CSN & IMPROVE Mega PE Program Overview & Update

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Overview

- Program Background
- What PE's are and why they are useful
- Participating CSN & IMPROVE Laboratories
- $PM_{2.5}$ Round Robin
- Referee laboratories and how qualified
- PE Characterization Plan
- Mega PE Sampling Matrix
- Updated data analysis methods
- TSA Overview
- Status

Mega PE Program Overview & History

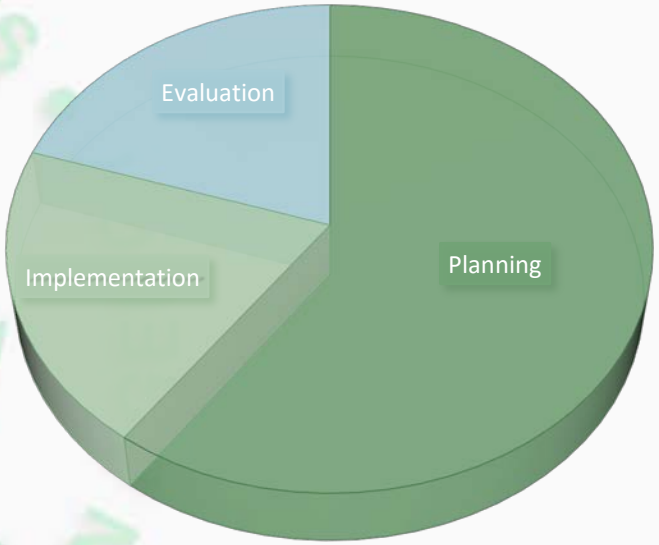
- **OAQPS Responsible for CSN & IMPROVE QA Oversight**
  - QAPPs, SOPs, Data Review, Network DQO's
- **ORIA QA Support for OAQPS (2000-2015)**
  - Mega PE, Laboratory TSA's, Special Studies
- **ORIA Functions Transition to OAQPS 2015**
  - Equipment and instrumentation transferred to OAQPS
  - Rebuilding program

What is the Mega PE Program?

- **Purpose**
  - Evaluate total network laboratory performance across the CSN and IMPROVE Networks
  - Quantitative and qualitative evaluation tools
  - Special studies to identify sources of error
- **Annual PE Audit Samples**
- **Biennial Technical and Quality Systems Audits**
  - Network Laboratories
  - Referee Laboratories
- **Includes Biannual Round Robin ($PM_{2.5}$ mass)**

Transition to OAQPS

- **Planning**
  - Development of PE sampling design, generation of appropriate data quality indicators, selection and challenge of referee laboratories, establish analytical measurement uncertainty, statistical analysis methods, development of standard operating procedures, refurbish and calibrate sampling equipment
- **Implementation**
  - Hope to collect first samples for characterization by late 2016
- **Evaluation**
  - Use of review, audit and statistical assessment tools to assess laboratory performance
    - Comparison of laboratory results to assigned reference values using z-scores



Performance Evaluation (PE)

- A type of audit in which the quantitative data generated in a measurement system are obtained independently and compared with a standard reference to evaluate the proficiency of an analyst or a laboratory.
  - Samples submitted in support of EPA programs where the analyte concentration is known to OAQPS, but not to the analyst or Network lab

Performance Evaluation (PE)

- Allows interlaboratory data comparisons.
  - Can identify individual laboratory bias relative to other laboratories
- Provides a level of confidence that laboratories are operating within an acceptable level of data quality so data users can make decisions with acceptable levels of certainty.
- Helps verify the precision and bias estimates performed by monitoring organizations.
- Identifies where improvements (technology/training) are needed.
- Assures the public of non-biased assessments of data quality.
- Provides a quantitative mechanism to defend the quality of data.
- Provides information to network laboratories on how they compare with other network labs, in relation to the acceptance limits, and to assist in corrective actions and/or data improvements.

