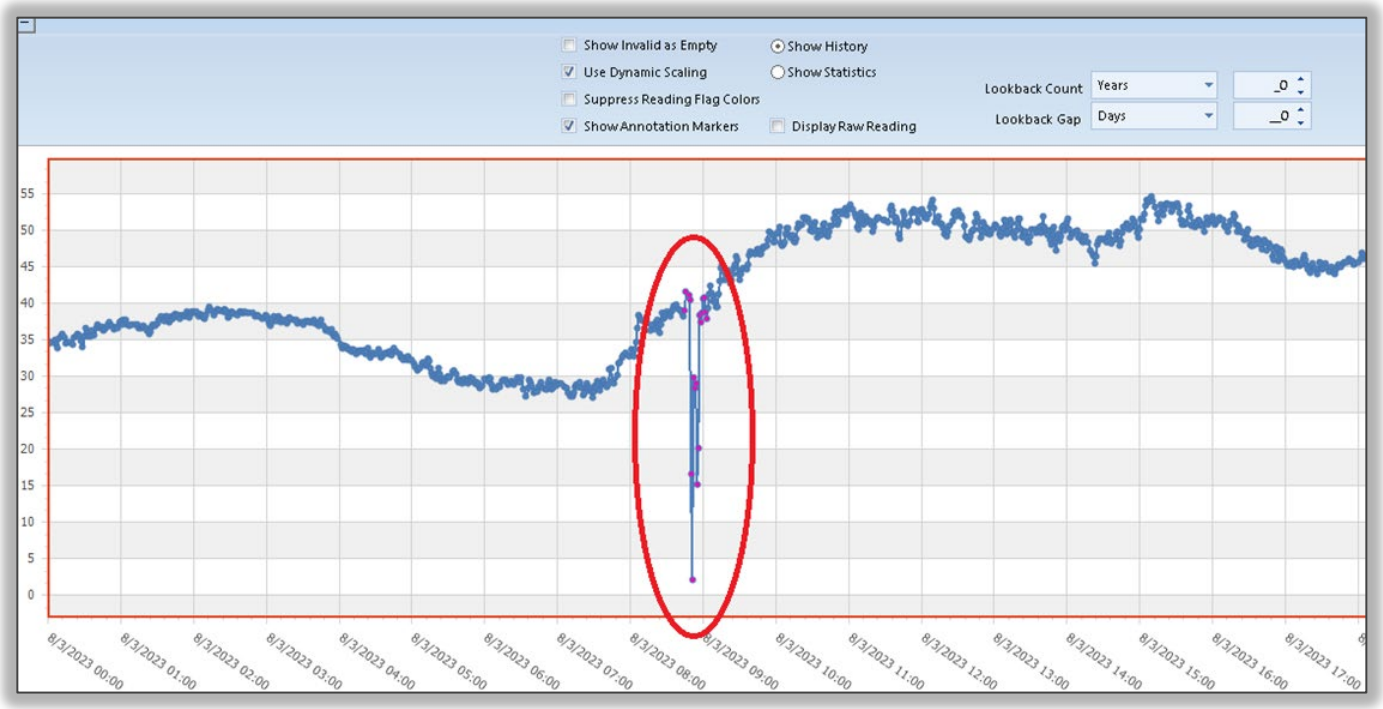
Reasonability Checks

Does the data make sense?

