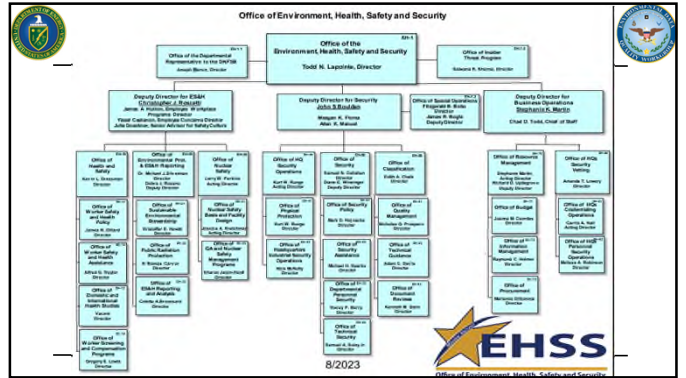


**DEPARTMENT OF ENERGY
ANALYTICAL SERVICES PROGRAM
CONSOLIDATED AUDIT PROGRAM
ACCREDITATION PROGRAM**

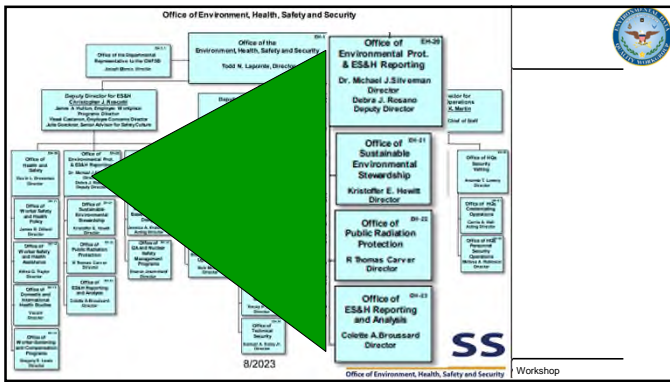
Debbie Rosano
Deputy Director
Office of Environmental Protection and ES&H Reporting (EHSS-20)
U.S. Department of Energy
Office of Environment, Health, Safety and Security (EHSS)

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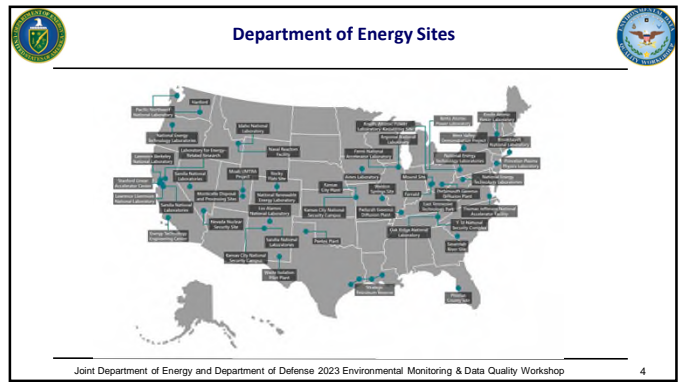
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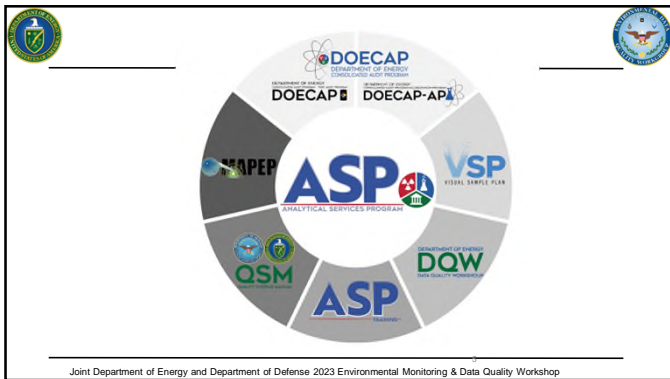
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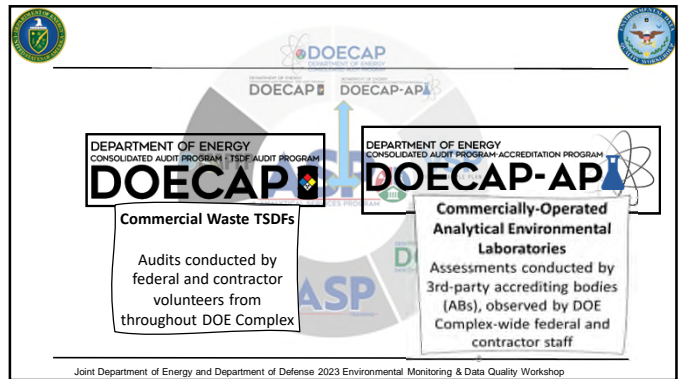
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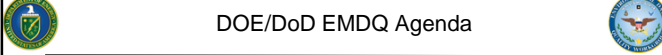
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Welcome to the 2023 Joint DoE/DoD Environmental Monitoring and Data Quality Workshop
 September 2023
 Jordan Adelson, Ph.D.
 Director, Navy Laboratory Quality & Accreditation Office
 Chair, DoD Environmental Data Quality Workgroup

Joint Department of Energy and Department of Defense 2023 Environmental Monitoring & Data Quality Workshop

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DOE/DoD EMDQ Agenda

Tuesday, September 19, 2023
Introduction and Welcome
DOECAP and DoD ELAP Updates
PFAS
QSM 6.0
Wednesday, September 20, 2023
QSM 6.0 (cont.)
DoE/DoD Open Forum
Thursday, September 21, 2023
Incremental Sampling Methodology
EPA's Guidelines on Validation of Non-Regulatory Methods
Data Review and Management Update
Improving Data and Decisions
Final Comments

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2

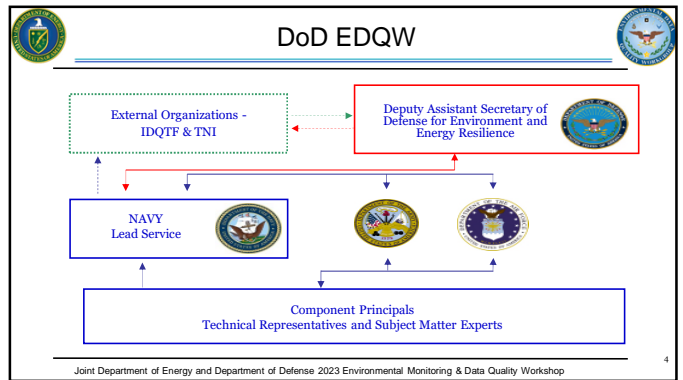


Environmental Data Quality Workgroup

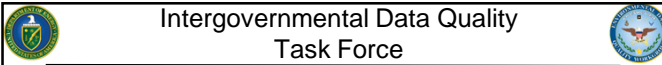
- Develop and recommend policy related to sampling, testing, and quality assurance for environmental programs to eliminate redundancy, streamline programs, improve data quality, and promote data integrity.
- Coordinate the exchange of information among DoD components.
- Develop DoD issuances to implement environmental quality systems and promote cost effective government oversight.
- Implement and provide oversight of the DoD ELAP.