Types of PE Samples for Mega PE

- $PM_{2.5}$ Mass Analysis on 47 mm and 25 mm Teflon® filter samples
- Ion Chromatography (IC) Analysis on 47 mm Nylon® filter samples
- Carbon by Thermal-Optical Reflectance (TOR) Analysis on 47 mm quartz filter samples
- Elemental analysis by X-Ray Fluorescence (XRF) on 47 mm and 25 mm Teflon® filter samples

Round Robin ($PM_{2.5}$ Mass)

- OAQPS distributes clean Teflon filters to each Network laboratory for tare weights
  - Filters returned to OAQPS for tare weights
- OAQPS collects $PM_{2.5}$ onto tared filters
  - Final weights obtained by OAQPS
  - Precision between collocated samples determined
- Samples shipped cool by express mail to each participating lab for final weights
  - $PM_{2.5}$ mass results compared with OAQPS weights
  - Interlaboratory results also compared

$$Final\ (\mu g) - Tare\ (\mu g) = PM_{2.5}\ Mass\ (\mu g)$$

Mega PE Participating Laboratories

Analytical Laboratories	Network	Analysis and Filter Types				
		OC/EC	Cations & Anions by IC	Elements by XRF	$PM_{2.5}$ Mass	
		Quartz (47 mm)	Nylon (47 mm)	PTFE (47 mm)	PTFE (25 mm)	PTFE (47 mm)
AMEC	CSN					✓
DRI	CSN & IMPROVE	✓	✓	✓		✓
UCD	CSN & IMPROVE			✓	✓	
CARB*	CSN	✓	✓	✓		✓
SCAQMD*	CSN	✓	✓	✓		✓
ODEQ*	CSN		✓	✓		
RTI	IMPROVE		✓			

* Independent CSN Laboratory

$PM_{2.5}$ Network Laboratories

Akron Regional Air Pollution Control Agency
Al Dept Of Env Mgt
Alaska Department Of Environmental Conservation
Albuquerque Environmental Health Department, AQD
Allegheny County,PA Health Department
Antelope Valley APCD
Appleton Health Department
Arizona Department Of Environmental Qu
Arkansas Department Of Environmental Quality
Bay Area Air Quality Management District
Broward County Environmental Protection Department
California Air Resources Board
Chattanooga Hamilton County Air Pollutio Control
City of Huntsville, Div of Natural Resources
Clark County, NV DAQEM
Cleveland Air Pollution Control Agency
Colorado Department of Public Health And Environment
Connecticut Department of Environmental Protection
Cook County Department of Environmental Control
Dayton Regional Air Pollution Control Agency
Delaware Dept Natural Resources and Environmental Control
Department of Energy & Environment, District of Columbia
Desert Research Institute
Environmental Resources Management (ERM)
Eri/University Of Connecticut
Fairbanks North Star Borough Environmental Services
Florida Dep Of Environmental Protection Lab, Tallahassee
Georgia Air Protection Branch Ambient Monitoring Program
Great Basin Unified APCD
Hamilton County Department Of Environmental Services
Lane Regional Air Pollution Authority
Louisville, KY Metro Air Pollution Control District
Maine D.E.P. Bureau Of Air Quality Control, Augusta
Mass Dept Environmental Protection Div Air Quality Control
Memphis Shelby County Health Department
Metropolitan Health Department/Nashville & Davidson County
Michigan Dept Of Environmental Quality Air Quality Division
Minnesota Pollution Control Agency, Division Of Air Quality
Mississippi DEQ, Office Of Pollution
Mojave Desert AQMD
Nebraska Department Of Environmental Control
New Hampshire Air Resources Agency
New Jersey State Department Of Environmental Protection
New Mexico Environment Department
New York State Department Of Environmental Conservation
Philadelphia Air Management Services
Pima County Department of Environmental Quality
Pinal County APCD
Pinellas County Department Of Environmental Management
Polk County Physical Planning
Portsmouth City Health Dept Division Air Pollution Control
Puerto Rico Environmental Quality Board
Puget Sound Air Pollution Control Agency
Research Triangle Institute RTP, NC
Rhode Island DEM And DOH
San Diego County Air Pollution Control District
South Carolina Department Health And Environmental Control
South Coast Air Quality Management District
South Dakota Dept Environmental Protection Air Quality Prog
Southern Ute Indian Tribe of Southern Ute Reservation, CO
Southwestern Ohio Air Pollution Control Agency
State Of Louisiana
State Of Maryland Air Management Administration
Tennessee Division Of Air Pollution Control
Texas Commission On Environmental Quality
University Hygenic Laboratory
University Of Medicine And Dentistry Of New Jersey
Utah Department Of Environmental Quality
Virgin Islands Department Of Planning & Natural Resources
Virginia Department of Environmental Quality
Virginia Division Of Consolidated Laboratories
WI State Lab Of Hygiene Environmental Science Section
WI State Lab Of Hygiene Occupational Health Laboratory
Washington State Department Of Ecology
Washoe County District Health Department
Wayne County Air Pollution Control Division
West Virginia Air Pollution Control Commission
West Virginia Northern Panhandle Regional Office