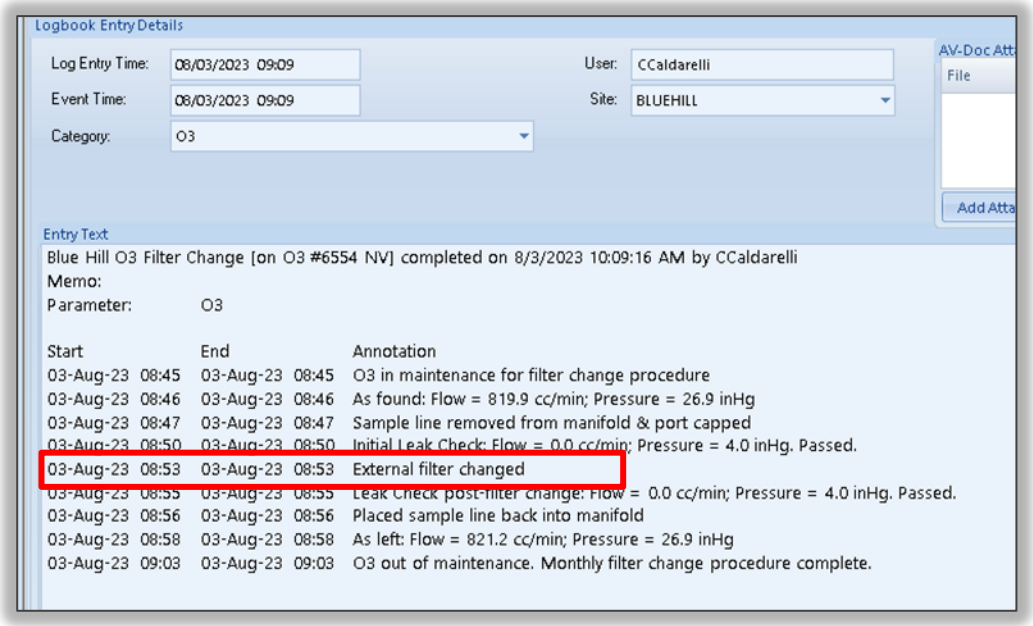


Reasonability Checks

Does the data make sense?



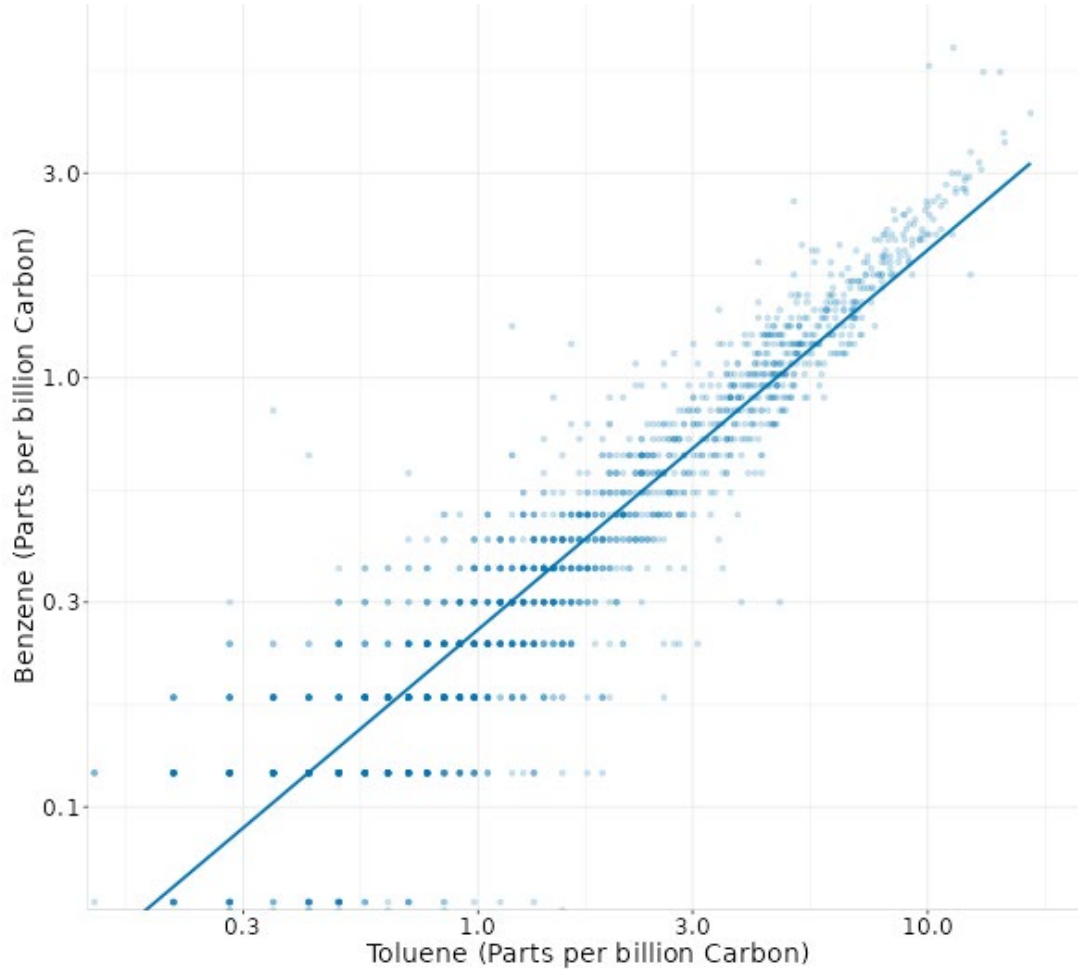
Verify suspect data against logbooks and other records



If it's not documented, did it really happen?