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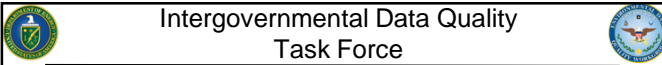


Intergovernmental Data Quality Task Force

- IDQTF Executive Committee
 - EPA
 - OEI/Quality Staff Director
 - Lead Region QAM for OEI
 - Lead Region QAM for OSWER
 - DoD
 - EDQW Principals
- Work collaboratively to:
 - Address environmental issues of emerging concern at federal facilities
 - Promote implementation of consistent and transparent intergovernmental quality systems
 - Ensure a scientific basis for environmental decision-making

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Intergovernmental Data Quality Task Force

- Working on PFAS Treatment Technology Review with SERDP/ESTCP
- Data Usability Guidance
- Munitions Response

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MR-QAPP Toolkit Overview

- Planning tool for characterization and remediation of buried munitions and explosives of concern (MEC) at MRS
- Module 1: RI/FS; Module 2: RA
- Based on Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP, IDQTF, 2005)
- Implements a systematic planning process (SPP)

UNIFORM FEDERAL POLICY FOR QUALITY ASSURANCE PROJECT PLANS
Munitions Response QAPP Toolkit
Version 1.0
Revised December 2008

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DoD EM/DQ Future Plans

- CY 2024 Webinar in Spring/Summer
- Planning Face-to-Face for Spring CY 2025
 - Location?
 - Topics?
- Please provide suggestions in the survey

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Improving Environmental Data Quality


... Because the Right Decisions Require Quality Data

Jordan.M.Adelson.civ@us.navy.mil (EDQW Chair)
www.denix.osd.mil/edqw/

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Office of the Secretary of Defense Welcome and Goals (Public Participation Encouraged)


Brian Jordan
Office of the Deputy Assistant Secretary of Defense
(Environment and Energy Resilience)

September 19, 2023

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1

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OSD Organization Chart

Assistant Secretary of Defense (Energy, Installations and Environment)
ASD (EI&E)

↓

Office of the Deputy Assistant Secretary of Defense
(Environment and Energy Resilience)
ODASD (E&ER)

↓


Air Force, Army and Navy
(Cleanup Programs)

ODASD (E&ER): Provides policy, guidance and resource management to the military services on cleanup programs.

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Relevant Policies and Guidance

1. Munitions Response Guidance:
 - Munitions Response Quality Assurance Project Plan Module 1 and 2
 - Risk Management Methodology (RMM)
 - DAGCAP (ELAP for the Munitions World)
 - www.denix.osd.mil/mmrp
2. PFAS Policies:
 - Establishing a Consistent Methodology for the Analysis of Per- and Polyfluoroalkyl Substances in Matrices Other than Drinking Water (7 August 2023)
 - Memorandum for Taking Interim Actions to Address Per- and Polyfluoroalkyl Substances Migration from DoD Installations and National Guard Facilities (11 July 2023)
 - www.acq.osd.mil/eie/eer/ecc/pfas/tf/policies.html

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Discussions and Goals


We need to take advantage of the conversations and ideas we can share this week.

- Policies and Guidance.
 - What did we get wrong? How can we improve?
- Resources
 - Money – Budget bills still in Congress.
 - People
- Capacity – This is a real problem.
- Services vs. Commodities

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Shameless Plug

Improving Data and Decisions (Thursday)

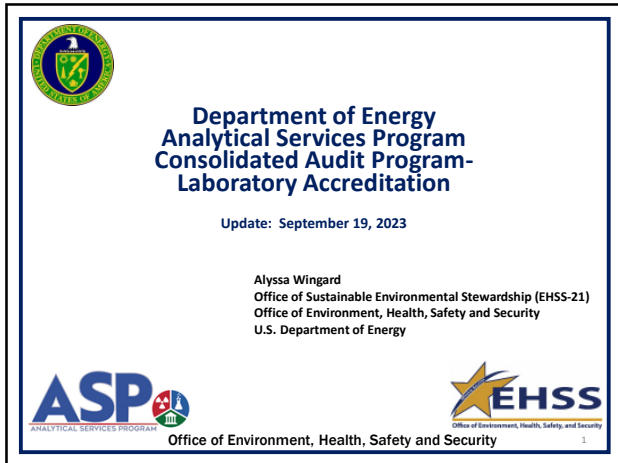
- Additional discussions and looking for your good ideas
- Interested in how we combine our resources to reach better environmental decisions

Contact Info: Brian Jordan, 703-409-8657
brian.d.jordan6.civ@mail.mil

Questions?

UNCLASSIFIED

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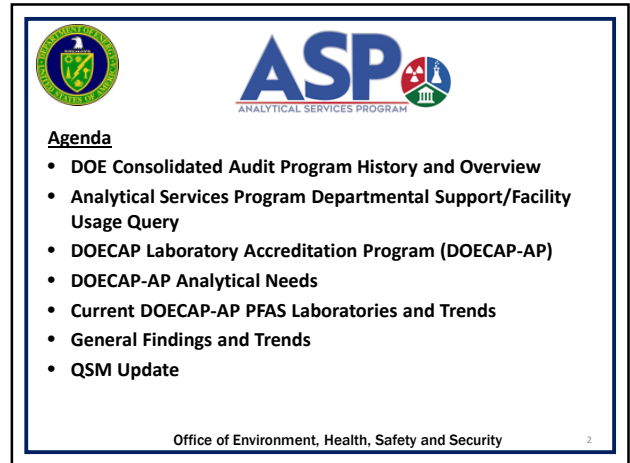
Department of Energy Analytical Services Program Consolidated Audit Program-Laboratory Accreditation

Update: September 19, 2023

Alyssa Wingard
Office of Sustainable Environmental Stewardship (EHSS-21)
Office of Environment, Health, Safety and Security
U.S. Department of Energy

ASP ANALYTICAL SERVICES PROGRAM **EHSS** Office of Environment, Health, Safety and Security

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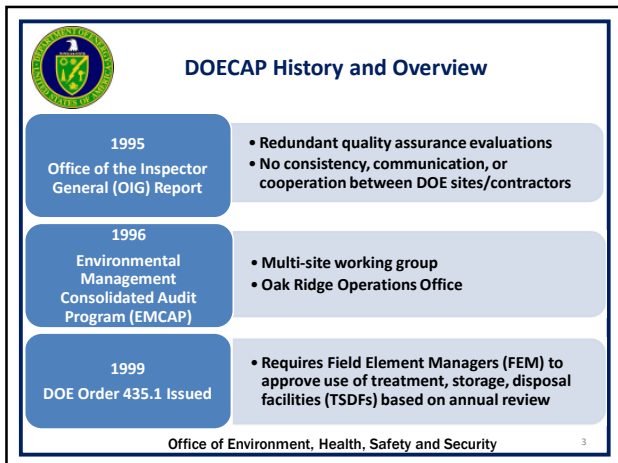
ASP ANALYTICAL SERVICES PROGRAM

Agenda

- DOE Consolidated Audit Program History and Overview
- Analytical Services Program Departmental Support/Facility Usage Query
- DOECAP Laboratory Accreditation Program (DOECAP-AP)
- DOECAP-AP Analytical Needs
- Current DOECAP-AP PFAS Laboratories and Trends
- General Findings and Trends
- QSM Update

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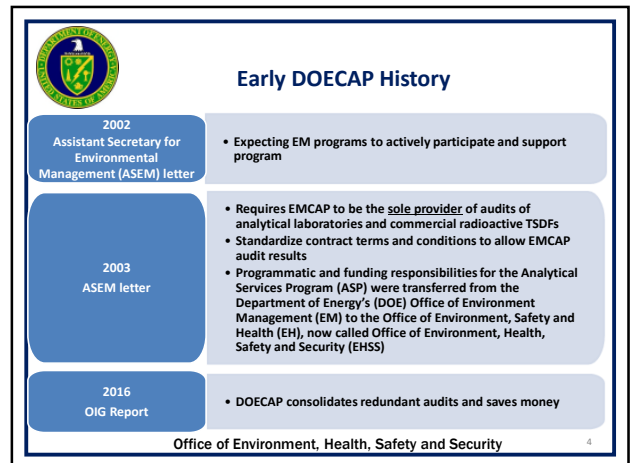


DOECAP History and Overview

- 1995**
Office of the Inspector General (OIG) Report
 - Redundant quality assurance evaluations
 - No consistency, communication, or cooperation between DOE sites/contractors
- 1996**
Environmental Management Consolidated Audit Program (EMCAP)
 - Multi-site working group
 - Oak Ridge Operations Office
- 1999**
DOE Order 435.1 Issued
 - Requires Field Element Managers (FEM) to approve use of treatment, storage, disposal facilities (TSDFs) based on annual review