PM2.5 Labs Routinely Audited (Biannually)

AMEC Foster Wheeler (Newberry, FL)

EPA Region 4 (Athens, GA)

EPA Region 2 (New York, NY)

EPA OAQPS (RTP, NC)

Inter-Mountain Laboratories (Gillette, WY)

Mega PE Referee Laboratories

EPA Laboratory	Analysis Type	Anticipated Sample Volume					
		Characterization Study	Annual Mega PE				
		2016		2017	2018	2019	2020
ORD NERL	IC Cations & Anions	32	32	32	32	32	32
	$PM_{2.5}$ Mass	32	64	64	64	64	64
ORD NRMRL	OC/EC	16	16	16	16	16	16
	ED-XRF Elements	16	16	16	16	16	16
Totals		96	128	128	128	128	128

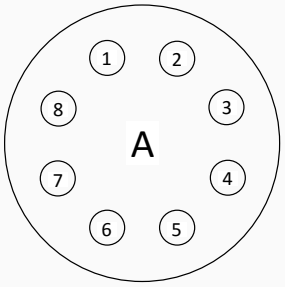
Referee Laboratory Qualification

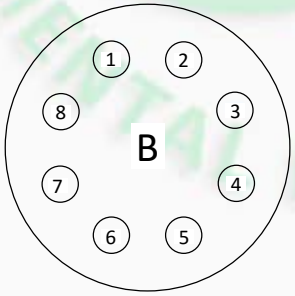
All referee laboratories will be qualified with NIST standard reference materials (SRMs) or NIST traceable standards:

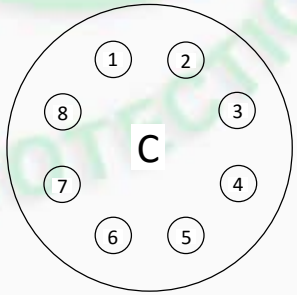
- NIST SRM filters loaded with ambient $PM_{2.5}$ with certified and reference values for selected elements will be used to qualify the XRF (Cooper).
- NIST SRM filters loaded with urban $PM_{2.5}$ dust with reference values for total carbon (TC) by the IMPROVE method will be used to qualify the EPA NRMRL laboratory.
- NIST SRMs for anions and cations are not available, so custom NIST traceable analytical standards will be used to qualify the NERL IC lab.
- NIST traceable gravimetric PE filter samples are not available, therefore the microbalance used for gravimetric analysis will be calibrated with NIST traceable standards, and the calibration will be checked by OEM calibration check weights.

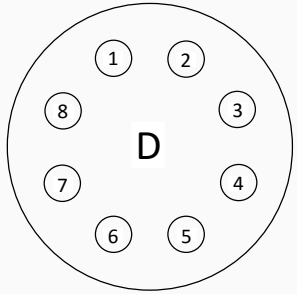
PE Sampling Method Characterization

- $PM_{2.5}$ mass initially
  - Evaluate precision between 4 collocated samplers (< 10% CV)
- Speciation after samplers check out
  - Submit for referee analyses
  - Confirm precision in limits
  - Estimate concentrations as *f(t)*
- Estimate sampling and analytical uncertainties
  - Will vary by sampling event
  - *f(t)*: less sample, higher uncertainties
- Generate and Characterize PE Samples