PAMS

Visualizing Data to Identify Issues



- Review and verify data frequently
  - ~1400 data points each day
- Configure data processing method to minimize manual data processing
  - Identification
  - Integration
- Check previous day's data each day
  - QC samples
  - Minimally 3 ambient hours
  - Expected data files recorded
  - Catches problems before losing data

Control Charting

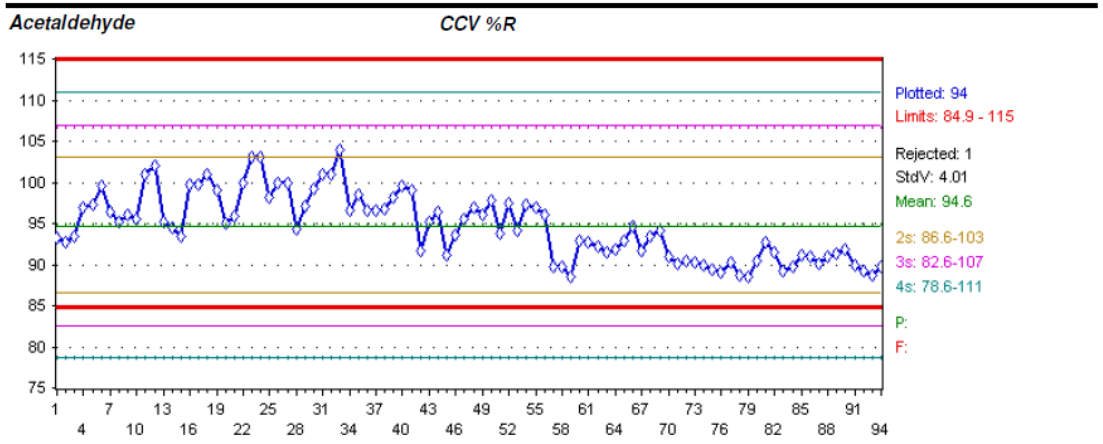
- Laboratory Quality Control Charting
  - NATTS TAD Rev. 4, Section 6.5 *In-House Control Limits*
  - ±2 Standard Deviations – Warning Limits
    - Exceedance should prompt monitoring
    - Troubleshooting, corrective actions should be taken for repeated exceedance
  - ±3 Standard Deviations – Control Limits
    - Troubleshooting, corrective action is required when exceeded
- Proficiency Testing Charting
  - NATTS TAD Rev. 4, Section 2.1.4.1 *Assessing Laboratory Bias – Proficiency Testing*
- Control charting to understand when maintenance is required

Control Charting

Control charts of laboratory QC

Project: All Projects

Prepared By: All Extractionists
Analyzed By: All Analysts
Extractions: TO-11A extraction



Rjct	Sample ID	Prepared	Analyzed	Spike Level	Result	%R	Limits	Qualifiers
	2304002-CCV1		4/1/23	0.15 ppbv	0.145	97.0	84.9-115.1	
	2304001-CCV1		4/1/23	0.15 ppbv	0.140	93.2	84.9-115.1	
	2304001-CCV2		4/1/23	0.15 ppbv	0.139	92.7	84.9-115.1	
	2304002-CCV2		4/1/23	0.15 ppbv	0.146	97.2	84.9-115.1	
	2304001-CCV3		4/2/23	0.15 ppbv	0.140	93.4	84.9-115.1	
	2304002-CCV3		4/2/23	0.15 ppbv	0.149	99.6	84.9-115.1	
	2304012-CCV1		4/6/23	0.15 ppbv	0.145	96.4	84.9-115.1	
	2304012-CCV2		4/6/23	0.15 ppbv	0.143	95.2	84.9-115.1	
	2304012-CCV3		4/6/23	0.15 ppbv	0.144	96.1	84.9-115.1	
	2304031-CCV1		4/13/23	0.15 ppbv	0.143	95.1	84.9-115.1	
	2304031-CCV2		4/13/23	0.15 ppbv	0.142	94.4	84.9-115.1	
	2304031-CCV3		4/13/23	0.15 ppbv	0.140	93.5	84.9-115.1	
	2304030-CCV1		4/14/23	0.15 ppbv	0.143	95.5	84.9-115.1	
	2304030-CCV2		4/14/23	0.15 ppbv	0.152	101	84.9-115.1	
	2304030-CCV3		4/14/23	0.15 ppbv	0.154	102	84.9-115.1	
	2304046-CCV1		4/18/23	0.15 ppbv	0.150	99.8	84.9-115.1	