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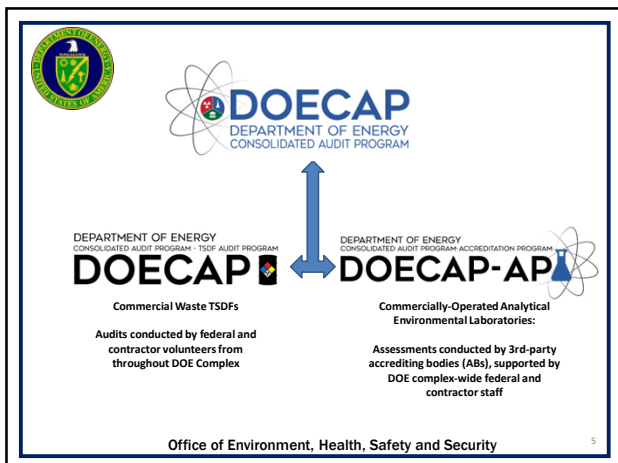


Early DOECAP History

- 2002**
Assistant Secretary for Environmental Management (ASEM) letter
 - Expecting EM programs to actively participate and support program
- 2003**
ASEM letter
 - Requires EMCAP to be the sole provider of audits of analytical laboratories and commercial radioactive TSDFs
 - Standardize contract terms and conditions to allow EMCAP audit results
 - Programmatic and funding responsibilities for the Analytical Services Program (ASP) were transferred from the Department of Energy's (DOE) Office of Environment Management (EM) to the Office of Environment, Safety and Health (EH), now called Office of Environment, Health, Safety and Security (EHSS)
- 2016**
OIG Report
 - DOECAP consolidates redundant audits and saves money

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DOECAP DEPARTMENT OF ENERGY CONSOLIDATED AUDIT PROGRAM

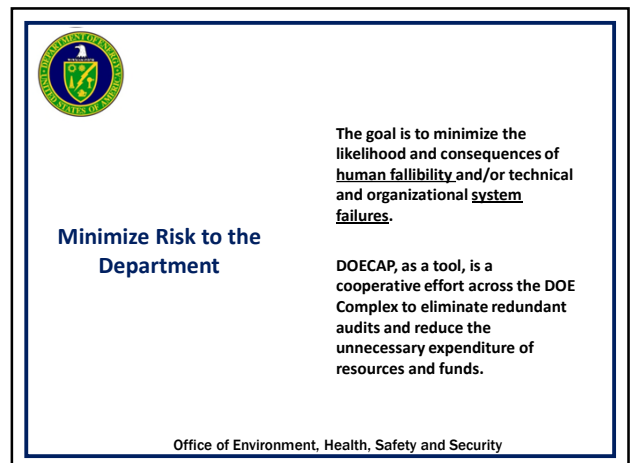
DOECAP-AP DEPARTMENT OF ENERGY CONSOLIDATED AUDIT PROGRAM ACCREDITATION PROGRAM

DOECAP
Commercial Waste TSDFs
Audits conducted by federal and contractor volunteers from throughout DOE Complex

DOECAP-AP
Commercially-Operated Analytical Environmental Laboratories:
Assessments conducted by 3rd-party accrediting bodies (ABs), supported by DOE complex-wide federal and contractor staff

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Minimize Risk to the Department

The goal is to minimize the likelihood and consequences of human fallibility and/or technical and organizational system failures.





DOECAP, as a tool, is a cooperative effort across the DOE Complex to eliminate redundant audits and reduce the unnecessary expenditure of resources and funds.

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ASP Support to the Department/Facility Usage Query - FY2023

-  **350 contracts** throughout the DOE Complex: (160 Laboratory/190 TSDF)
-  **\$102M** in contracts
 - \$44.5M - Laboratory
 - \$57.5M - TSDF
-  **33 DOE Sites & Contractors**
-  **7 Program Offices Supported:** SC, EERE, EM, NNSA, FECD, LM, NE


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DEPARTMENT OF ENERGY
CONSOLIDATED AUDIT PROGRAM-ACCREDITATION PROGRAM

DOECAP-AP



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Importance of the DOECAP Accreditation Program

- DOECAP-Accreditation Program (DOECAP-AP) provides DOE sites assurance that a contracted commercial laboratory is fully capable of providing accurate data analysis
- Requires third-party assessments which assure that environmental sample analysis is performed using proven methods, provide valid, reliable, and defensible data, and are managing sample waste streams responsibly
- Assessments are conducted by one of four DOECAP approved third-party accreditation bodies (ABs)
- Laboratories are assessed to the most recent version of the Department of Defense/Department of Energy (DoD/DOE) Quality Systems Manual (QSM)

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Third-Party Accrediting Bodies





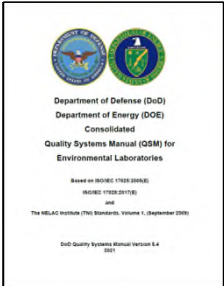

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DoD/DOE Quality Systems Manual (QSM) for Environmental Laboratories

- Provides baseline requirements for establishing and managing quality systems for laboratories performing analytical testing for DoD and DOE.
- Only approved Accrediting Bodies assess and accredit laboratories to the most recent version of the QSM.
- EHSS-21 works in conjunction with DoD to advance the interests of the Department in supporting the continued development and implementation of the QSM.
- The QSM supports DOE analytical needs with emerging contaminants, such as per- and polyfluoroalkyl substances (PFAS).




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of DOECAP-AP Accredited Laboratories



20 ACCREDITED LABORATORIES PERFORMED BY APPROVED 3RD-PARTY ACCREDITING BODIES




1 DOECAP AUDITED LABORATORY - ALS OHIO

*** 3 ADDITIONAL LABORATORIES PENDING FOR PFAS**

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Increasing DOE Analytical Needs

- The DOE is expecting a significant need for laboratory testing and analysis for PFAS. The two links below can help you see the magnitude of the testing at over 50 sites and how early in the process we are.
- DOE PFAS Strategic Roadmap – DOE commitments to action 2022-2025
 - <https://www.energy.gov/sites/default/files/2022-08/DOE%20PFAS%20Roadmap%20August%202022.pdf> [energy.gov]
- Initial Assessment of Per and Polyfluoroalkyl Substances at DOE sites
 - <https://www.energy.gov/sites/default/files/2022-11/DOE%20Initial%20PFAS%20Assessment%20-508.pdf> [energy.gov]

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Adding DOECAP Accreditation

- Very few “DOE only” requirements in QSM Version 6.0
- Laboratories do not need a Radioactive Materials License unless they are receiving radioactive samples
- The DOE sites and/or their Prime Contractors contract directly with the laboratories and often use a “Best Value Approach” in their laboratory selection

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PFAS Laboratory Status Last Updated: September 5, 2023

Audit Location	PFAS	EPA 533	EPA 537.1	EPA Draft Method 1633	Cert Expiration Date
ALS Laboratory Group - Salt Lake	No	No	No	No	1/31/2024
ARS Aleut Analytical, LLC	No	No	No	No	10/12/2024
Eberline Analytical Corporation, Oak Ridge	No	No	No	No	9/30/2024
EMSL Analytical, Inc.	Yes	Yes	Yes	No	7/31/2024
Eurofins Denver	Yes	No	No	Yes	10/31/2023
Eurofins Environment Testing Northern California	No	No	No	No	4/27/2024
Eurofins Knoxville	No	No	No	No	2/13/2025
Eurofins Sacramento	Yes	Yes	Yes	Yes	1/20/2024
Eurofins Seattle	No	No	No	No	1/19/2025
Eurofins St. Louis	No	No	No	No	4/06/2025
GEL Laboratories, LLC	Yes	Yes	Yes	Yes	6/30/2025
Materials and Chemistry Laboratory, Inc.	No	No	No	No	9/30/2024
Pace Analytical - South Carolina	Yes	No	Yes	Yes	11/18/2024
Pace Analytical National	No	No	No	No	11/30/2023
Pace Analytical Services, LLC dba BC Laboratories, Inc.	No	No	No	No	3/31/2024
RJ Lee Group-CBAL	No	No	No	No	7/31/2024
Southwest Research Institute	No	No	No	No	8/31/2024
Teledyne Brown Engineering, Inc.	No	No	No	No	1/31/2025
Torrent Laboratory, Inc.	Yes	No	No	Yes	12/31/2024
Weck Laboratories Inc.	Yes	No	Yes	Yes	9/22/2023

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PFAS Findings and Trends

- In 2021, there were 4 findings and zero observations for PFAS
- In 2022, there were 16 findings and 5 observations for PFAS
- So far in 2023, there were 14 findings and zero observations for PFAS