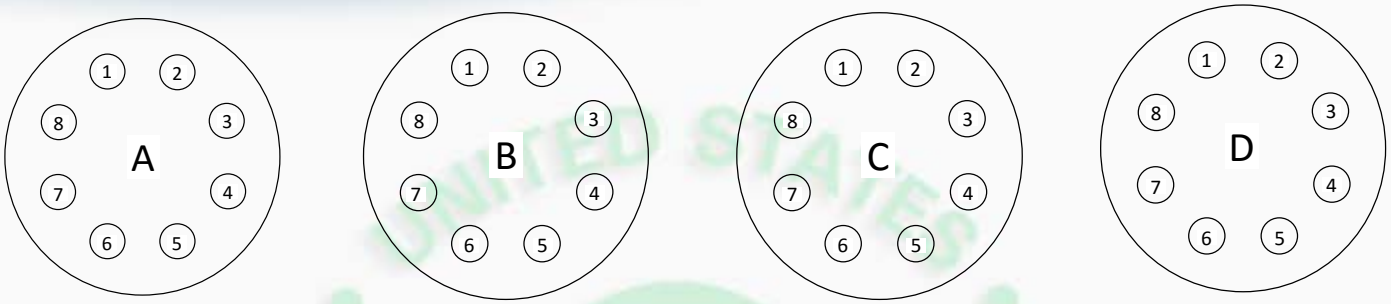






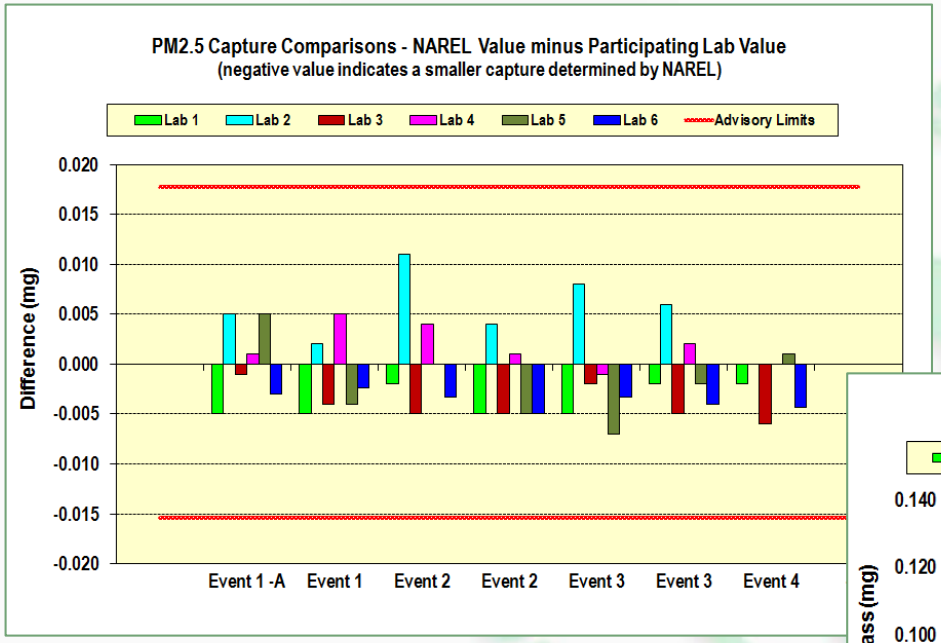


PE Sampling Matrix

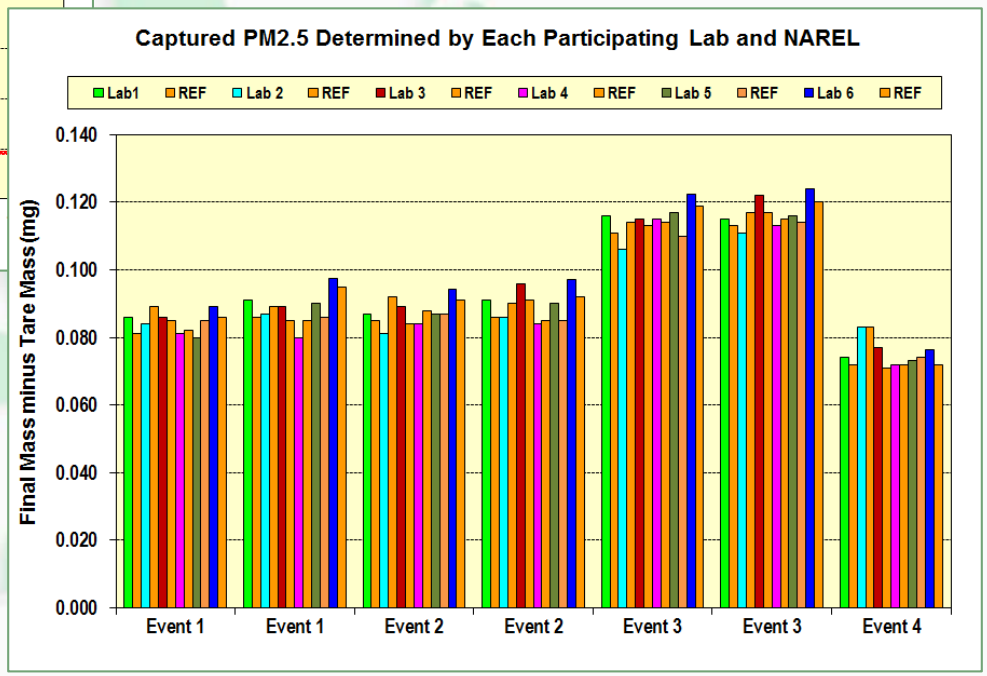


Sampling Event[a]	Filter Type	# Loaded PE's	Disposition						Analyses Type			
			Labs		REF[b]		Spares		OC/ EC	IC	XRF	Grav
			PE	Blk	PE	Blk	PE	Blk				
1	47 mm Quartz	32	12	12	8	8	12	4	✓			
2	47 mm Teflon	32	4	4	8	8	4	4		✓		
	25 mm Teflon		4	4	4	4	8	4				✓
3	47 mm Nylon	32	20	20	8	8	4	4		✓		
4	47 mm Teflon	32	20	20	8	8	4	4			✓	
5	47 mm Teflon	32	16	16	8	8	8	4				✓
Totals			76	76	44	44	40	24				

Mega PE Output - Historical



- Interlaboratory Gravimetric Data from Mega PE #9 (2014)