- Quarterly tracking of laboratory QC samples
  - Continuing Calibration Verifications (CCV) – Analyzed at beginning, middle, and end of every sequence
  - Internal Standards (IS) – Either deuterated isotopes or target analytes or analytes that behave similarly to, but are not, targets.
  - Blanks and Blank Spikes
- Results assessed against acceptance criteria ranges and standard deviations from the mean

Control Charting

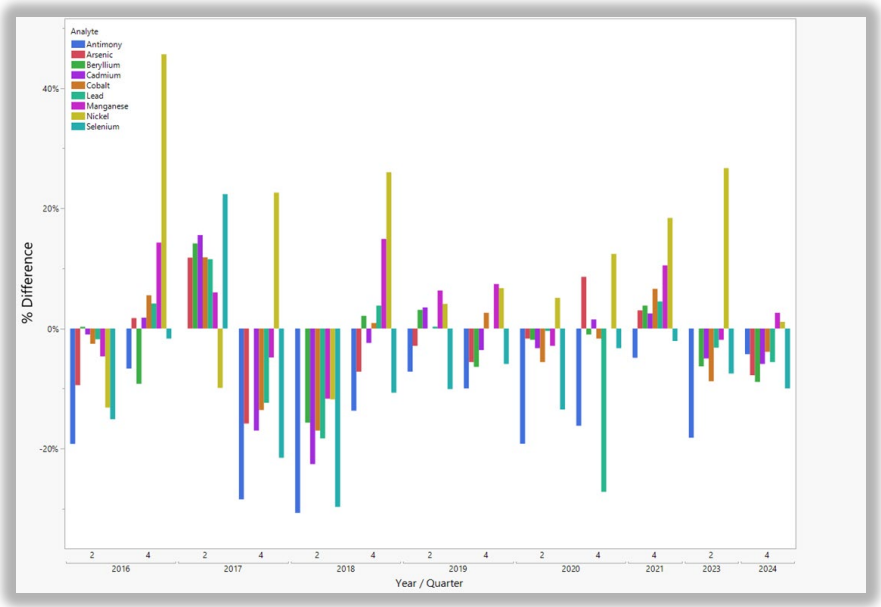
Track PT performance

NATTS TAD Requirements:

When two consecutive failed PTs for a given analyte, measurement data for any analyte(s) are to be qualified when reported to AQS.