Every finding from laboratory assessments was reviewed individually for key words buried in the text such as: 537.1, 537, 533, 8327, 1633, B-24, 1621 TOF, OTM 45, B-15 or any individual PFAS target analytes

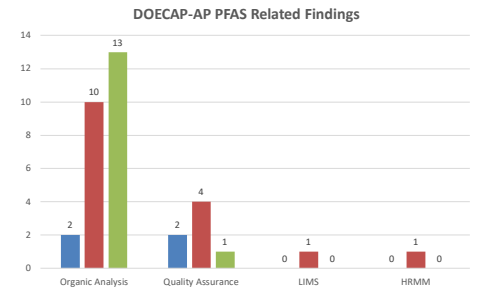
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PFAS Related Findings by Concentration


DOECAP-AP PFAS Related Findings



Concentration	2021	2022	2023
Organic Analysis	2	10	13
Quality Assurance	2	4	1
LIMS	0	1	0
HRMM	0	1	0

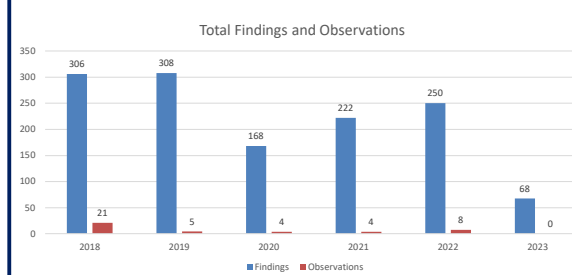
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Total Laboratory Findings and Observations (2018 to Present)

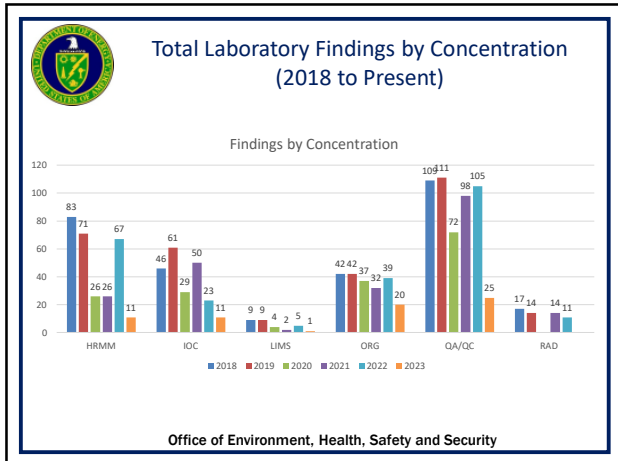
Total Findings and Observations



Year	Findings	Observations
2018	306	21
2019	308	5
2020	168	4
2021	222	4
2022	250	8
2023	68	0

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QSM 6.0 Implementation

- **Release Anticipated late calendar year 2023**
 - Accreditation can begin six months after release date
- **Implementation with routine accreditation cycle**
 - Implementation should occur during the routine accreditation cycle. Laboratories with accreditation expiration dates close to the implementation date should work with their AB on scheduling.
- **Accreditation mandatory 30 months after release**
 - Accreditation is required two years after implementation begins. Some ABs operate on a three-year cycle so some laboratories may require accreditation before their current certificate expires.

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Questions or Comments Always Welcome from ASP Stakeholders

ALYSSA WINGARD
Office of Environment, Health, Safety and Security
Office of Sustainable Environmental Stewardship (EHSS-21)
U.S. Department of Energy
Alyssa.Wingard@hq.doe.gov
240-961-0401 (cell)

Office of Environment, Health, Safety and Security

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DoD ELAP Update

William Corl Ph.D.
 William.e.corl.civ@us.navy.mil
 NAVSEA Laboratory Quality and Accreditation Office
 (LQAO)

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1






OBJECTIVES

- DoD ELAP Status
 - General Updates
 - PFAS Accreditations
 - Draft EPA Method 1633, 537.1, & 533
- DoD EDQW & ASD Memorandums
- DENIX

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DOD ELAP STATUS AUGUST 2023

AB	2023	2022	2021	2020	2019	2018	2017	2016	2015	2014	2013	2012	2011
A2LA	19	19	21	17	19	22	25	25	22	27	29	30	24
PJLA	30	32	32	32	34	31	26	27	29	28	31	27	23
ANAB*	25	22	28	30	29	32	39	41*	11	11	11	14	24
LAB*	*	*	*	*	*	*	*	*	*	31	32	28	31
IAS	0	**	**	**	**	**	**	**	**	**	**	**	**
Total Labs	74	73	81	79	82	85	90	93	93	98	99	102	99

*ANAB acquired LAB December 2015.
 **IAS rec'd DoD ELAP recognition late 2022.

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DRAFT EPA METHOD 1633 ACCREDITATIONS

Matrix	Number of Accredited Laboratories
AFFF	27
Aqueous	24
Solid	8
Tissue	3

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PFAS DRINKING WATER ACCREDITATIONS

	EPA Method 537.1	EPA Method 533
Number of Accredited Laboratories	22	14

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DOD ELAP OVERSIGHT OBSERVATIONS


EDQW Oversight Observations performed in CY2023

Accreditation Body	Number of Visits
A2LA	2
ANAB	5*
PJLA	2
IAS	0
Total	9

* One remote assessment (attended in/out brief only)

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DOD ELAP METHOD 1633 EDQW MEMORANDUM

EDQW distributed a memorandum on February 1, 2023


- Provided clarifications/instructions on 1633 accreditation listings
- Outcome: Accreditation shall not be given for a "1633 Modified" methods
- The EDQW ELAP does not require laboratory accreditation to any particular Draft version, and will not identify version numbers on a laboratory's DENIX listing

EDQW updated 1633 memorandum on August 15, 2023

- Clarifying the DoD has not change direction on the intent of the method based on the release of Draft EPA Method 1633 Version 4

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ASSISTANT SECRETARY OF DEFENSE MEMORANDUM

Sampling of Per- and Polyfluoroalkyl Substances in DoD-owned Drinking Water Systems– July 2023

- Applies to DoD-owned drinking water systems world-wide
- EPA methods 533 and 537.1 are **BOTH** required
- Stipulates analytes that are to be reported by each method
- Requires the use of DoD ELAP accredited Laboratories where available
- Laboratories must participate in PFAS PT's

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ASSISTANT SECRETARY OF DEFENSE MEMORANDUM

Establishing a Consistent Methodology for the Analysis of Per- and Polyfluoroalkyl Substances in Matrices Other than Drinking Water – August 2023

- For definitive analysis of matrices other than drinking water, the DoD Components will use Draft Method 1633
- Other methods for analysis may be considered for screening samples to determine the presence or magnitude of PFAS concentration, **but not to confirm absence.**
- The use of alternative screening methods for a location must be approved by a DoD project representative (e.g., chemist).
- Methods other than Draft Method 1633 shall not be used to analyze samples for regulatory compliance, risk assessment, or comparison to a project screening or action level.

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QSM VERSION 6.0

- Implementation six months after release date
- Implementation with routine accreditation cycle
- Accreditation mandatory 30 months after release
- No advantage to early accreditation

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DENIX

This information may not be up to date. Please verify all information found on this page with th

Lab Name: All

State: All

Matrix: All

Subfield: All


Method: All

Return Lab Info Only? No Yes

The DENIX "Subfield" section removed from the DoD ELAP accredited search.

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DENIX

Environmental Data Quality Workgroup

Contact POC

Search Environmental Data Quality

Contact the POC for Environmental Data Quality Workgroup

Home

Training

Accreditation

Contact POC

Privacy Advisory: The use of information provided is subject to the 163303 Privacy Policy. You may opt out of providing contact information in this form. If you do so, we will not be able to provide a response.

*Required
Your Name:

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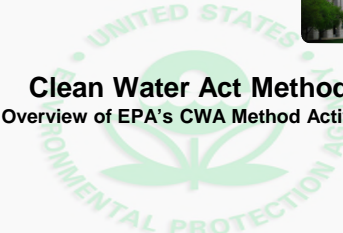

12



QUESTIONS?