- Historical advisory limits derived from historical interlaboratory standard deviations of PE measurements
  - Not reference values
  - LL = *X*-0.015 UL = *X*+0.018

Mega PE Output - Updated

- Reference values derived from analysis of multiple filter samples from each sampling event
- Z-scores used to evaluate participant laboratory results against reference values:

$$|z| = \left| \frac{x_{lab} - \bar{X}_{ref}}{\sigma_{ref}} \right|$$

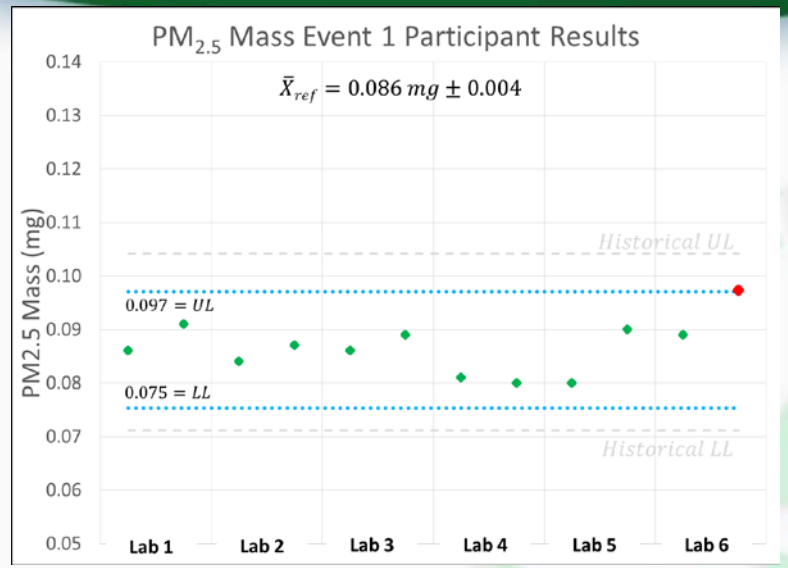
Sampling Event	Sampling Duration (hr)	Reference Average mg	σ	n	Advisory Limits	
					LL	UL
1	42	0.086	0.004	12	0.075	0.097
2	36	0.088	0.003	12	0.079	0.097
3	24	0.115	0.003	12	0.106	0.124
4	20	0.074	0.005	12	0.060	0.088