QA Qualifier LK or LL indicating a high or low bias, respectively

Analyte	2023				2022	2021		2020	
	QTR 4	QTR 3	QTR 2	QTR 1	QTR 1	QTR3	QTR1	QTR 3	QTR 1
1,1,2,2-Tetrachloroethane	-3.1%	284.2%	11.1%	9.6%	15.3%	-29.2%	-0.6%	7.9%	22.2%
1,2-Dibromoethane	23.8%	114.2%	32.3%	12.4%	26.6%	-36.2%	-9.7%	16.3%	11.4%
1,2-Dichloroethane	13.9%	37.0%	10.2%	-4.4%	11.9%	-23.9%	19.6%	3.2%	7.9%
1,2-Dichloropropane	8.6%	9.5%	-0.4%	-1.3%	14.3%	-16.7%	12.7%	12.6%	31.4%
1,3-Butadiene	-11.7%	4.7%	-26.0%	-16.3%	1.3%	-47.6%	-22.8%	-10.0%	-5.4%
1,3-Dichloropropene - cis	-11.2%	12.9%	18.6%	8.8%	24.3%	-30.9%	-6.8%	7.5%	14.6%
1,3-Dichloropropene - trans	-13.7%	42.0%	41.4%	21.7%	42.1%	-29.3%	-11.0%	22.4%	21.4%
*Acrolein	-56.3%	F NEG	F NEG	92.9%	157.8%	59.4%	33.8%	NR	18.8%
Benzene	24.2%	26.7%	11.8%	16.1%	64.9%	-15.8%	11.2%	8.0%	23.1%
Carbon Tetrachloride	-6.2%	-6.8%	-12.3%	-6.4%	9.2%	-30.0%	22.7%	18.2%	43.8%
Chloroform	7.4%	2.9%	-2.9%	-7.5%	8.8%	-26.7%	-1.7%	-3.2%	7.2%
Dichloromethane	3.4%	7.5%	28.1%	-2.6%	17.7%	-18.8%	12.5%	6.1%	17.1%
*Ethylene Oxide	96.7%	16.7%	-40.5%	143.5%	68.6%	7.7%	-16.7%	24.5%	19.6%
Tetrachloroethylene	7.5%	52.3%	1.5%	-8.0%	-4.4%	-33.3%	-6.7%	0.5%	13.4%
Trichloroethylene	27.0%	44.7%	1.6%	-0.7%	11.0%	-28.3%	4.7%	-9.1%	11.9%
Vinyl Chloride	-35.4%	-7.5%	-9.6%	-6.7%	17.1%	-31.5%	14.8%	2.2%	3.8%



Lessons Learned from Technical Systems Audits

Audit of Data Quality

What?

Independent review of reported data from cradle to grave

How?

Select a result for an example parameter in AQS and trace the result back to the measurement and instrument calibration

Who?

QA Managers/Staff independent of data generation

Records Assessed During an ADQ

Monitoring Site Records and Data

Evidence of data verification is ALWAYS part of an ADQ!

- Review sample collection information:
  - Dates: Set up date, sampling date, collection date
  - Sampling Media: initial canister pressure, media hold times, canister cleaning date, date of PUF surrogate addition
  - Sample Volume: Flow rates, elapsed run time
  - Supporting Records: Electronic run file to confirm any errors, logbooks
  - Sample Custody: Receipt and transfer dates, storage/shipping temperature
- Review sampler information:
  - Sampler certifications
  - Calibration, flow rate verification, independent flow rate audit
  - Maintenance

Records Assessed During an ADQ

Laboratory Records and Data

Evidence of data verification is ALWAYS part of an ADQ!

- Sample Custody:
  - Receipt date
  - Condition upon receipt (temperature, damage, final canister pressure)
  - Data input from the COC (IDs and sample volume)
- Media Preparation (Before shipment to the site)
  - Canister qualifications, canister cleaning batch evaluation
  - PAH surrogates
- Sample and Standard Preparation Batch:
  - QC samples included with the extraction/digestion batch and their results
  - Hold time requirements
  - Review of standards including calculations, certificate of analysis, and results

Records Assessed During an ADQ

Laboratory Records and Data

Evidence of data verification is ALWAYS part of an ADQ!