William (Ed) Corl, Ph.D.
Chemist & Deputy Director
NAVSEA LQAO
757-396-2227
william.e.corl.civ@us.navy.mil

Judy Solomon
Chemist, NAVSEA LQAO
757-396-1511
judith.a.solomon7.civ@us.navy.mil





Clean Water Act Methods

Overview of EPA's CWA Method Activities


August 2023, Adrian Hanley, U.S. EPA

1




CWA Analytical Methods Program

- Many industries and municipalities are permitted to discharge pollutants under the CWA NPDES
- They use analytical methods to analyze the chemical, physical, and biological components of wastewater and other environmental samples for monitoring compliance
- CWA requires that EPA establish test procedures to measure pollutants for CWA programs through rulemaking, including taking public comments
- EPA promulgates test procedures in 40 CFR Part 136. A method is approved for national use in NPDES permits when it is promulgated.



2




EPA's CWA Methods Team

Jesse Pritts – Branch Chief and Manager for method activities in the Engineering and Analysis Division

Team Members:

- Adrian Hanley – Methods Team Leader, Chemist
- Lemuel Walker – National ATP Coordinator, Chemist
- Bekah Burket – Chemist
- Tracy Bone – Microbiology Lead, Microbiologist
- Meghan Hessenauer – Whole Effluent Toxicity Lead, Biologist


3



Methods Update Rules (MURs)

- Plan to propose and finalize MURs more frequently
 - Smaller rules
 - Less wait time for revisions, Alternate Test Procedures (ATPs), corrections
- A "Routine MUR" every 1-3 years
 - Routine MURs will contain non-controversial items
 - ATPs, minor editorial updates and revisions to methods (EPA, VCSBs, etc.)
- Full MURs will contain more controversial items (i.e., new methods) and be proposed separately and less frequently


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Routine MURs

- 2023 Routine MUR
 - Proposed February 21, 2023
 - Accepted public comments through April 24, 2023
 - <https://www.epa.gov/cwa-methods/methods-update-rules#current>
 - Proposed standardized language to revise EPA membrane filtration Methods 1103.2, 1106.2, 1600.1, and 1603.1 found in Tables IA and IH
 - 7 ASTM method revisions, 39 SM revisions
 - 5 New SM methods – same as previously approved technologies
 - 2 Alternate Test Procedures for Dioxins and Furans (EPA Method 1613B)

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CWA Microbiology Method Activities

Update EPA Microbial Methods in the 2022 Routine MUR


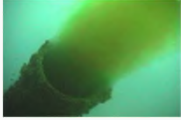
- Updated some of the older EPA Micro Methods
- Revisions include:
 - Update equipment (e.g., no mercury thermometers, add disposable culture dishes)
 - Standardize language between methods, e.g., QA, scope, legal disclaimer

Revising the CWA Microbiology Alternate Test Procedure (ATP) Protocol

6

CWA Microbiology Method Activities


- Rapid methods for *E. coli* and enterococci by droplet digital PCR in ambient water
- Single-laboratory validation completed
 - Two laboratories participated
- Shortens response time for swimming advisories

7

Absorbable Organic Fluorine (AOF) Draft Method 1621

- Thousands of PFAS exist
- Increasing demand for aggregate methods like AOF
- Naturally occurring organofluorines are rare
- Collaborated with ASTM D19 and EPA ORD on single-laboratory validation of AOF screening method



8

AOF, Draft Method 1621 (cont.)

- Single-Laboratory Validation Included:
 - Calibration and sorbent testing
 - Recovery ranged from about 40-200% for analytes tested:
 - 36 individual PFAS
 - 3 different mixed PFAS standards
 - 3 fluorinated pharmaceuticals
 - 3 fluorinated pesticides
 - Method detection limit of 3 ppb
 - Ten wastewater and surface water matrices were tested at two spike concentrations
- Draft method and single laboratory validation report:
 - <https://www.epa.gov/cwa-methods/cwa-analytical-methods-and-polyfluorinated-alkyl-substances-pfas>

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AOF, Draft Method 1621 (cont.)

Multi-Laboratory Validation

- Study Plan/QAPP finalized
- Recruited 6 contract laboratories and 5 volunteer laboratories
- 9 Laboratories have successfully completed calibration and initial demonstration of capability
- Analysis of 9 wastewater and surface water matrices currently underway
- Anticipate finalizing the method in 2023

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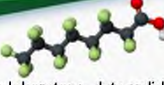
PFAS Method 1633 Validation

- Solid-phase extraction isotope dilution method
 - Based on an SOP originally developed by SGS AXYS
 - DoD is funding and managing both single- and multi-laboratory validation studies of the method, in consultation with EPA OW and OLEM
 - The goal is to provide EPA OW with the documentation needed to consider promulgation of this method at 40 CFR 136. OLEM plans to leverage the validation data to support an SW-846 method.
 - Test matrices include: wastewater, surface water, groundwater, landfill leachate, soil, sediment, biosolid, and fish tissue (includes shellfish)
- Single-Laboratory Validation Completed
 - Draft Method 1633 and single laboratory validation study report are both posted on the web: <https://www.epa.gov/cwa-methods>

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PFAS Method 1633 Validation (cont.)

- Multi-Laboratory Validation
 - Includes 10 participant laboratories, referee laboratory, data validators, and statisticians
 - All laboratory analyses have been completed, and data packages have been received and reviewed
 - MLV Report for aqueous samples (WW, SW, GW) published on EPA and DoD websites July 2023
 - Currently reviewing data, performing statistical analysis, and writing the multi-laboratory report for remaining matrices
- Method Revisions
 - Draft 4 released July 2023 contains final aqueous QC criteria
 - Final method anticipated in late 2023



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Method 1633 Aqueous MLV

- Method Detection Limit – Blank Calculation (MDL_b)
 - 400 individually calculated MDL values
 - 40 analytes X 10 laboratories = 400 individual MDLs
 - 4 MDL_b values above ND, from 2 laboratories
 - 1 MDL_b value above the MDL_s
- Pooled Method Detection Limit (MDL)
 - 27 of 40 below 1 ng/L
 - 6 between 1 and 2 ng/L
 - 3 between 2 and 3 ng/L (6:2 FTS, 8:2 FTS, and 3:3 FTCA)
 - 4 between 3 and 10 ng/L (NMeFOSE - 3.8, NEtFOSE - 4.8, 7:3FTCA - 8.7, and 5:3FTCA - 9.6)

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Method 1633 Aqueous MLV (cont.)

- Ongoing Precision and Recovery (OPR) **Low-Level OPR (LLOPR)**
 - The performance was about the same for the OPR and LLOPR, so the data were combined and used to develop a single set of criteria
 - Most criteria are inclusive of the highest and lowest observed data point from all 10 laboratories
 - No criteria are more stringent than 70-130%
 - Lowest lower acceptance limit was 50%, highest upper acceptance limit was 160%

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Method 1633 Aqueous MLV (cont.)

- 24 Extracted Internal Standards (EIS)
 - Single set of EIS criteria made from only matrix samples (no blank spikes)
 - Roughly 700 sample results per EIS, from 10 laboratories
 - Used a non-parametric approach (p1 and p99) and professional judgement (e.g., eliminate the EIS compound recoveries from 1 to 2 laboratories for a specific parameter)
 - No criteria are more stringent than 40-130%
 - Lower Limits: 15 at 40%, 1 at 30% (¹³C₇-PFUnA), 1 at 25% (D₉-NEtFOSAA), 6 at 10% (¹³C₂-PFDoA, ¹³C₂-PFTeDA, D₃-NMeFOSA, D₅-NEtFOSA, D₇-NMeFOSE, and D₉-NEtFOSE), and 1 at 5% (¹³C₄-PFBA)
 - Upper Limits: 17 at 130%, 3 at 135%, 1 at 170% (D₃-NMeFOSAA), 2 at 200% (¹³C₂-4:2FTS and ¹³C₂-6:2FTS), and 1 at 300% (¹³C₂-8:2FTS)

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Method 1633 Aqueous MLV (cont.)