Lab	Event 1		Event 2		Event 3		Event 4	
	|z|	(mg)	|z|	(mg)	|z|	(mg)	|z|	(mg)
1	0.046	0.086	0.328	0.087	0.414	0.116	0.000	0.074
	1.337	0.091	0.985	0.091	0.083	0.115		
2	0.600	0.084	2.299	0.081	2.898	0.106	1.993	0.083
	0.231	0.087	0.657	0.086	1.242	0.111		
3	0.046	0.086	0.328	0.089	0.083	0.115	0.664	0.077
	0.784	0.089	2.627	0.096	2.402	0.122		
4	1.430	0.081	1.314	0.084	0.083	0.115	0.443	0.072
	1.706	0.080	1.314	0.084	0.580	0.113		
5	1.706	0.080	0.328	0.087	0.745	0.117	0.221	0.073
	1.061	0.090	0.657	0.090	0.414	0.116		
6	0.784	0.089	2.080	0.094	2.512	0.122	0.517	0.076
	3.090	0.097	2.956	0.097	3.064	0.124		

|z| ≤ 2	Satisfactory
2 < |z| 3	Questionable
|z| ≥ 3	Unsatisfactory

z-score based on ISO 13528:2015 standard, *Statistical methods for use in proficiency testing by interlaboratory comparisons*.