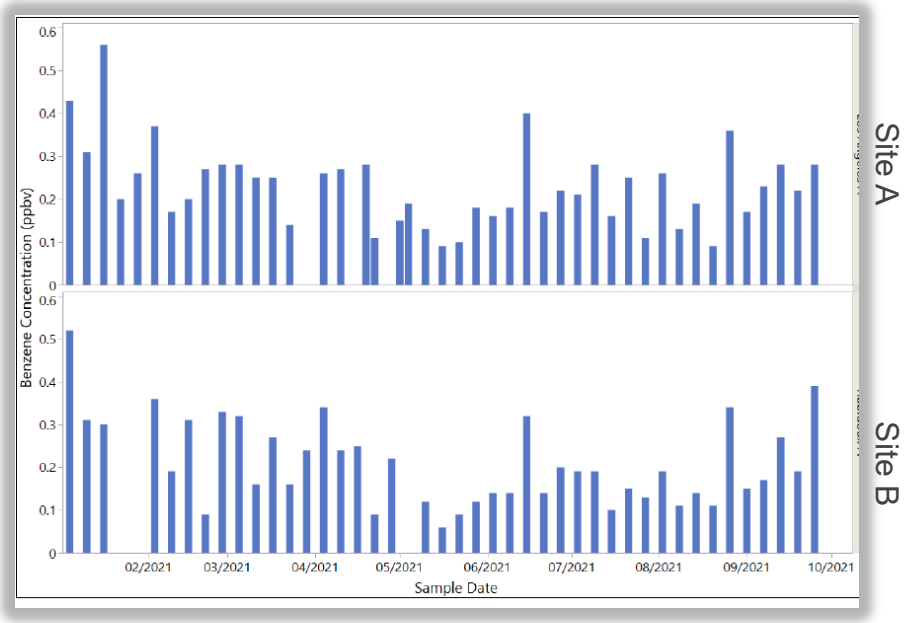
- Analytical Results:
  - Calibration curves
    - How many standard levels are used?
    - How was the curve evaluated and is it acceptable?
      - Retention Times
  - QC and precision samples
    - Continuing Calibration Verification (CCV)
    - Laboratory Control Samples (LCS)
    - Blanks, replicates, duplicates
  - MDL
    - How were MDLs determined?
    - Do MDLs meet NATTS (or PAMS) MQO?
  - Chromatography review to assess peak identification and manual integrations
  - Flagged data investigation
  - Lot Blanks
  - Calculations and confirmation of automated functions

Records Assessed During an ADQ

How does the monitoring agency handle laboratory results?

Evidence of data verification is ALWAYS part of an ADQ!

- How are laboratory reports stored?
  - Don’t rely on emails
- What verification and validation activities are performed?
  - Comparison of lab results to field records
  - Sample collection
  - Sampler information
- AQS strings
  - Who generates? Who verifies?
    - Manual entry or automated function



Verification of a Laboratory Report

Performed by the Monitoring Agency

Summary of Results

Description: [redacted]
Matrix: Air
Comments:

Lab ID: [redacted]
Sample Volume: 481.9 L
Cartridge Pouch #: 21G5681

Sampled: 07/13/21 04:00
Received: 07/15/21 12:14
Analyzed: 07/20/21 08:52

Air Toxics by EPA Compendium Method TO-11A

Analyte	Results ug/m³	Flag	MDL ug/m³
Formaldehyde	3.01	DNPH	0.148
Acetaldehyde	1.94	DNPH	0.0703
Acetone	0.527	DNPH	0.510
Propionaldehyde	0.567	DNPH	0.0255
Crotonaldehyde	0.0336	DNPH	0.00629
Butyraldehyde	0.327	DNPH	0.0255
2-Butanone	0.114	DNPH	0.0409
Benzaldehyde	0.334	DNPH	0.0782
Valeraldehyde	0.607	DNPH	0.0448
Hexaldehyde	1.00	DNPH	0.207

- Review and confirm information
- Verify flagging information
- Note: In most cases, data flags are only applied based on laboratory result. Knowledge about applicable field flags may not be available to the lab.

TO-15 VOC Data

NATTS TAD Requirements with Qualifier Implications

Sampling

- Sampler Certification
  - Zero Air & Known Standard
  - Flag data for failing compounds for each sampling event
- Sampler Flow Verification

Laboratory

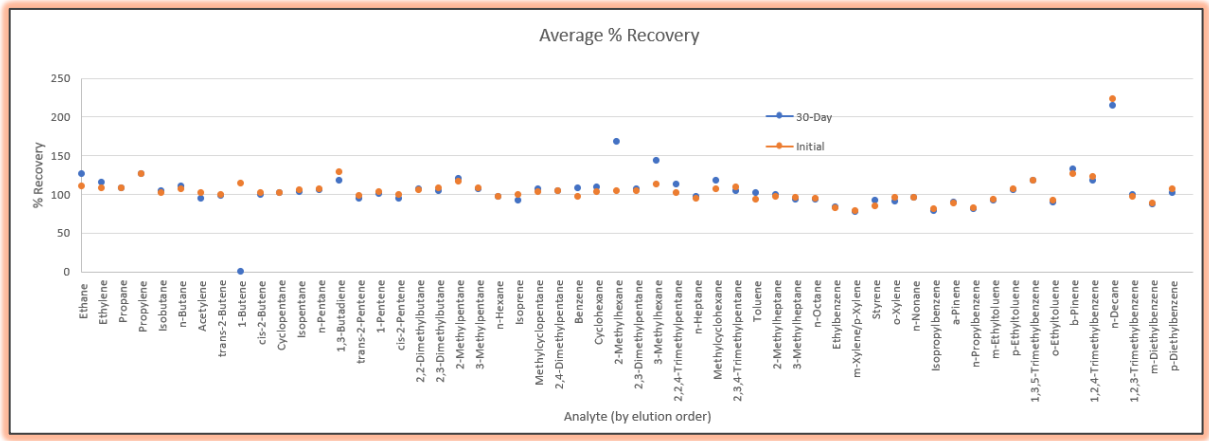
- Canister Qualification
  - Leak Check, Zero Air & Known Standard
  - Associated data flagging per canister
- Canister Hygiene
  - Cleaning batch verification
  - Sample collection 30 days from cleaning
  - Sample analysis 30 days from collection