- Matrix Spike Results

16

608.3, 624.1, 625.1 QC Criteria Update

- TNI, ACIL, APHL, and WEF have volunteered to provide data to update QC criteria
 - Initial calibration, MDLs, calibration verification, ongoing precision and recovery, surrogate recovery, MS/MSDs
- Secondary Data Collection
 - Use existing data anonymously
 - Volunteer laboratories
 - Perform NPDES compliance monitoring
 - Have an SOP and formal quality system
 - Coordinate with laboratory associations
- Over 20 laboratories recruited, currently beta testing electronic deliverable


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Gross Alpha Beta Method 900.0 Revision


- Clean Water Act approved the original version of EPA Method 900.0 for Gross Alpha Beta in 1980
- In 2018, EPA's OGWDW approved Revision 1 to EPA Method 900.0 for use in drinking water at 40 CFR 141.66(c)
- Plan is to evaluate the performance of the revised method in wastewaters with high total dissolved solids (TDS)
- Study plan finalized
- Laboratory testing ongoing

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Continuous Monitoring Collaboration




- Total residual chlorine pilot study
- Based on EPA Drinking Water Method 334.0
- Hampton Roads Sanitation District's (HRSD) SOP for Online Total Residual Chlorine Analysis approved as a limited use ATP by VA DEQ for compliance analysis of total residual chlorine (TRC) in the contact tank to meet VPDES permit requirements.
- Collaborating with a new Standard Methods Joint Task Group to develop an approach for validating the calibration and measurements resulting from online analyzer technology



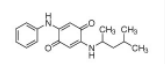
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6-PPDQ Single Laboratory Validation




- University Washington Publication (Science, December 2020)
 - Widespread occurrence of 6-PPDQ at concentrations toxic to salmon
- Method Team: EPA OW, EPA R10, and Eurofins Sacramento
- Eurofins Sacramento SOP: Strata XL cartridge, acetonitrile elution, LC/MS/MS analysis with extracted and non-extracted internal standard (¹³C₁₂-6PPDQ and D₅-6PPD-Q)
- Validation Study
 - Calibration Study
 - Stability/holding time study
 - Initial Demonstration of Capability
 - Testing of 3 stormwaters and 3 surface waters (low and high spike)



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EMC Acrylonitrile and Acrolein Holding Time Study




- At 40 CFR Part 134.3(e) Table II, acrolein and acrylonitrile have a different preservative requirement than the rest of the analytes in Method 624.1 (pH of 4-5 instead of a pH of ≤ 2)
- The Environmental Monitoring Coalition (EMC) led a holding time study determine how long these 2 analytes would remain stable if they were preserved at pH ≤ 2
- EPA reviewed and agreed to the study plan and then reviewed the resulting data and study report
- EPA OW plans to propose a change to the preservation requirement at 40 CFR Part 136.3 for acrylonitrile and acrolein to a pH of ≤ 2 during the next Full MUR

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ATP Reviews




- Alternate test procedures (ATPs) for nationwide use are submitted to EPA HQ for review
 - Codified at 40 CFR 136.4 and 136.5
- Protocols for EPA review of ATPs and new methods are available at:

<https://www.epa.gov/cwa-methods/alternate-test-procedures>


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Contact Information



For more information or additional feedback, please contact:



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23

23




METHOD 1633 QSM TABLE B-24 UPDATE

Janice L. Willey
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 September 2023

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1



Deletions from Table B-24


Ion Transitions

Reason – Inclusion in Method 1633, Section 10.1.1:

“If a qualitative or quantitative standard containing an isomeric mixture (branched and linear isomers) of an analyte is commercially available for an analyte, the quantification ion used must be the quantification ion identified in Table 9, unless interferences render the product ion unusable as the quantification ion. In cases where interferences render the product ion unusable, consult the client before using the alternative product ion and document the reason for the change when reporting results.”

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Deletions from Table B-24


Ion Ratio

Reason – Inclusion in Method 1633, Section 15.1.4:

“If the field sample result does not all meet the criteria stated in Sections 15.1.1 through 15.1.3, and all sample preparation avenues (e.g., extract cleanup, sample dilution, etc.) have been exhausted, the result may only be reported with a data qualifier alerting the data user that the result could not be confirmed because it did not meet the method-required criteria and therefore should be considered an estimated value. If the criteria listed above are not met for the standards, the laboratory must stop analysis and correct the issue.”

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Deletions from Table B-24


Instrument Sensitivity Check (ISC)

Reason – Inclusion in Method 1633, Section 14.1:

“In addition, the measured concentration of each target analyte in the ISC must fall within $\pm 30\%$ of its nominal concentration. If that requirement cannot be met for any target analyte relevant to a project, analysis must be halted, and the sensitivity of the LC-MS/MS system adjusted before analysis of field or QC samples.”

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Deletions from Table B-24

Initial Calibration Verification (ICV)

Not required by Method 1633
 Reason - Deleted in all Table B's

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Deletions from Table B-24


Non-extracted Internal Standard (NIS) Compounds

Reason – Inclusion in Method 1633, Section 14.9:


“The NIS areas in the field samples and QC samples must be within 50 to 200% of the area of the calibration verification standard run at the beginning of the analytical sequence (i.e., a factor of 2).”

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Deletions from Table B-24




Bile Salt Standards

Reason – Inclusion in Method 1633, Section 10.3.5:


“The laboratory must analyze a bile salt interference check standard (see Section 7.5) after the initial calibration and during each analytical sequence (see Section 13.3) as a check on the chromatographic conditions, regardless of the sample matrix to be analyzed.”

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Deletions from Table B-24




Bile Salt Standards

Reason – Inclusion in Method 1633, Section 14.2:


“The retention time of the bile salts in the standard in Section 7.5 must fall at least one minute outside the retention time window for any of the linear or branched PFOS isomers in the standard described in Section 7.3.3. If this requirement cannot be met, the chromatographic conditions must be adjusted to meet the requirements and the initial calibration must be repeated before any field samples are analyzed.”

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Deletions from Table B-24




Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Not required by Method 1633


Reason – MLV data provided technical justification for exclusion.

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AFFF Samples




Minimum Frequency


- Each AFFF sample.
- Note: This does not include AFFF samples that are to be evaluated for MIL-PRF-14385 compliance. Those AFFF samples shall be evaluated in compliance with DoD AFFF01, not Method 1633.
- A copy of the latest version of DoD AFFF can be found at <https://denix.osd.mil/edqw/>

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AFFF Samples




Acceptance Criteria

- AFFF samples shall be subsampled in duplicate for analysis in accordance with DoD AFFF01, Section 11.2.1 through 11.2.9.
- All AFFF samples shall be prepared and analyzed in duplicate in the same manner as aqueous samples (SPE, carbon cleanup).


Fluorine-free fire-fighting foams (F3s) fall under the AFFF media type

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Instrument Blank (IB)



Acceptance Criteria


- No analyte detected > ½ LOQ.

Corrective Action & Qualification Criteria


- If acceptance criteria are not met after the highest calibration standard, calibration shall be performed using a lower concentration for the highest standard until acceptance criteria is met.