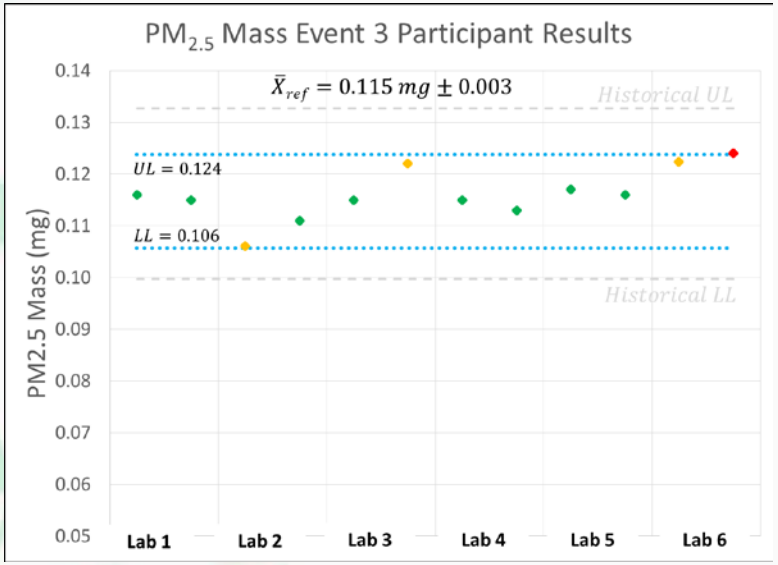
Mega PE Output - Updated

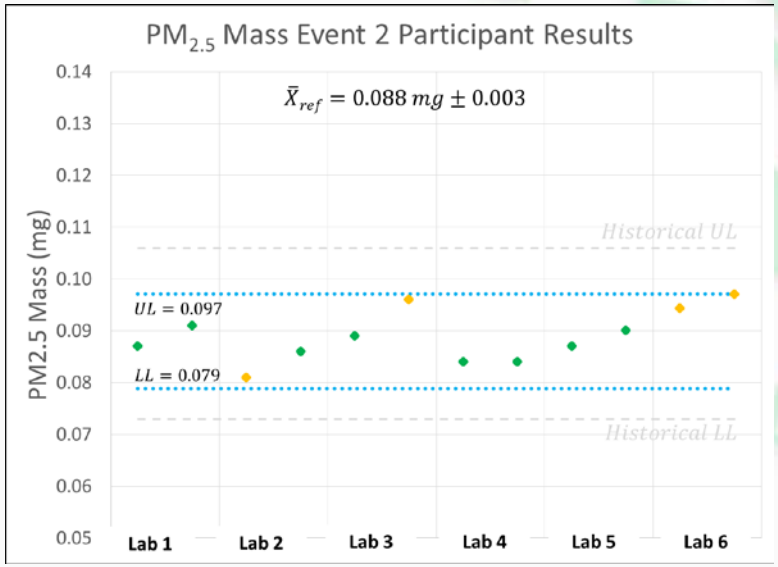


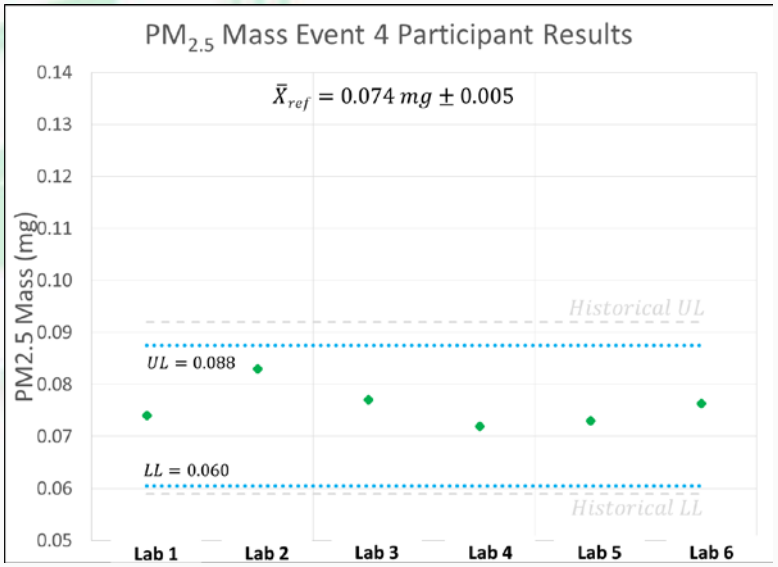
Historical Advisory Limits
LL: $-0.015\ mg$
UL: +0.018 mg

Updated Advisory Limits
$\bar{X}_{ref} \pm 3\sigma$

$\lvert z \rvert \leq 2$	Satisfactory
$2 < \lvert z \rvert\ 3$	Questionable
$\lvert z \rvert \geq 3$	Unsatisfactory