Canister Tracking

Recommendation: Establish a canister tracking system to easily query canisters which may require qualification on a per analyte basis

- Failing Zero-Air Challenge? Qualify with…
  - CF to indicate canister failed qualification
  - And LK to indicate high bias
  - Or EC if exceeds 5x MDL – Null code to indicate exceedance of critical parameter
- Failing Known Standard Challenge? Qualify with…
  - CF to indicate canister failed qualification
  - And LL or LK to indicate low or high bias

		ethylene	acetylene	ethane	propylene	propane	isobutane	1-butene	1,3-butadiene	n-butane	trans-2- butene	cis-2-butene	ethanol	isopentane	1-pentene	n-pentane	isoprene	trans-2-pentene	cis-2-pentene	2,2-dimethylbutane
R6979	30	28%	44%	26%	105%	130%	1080%	101%	98%	115%	102%	105%	574%	187%	103%	99%	94%	96%	101%	104%
R7002	30	16%	29%	17%	125%	174%	1419%	128%	127%	146%	129%	130%	302%	233%	140%	131%	121%	124%	130%	139%
R6996	30	33%	24%	29%	95%	146%	517%	101%	114%	121%	101%	116%	447%	142%	107%	109%	94%	100%	104%	108%
R6987	30	12%	20%	14%	114%	145%	755%	135%	119%	129%	135%	124%	693%	176%	110%	119%	95%	113%	110%	118%
R6972	30	0%	24%	21%	132%	164%	335%	138%	131%	151%	138%	131%	722%	205%	132%	139%	119%	135%	134%	137%
R6966	30	84%	49%	59%	100%	133%	417%	96%	103%	113%	96%	106%	491%	160%	104%	104%	101%	107%	101%	105%
R6965	30	0%	26%	25%	106%	188%	457%	120%	126%	129%	120%	119%	682%	159%	108%	125%	117%	120%	116%	126%
R6980	30	15%	12%	0%	137%	182%	1113%	142%	145%	151%	142%	145%	1171%	470%	109%	143%	119%	126%	137%	147%
R6986	30	11%	48%	35%	139%	196%	1510%	143%	143%	153%	144%	152%	810%	254%	138%	144%	131%	136%	139%	155%
R6997	30	0%	18%	16%	94%	81%	163%	102%	97%	97%	102%	108%	157%	115%	110%	99%	102%	98%	110%	146%
R6955	0	35%	41%	32%	93%	93%	128%	140%	106%	104%	141%	110%	225%	141%	119%	107%	120%	101%	108%	126%
R6956	0	0%	29%	28%	113%	93%	113%	109%	102%	114%	109%	114%	97%	113%	124%	111%	111%	108%	114%	119%



PM10 Metals

Missing or Incomplete QC Data

- Filter Lot Blanks
  - Lot blank subtraction is not permitted
- Gravimetric Analysis
  - Are filter handling procedures documented?
  - Gravimetric lab meet NATTS Quality Systems requirements?
  - Are filters analyzed for metals <180 days?