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Instrument Blank (IB)




Corrective Action & Qualification Criteria


- *If field sample analyte concentrations exceed the highest calibration standard and the same analytes in the following field sample or in consecutive following field samples also exceed the IB acceptance criteria (i.e., > 1/2 LOQ), the affected samples shall be reanalyzed using a fresh aliquot of the sample extract.*
- *If the extract cannot be reanalyzed and re-extraction is not possible, apply qualifier to affected results and explain in the case narrative.*

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Extracted Internal Standard (EIS)




Acceptance Criteria


- *Isotopically labeled analogs of analytes shall be used when they are commercially available.*
- *Where Method 1633 does not provide EIS recovery acceptance criteria for the sample matrix under evaluation, a laboratory shall use laboratory-developed recovery acceptance criteria no wider than limits any acceptance criteria provided by the customer. Preliminary laboratory-developed acceptance criteria of 20-150% shall be used until laboratory acceptance criteria are developed in accordance with Method 1633.*

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Extracted Internal Standard (EIS)



Acceptance Criteria


- *Where Method 1633 does not provide EIS recovery acceptance criteria for the sample matrix under evaluation, the lower limit of in-house acceptance criteria cannot be < 20%.*

Corrective Action & Qualification Criteria


- *Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, follow the requirements listed in Method 1633, Section 15.3.2. If EIS recoveries still fall outside of the acceptance range, the client must be contacted for additional measures to be taken.*

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Method Blank (MB)




Acceptance Criteria


- *No analytes detected > 1/2 LOQ.*

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LCS & LLLCS




Acceptance Criteria


- *Where Method 1633 does not provide LCS and LLLCS recovery acceptance criteria for the sample matrix under evaluation, a laboratory shall use laboratory-developed acceptance criteria no wider than the project limits. Preliminary in-house acceptance criteria of 40-150% shall be used until in-house limits are generated in accordance with Method 1633.*
- *Where Method 1633 does not provide LCS and LLLCS recovery acceptance criteria for the sample matrix under evaluation, the in-house acceptance criteria cannot be < 40%.*

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Matrix Duplicate (MD)



Minimum Frequency


- *Each AFFF sample prepared using an aliquot of the field sample must be prepared in duplicate.*

Acceptance Criteria


- *RPD of all analytes ≤ 30% between sample and MD.*
- *RPD criteria does not apply if both results are below the LOQ.*

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Matrix Duplicate (MD)




Corrective Action & Qualification Criteria


- If MD results are not within the acceptance criteria, the data shall be evaluated to determine if the source(s) of failure is an analytical error. If so, reprepare and analyze if sufficient sample material is available.
- Qualify specific analyte(s) in the parent sample if results are not within acceptance criteria and explain in the case narrative.

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Frequent Errors Observed




- ❖ Calibration curve forced through the origin
- ❖ EIS concentration lower than required
- ❖ ISC concentration not at the LOQ
- ❖ LLOPR concentration not at 2 x LOQ
- ❖ EIS associations were not compliant with EPA 1633
- ❖ Required forms were missing from data packages
- ❖ Sample preparation instructions from client were not followed
- ❖ Samples passed through the SPE too fast

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Frequent Errors Observed




- ❖ Reporting requirements were not followed:
 - Multiple results were reported in EDD for an analyte in sample
 - NIS and/or EISs associated with reanalysis/dilution not included in EDD
 - EDD included data that were not reported (confirmation of failures or dilutions)
 - EDD was missing NIS recoveries or NIS recoveries were incorrect
 - EDD required naming conventions were not used

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Frequent Errors Observed




- ❖ Batching different matrices together
- ❖ Lack of sufficient review of integration of NIS and EIS
- ❖ Lack of explanation of issues in Case Narratives
- ❖ CALCULATION ERRORS!!!


**FULL DATA PACKAGES & STAGE 4 VALIDATION OF METHOD
1633 IS HIGHLY RECOMMENDED**

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Interferences




In addition to bile salts (PFOS):

PFPeA (263 → 219) and PFBA (213 → 169)

- PFPeA confirmed interference: diprotic unsaturated fatty acid (mass 263.1288)
- PFBA confirmed interference: saturated oxo-fatty acids (mass 213.1496)

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Thank you for your attention!

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EPA PFAS Methods Update, Part 2: Source Characterization Methods

Erin Shields*, Lara Phelps, Jeff Ryan, Stephen Jackson, Ariel Wallace, William Roberson



Office of Research and Development
Center for Environmental Measurement and Modeling, Air Method and Characterization Division

September 19, 2023

1

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Overview

- Stationary source air measurement needs
- What is an Other Test Method (OTM)?
- OTM-45 – Modified Method 0010 (MM0010) train with liquid chromatography – mass spectrometry (LC/MS)
- OTM-50 – Canister and gas chromatography – mass spectrometry (GC/MS)
- Potential future method for semivolatile nonpolar PFAS/fluorochemicals
- Application of methods

2

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Needs for PFAS Air Emissions Measurements

- Reliable and **accepted** emission measurement methods are needed to measure volatile, semivolatile, nonvolatile, polar, and nonpolar per- and polyfluoroalkyl substances (PFAS) for multiple purposes and sources
- Ability to measure parent compounds and products of incomplete combustions or destruction (PICs or PIDs)
- Reliable and comprehensive emissions data are needed to:
 - Support State regulatory processes
 - Inform Federal decision making
 - Support research
 - **Conduct comprehensive source characterizations and technology assessments**

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3

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Measurement Challenges

- Process can alter emission composition ...

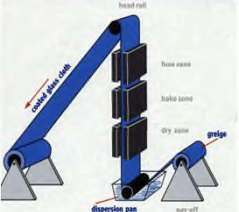
PFPO-DA, GenX, HFPO-DA
(CAS 14870-33-6
CF3(CF2)6OH/CF3(CF2)7OH)

Soluble, polar
boiling point = 151 °C

→ 90 °C

HFPO-DA decarboxyl
(CAS 4888-65-2
CF3(CF2)6F)

Insoluble, nonpolar
boiling point = 39 °C



4

4

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Evaluating Destruction – Source Methods

<p>Volatile nonpolar fluorochemicals OTM-50 – to be released 12/2023</p> <ul style="list-style-type: none"> • Evacuated canister method with GC/MS analysis • Measures known PIDs and commercial compounds • -128 – 118 °C boiling point range • Lower DL than FTIR 	<p>Semivolatile nonpolar fluorochemicals OTM-55 – under development</p> <ul style="list-style-type: none"> • Modified MM0010 train • GC/MS, like 8270 with TICs • Nonpolar molecular growth and other compounds • 100 - 300 °C boiling point range
<p>Volatile polar fluorochemicals No current method</p> <ul style="list-style-type: none"> • Ultra-short chain PFAS from one to four carbons long • Many, like trifluoroacetic acid, are in most background samples • Volatile carboxylic acids could be degradation products • Development may start in 2024 	<p>Non- and semivolatile polar PFAS OTM-45 – out since 2021</p> <ul style="list-style-type: none"> • Can measure polar “legacy” PFAS (C4 and longer) • LC/MS analysis related to Methods 533, 537, and 1633 • Sub-nanogram per cubic meter detection limits are possible

5

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What is an Other Test Method (OTM)?

- Formal method posted by Office of Air Quality Planning and Standards (OAQPS) in the Emission Measurement Center (EMC)
- <https://www.epa.gov/emc/emc-other-test-methods>
- Method not yet subjected to Federal rulemaking process
- Useful and available to the measurement community
- Supported by field and laboratory data
- Reviewed by OAQPS technical staff
- May be basis for promulgated method

6

6

OTM-45 Basics

- Based on a Modified Method 5 (MM5) train
- Incorporation of a breakthrough (BT) trap for method testing
- Alkaline methanol extraction
- Uses pre-sampling and pre-extraction standards
- Analysis by LC/MS with isotope dilution – influenced by Methods 533 and 1633
- Extensive use of blanks (proof, field, reagent blanks, and more)

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OTM-45 Feedback

- Need to ensure that final extract from wet XAD is 80% alkaline methanol to ensure passing extraction standard recoveries
- The number of required blanks – help determine contamination
- XAD regularly is contaminated with targeted PFAS
- Breakthrough trap – calculations and necessity
- Appropriate materials – PTFE, stainless. . .
- Hold times

8

OTM-45 Future Directions

- Revise OTM-45 (in progress)**
 - Incorporate internal standards from 1633 to simplify standard acquisition and preparation
 - Address comments provided from users
- Continue laboratory work
- Continue in-house pilot process
- Continue to seek operating facility field evaluations
- Continue to solicit feedback for potential method revisions
- Consider potential Method 301 studies
 - Needed to support the regulatory process
 - Spike combustion emissions with targeted compounds
 - Collect four trains simultaneously to evaluate precision and bias

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OTM-50 for Nonpolar Volatile Fluorochemicals

- Approach largely based on TO-15A and Proposed Method 327
- Sample collected using evacuated canisters
- Use impingers to manage moisture/acid gases for source sampling (limits use to nonpolar)
- Analysis by GC-MS with cold trap preconcentration
- Applicable to multiple sources
 - No impingers needed for acid free emissions
 - Near-source or fence-line sampling possible