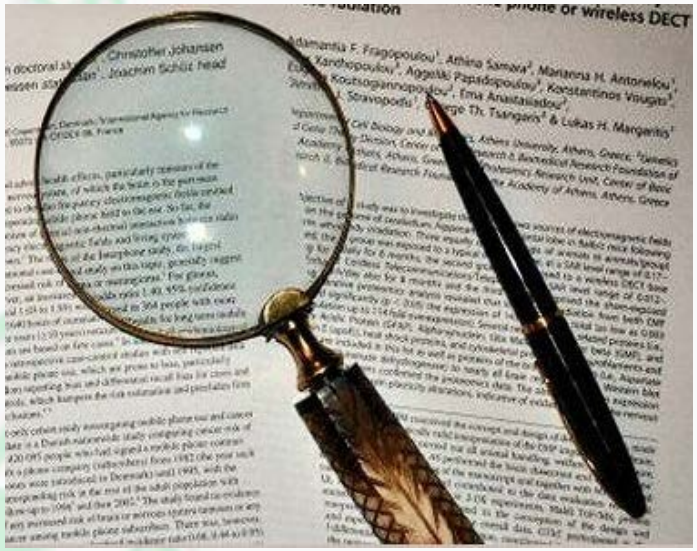




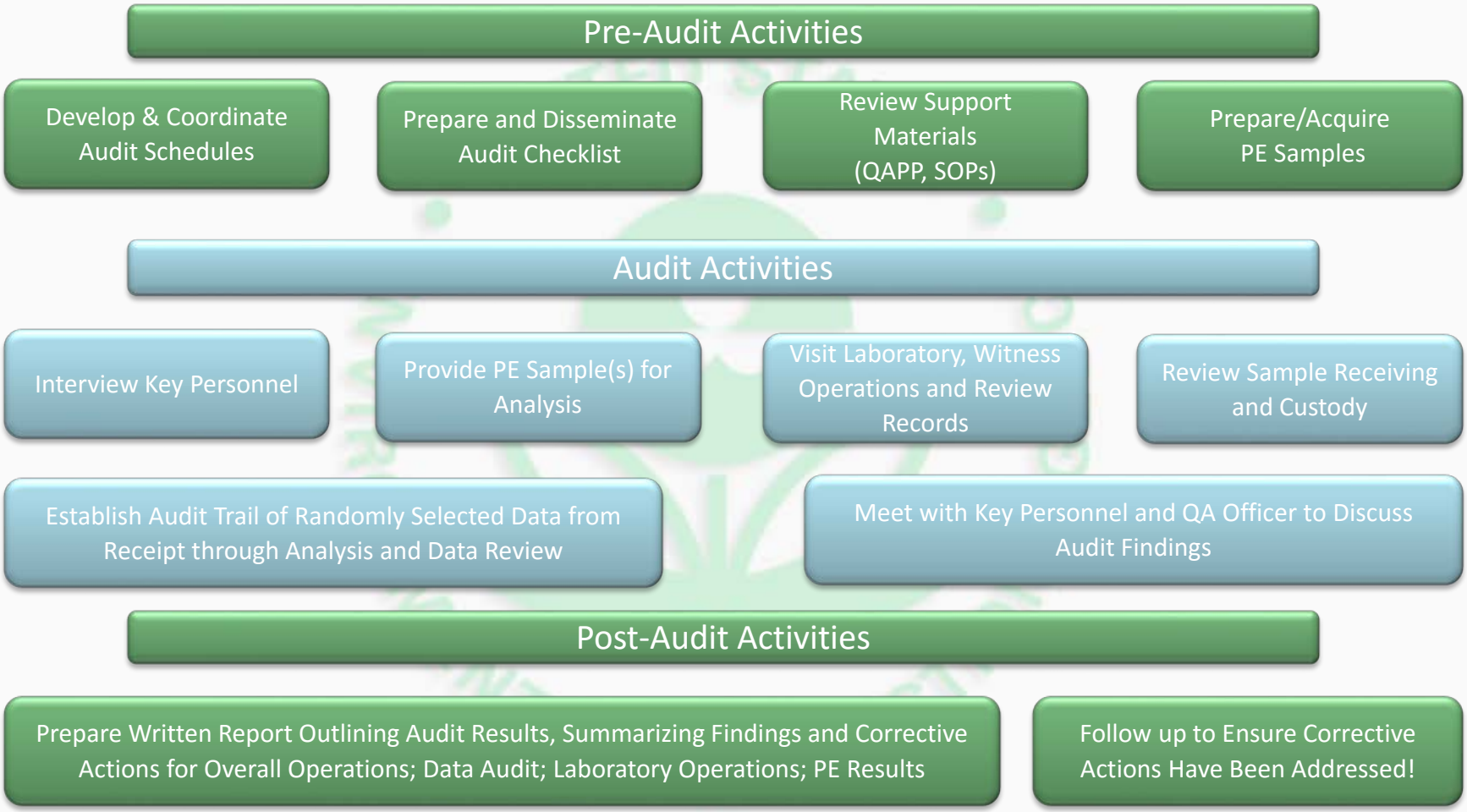


Technical Systems Audit (TSA)

- What is it?
  - Thorough, systematic, on-site, qualitative audit of facilities, equipment, personnel, training, procedures, record keeping, data validation, chain of custody, data management, and reporting aspects of a system
- Includes Reviews of..
  - QAPP and SOPs
    - To ensure both are followed or deviations documented
  - Training Records; Chain-of-Custody; Documentation (calibrations..)
  - May Include a PE Sample for analysis
- Checklist provided prior to audit so lab is prepared
  - Not meant to be a surprise



Technical Systems Audit (TSA)



Network Lab Locations

Year	TSA Conducted (Plan for 2016/2017)
2000	RTI
2002	RTI
2003	RTI
2005	RTI
2006	CARB, DRI, ODEQ, RTI, UCD
2007	DRI, UCD
2008	CARB, ODEQ
2009	RTI, SCAQMD
2010	DRI, UCD
2011	CARB, ODEQ
2012	RTI, SCAQMD
2013	DRI, UCD
2015	CARB, ODEQ
2016	RTI
2017	AMEC, CARB, DRI, ODEQ, SCAQMD, UCD

To Do

- Measurement uncertainty (sampling + analysis)
- ORD Laboratory setup, calibration
- Referee laboratory qualifications
- Revise CSN & IMPROVE QAPPs
- Program SOPs
  - Sample collection, analytical methods, data analysis and interpretation, Report output, LIMS, & TSAs.
- Biggest hang-up currently is sampler status

QUESTIONS?

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nference August 201