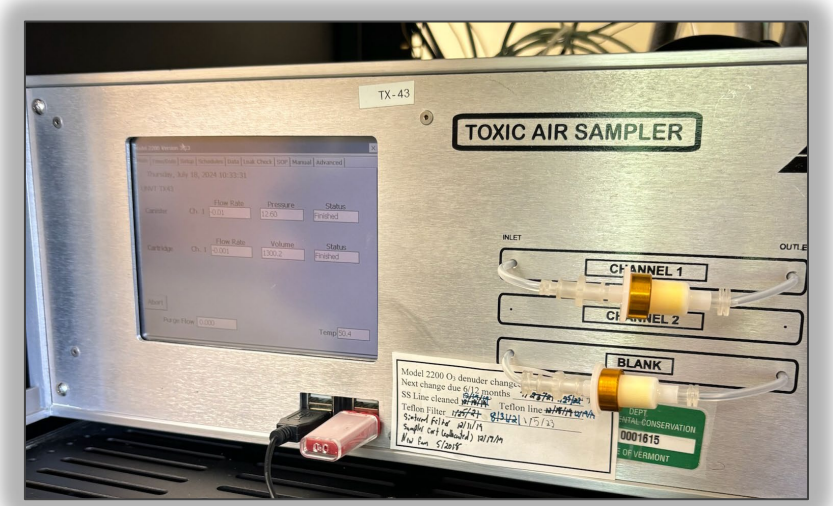
Carbonyl Data

Sampling

- Sampler Certification
  - Zero Air
  - Data flagging for each sampling event
- Sampler maintenance
  - Denuder change/recharge; filter change
- Data download
- Correct collection of a field blank

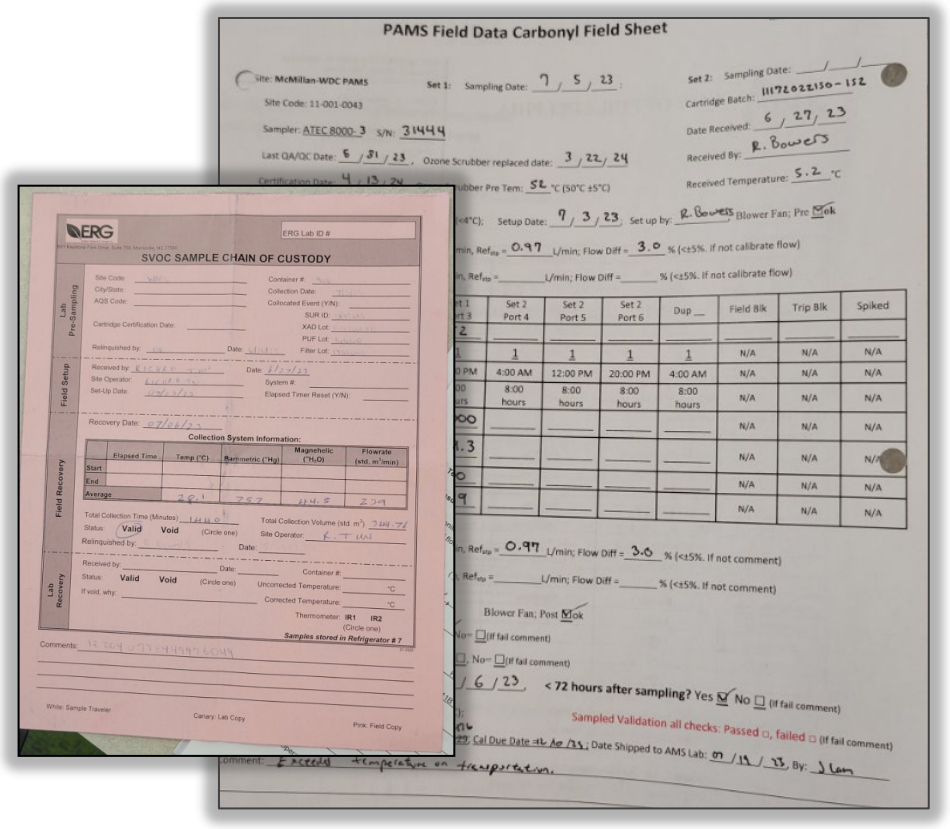
Laboratory

- Perform lot blank analysis
- Hold time exceedance
- DNPH chromatography evaluation
  - DNPH peak is present



Documentation

Records are incomplete, missing, or not reviewed



"If it hasn't been documented, did it even happen?"

- Update forms to include a sign-off for secondary review
- Store hard copy forms with redundancy so they can be easily accessed later

Acknowledgements

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Questions and Open Discussion

BATTELLE

It can be done

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