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OTM-50 Train

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OTM-50 Analytical Approach

1	88.0, 128 C	70.01, 82 C	52.02, 52 C	34.03, 79 C	104.46, 81 C	86.47, 41 C	137.37, 24 C	6	338.04, 96 C	320.90, 70 C
2	138.01, 78 C	120.01, 48.5 C	102.3, 27 C	84.04, 47 C	100.01, 76 C			7	388.05, 80 C	370.06, 96 C
3	188.02, 39 C	170.03, 58 C	150.02, 27 C	166.02, 27 C				8	438.06, 103 C	420.07, 118 C
4	238.01, 1 C	220.04, 13 C	200.03, 6 C					E	288.03, 39 C	432.08, 101 C
5	288.03, 39 C	270.04, 42 C	252.04, 27 C							

- Currently using a Porous Layer Open Tubular (PLOT) column to target 30 C₁-C₈ per/polyfluorocarbon compounds, most with <0.4 µg/m³ detection limits
- Combination of PICs and industrial compounds
- Target list is expanding using tentatively identified compounds (TICs)

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Generating OTM-50 Target List

PFAS-containing samples collected and analyzed from sources including:

- Combustion/Incineration
- Pyrolysis
- Super Critical Water Oxidation (SCWO)
- Thermal Treatment Applications
- Others

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OTM-50 Status and Future Directions

- Post it by **12/31/2023**, on www.epa.gov/emc
- Preparation of OTM-50 in progress
 - Guidance standard preparation
 - Guidance for "tentatively identified compounds" (TICs)
- Continue to identify unknowns or commercial products & incorporate into target list as needed
- Continue to evaluate measurement performance
 - Determine recoveries of targets through impingers for source sampling
 - Explore standard addition of targets for sample-specific overall measurement quality assessment
- Evaluate options for improving measurement sensitivity

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Method(s) for Nonpolar Semivolatile "PFAS"

- Need a method for fluorotelomer alcohols (FTOHs)
- Need a method to characterize potential PICs/PIDs of concern
- Approach based on SW-846 Methods 0010/3542/8270
- Method OTM-55 ...

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Status and Future Directions

- Developing isotope dilution analytical approach for FTOHs
- Developing a PICs/PIDs target list and analytical approach
- Evaluating sequential extraction approach to enable single sample train for polar and nonpolar semivolatile PFAS
- Ultimate goal is preparation of OTM-55 for commercial use
- Guidance for TICs an important component

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Application of Source Methods

It is important to characterize emissions from PFAS treatment technologies to evaluate their efficacies

- Need to know the extent of the initial PFAS' destruction
- Need to determine what byproducts of destruction are emitted
- OTM-45 and OTM-50 have been applied to PFAS incineration studies with the EPA's pilot-scale incinerator

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ORD's Pilot-scale combustor

- Rainbow Furnace – a pilot-scale tunnel furnace
 - Uniform plug-flow furnace
 - Best-case scenario for incineration

18

18

Light Water AFFF



- Light Water is a common pre-2001 AFFF that contains perfluorinated surfactants, PFOS predominantly
- Contains around 2% PFAS and only about 1% of those can be quantitated

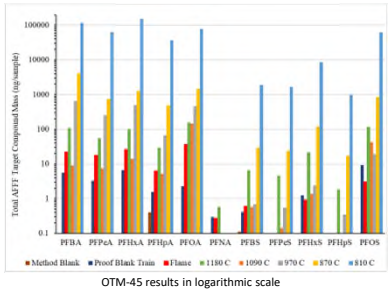
Compound	ppb (ng/g)	weight %
Perfluorobutanoic acid (PFBA)	6850	0.000685
Perfluorobutanesulfonic acid (PFBS)	202000	0.0202
Perfluoropentanoic acid (PFPeA)	34100	0.00341
Perfluoropentanesulfonic acid (PFPeS)	164000	0.0164
Perfluorohexanoic acid (PFHxA)	111000	0.0111
Perfluorohexanesulfonic acid (PFHxS)	1180000	0.118
Perfluoroheptanoic acid (PFHpA)	31600	0.00316
Perfluoroheptanesulfonic acid (PFHpS)	136000	0.0136
Perfluorooctanoic acid (PFOA)	123000	0.0123
Perfluorooctanesulfonic acid (PFOS)	8020000	0.802
Targeted Organic Fluorine	6410000	-

3M Light Water 3% AFFF, FC-203CF

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OTM-45 PFAS Emissions

- Blank trains and QA samples had trace levels of PFAS
- Around 1100+ °C samples had PFAS concentrations near the blank levels
- Below 900 °C microgram per cubic meter concentrations of PFAS were emitted
- Exposure to the flame created the lowest concentrations
- Some Carryover may have impacted the 1180 °C run, it was after the lowest temperature run



OTM-45 results in logarithmic scale

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PFAS Destruction Efficiencies (DEs)

- Flame exposure produced 99.999+% destruction for PFAS, except PFBA (possible contamination?)
- The sulfonates had higher DEs than the carboxylic acids
 - >99.999% DE even below 900 °C
 - May indicate carboxylic acids are a product of incomplete destruction (PIC)
- Around 1100 °C the DEs are all above 99.99%
- Do high DEs mean that the PFAS are mineralized?

Method 19 Destruction Efficiencies						
Temperature, (°C)	Flame	1180	1090	970	870	810
PFBA	99.9958	99.9725	99.9978	99.8443 ^a	98.3336 ^a	43.7362
PFPeA	99.9993	99.9971	99.9996	99.9875 ^a	99.9372 ^a	94.0300
PFHxA	99.9997	99.9984	99.9998	99.9925	99.9678	95.6188
PFHpA ^a	99.9997	99.9984	99.9997	99.9965	99.9566	96.3086
PFOA	99.9996	99.9978	99.9981	99.9938 ^a	99.9663 ^a	97.9522
PFBS ^a	-99.9999	-99.9999	-99.9999	-99.9999	99.9996	99.9704
PFPeS	-99.9999	-99.9999	-99.9999	-99.9999	99.9996	99.9671
PFHxS	-99.9999	-99.9999	-99.9999	-99.9999 ^a	99.9997	99.9768
PFHpS	-99.9999	-99.9999	-99.9999	-99.9999 ^a	99.9996	99.9766
PFOS	-99.9999 ^a	-99.9999	-99.9999 ^a	-99.9999 ^a	99.9997	99.9751

^aPFBS and PFHpA were detected in the analytical method blanks. ^bThe extraction internal standards were outside of acceptance criteria. DEs used estimated maximum concentrations.

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OTM-50 AFFF Fluorinated By-Products

- Nonpolar volatile fluorochemical emissions (PICs) were sampled and analyzed using canisters and GC/MS

The major volatile fluorochemical PICs were a homologous series of 1H- and perfluoroalkanes

Total concentration of volatile PFAS

Temperature (°C)	Concentration (µg/m ³)
Flame	1.1
1180	3.0
1090	1.22
970	294
870	2460
810	26,500

1H molecules can oxidize into carboxylic acids in the environment, forming PFCAs again¹

¹ Ehl, D.A., et al., 2004. Environ Sci Technol 38, 3316-3323. <https://doi.org/10.1021/es040640w>

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Conclusions

- The presence or absence of PICs is the best indication of mineralization
 - DE or destruction and removal efficiency (DRE) should not be the primary metric used to evaluate the efficacy of PFAS destruction technologies
 - There can be 99.99% DE for targeted PFAS loss, but still have near ppm levels of other PFAS being emitted
- Accepted PFAS and PFAS-related emissions measurement methods are needed to identify the parent PFAS and PIDs
- Development of OTMs are recognized as what's needed for accepted use
 - OTM-45 is currently available for polar semivolatile PFAS
 - OTM-50 in development for nonpolar volatile PFAS
 - Currently accelerating development of method for nonpolar semivolatile PFAS
- Identifying what compounds need to be targeted for measurement is the hard part
- Application to thermal treatment/incineration/combustion sources is a major focus

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Disclaimer:

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- Support from the EPA's Sustainable and Healthy Communities (SHC) and Air, Climate, and Energy (ACE) Research Programs
- Application research was supported by the EPA and the DoD's Strategic Environmental Research and Development Program (SERDP)

Questions??

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SERDP ESTCP

SERDP & ESTCP PFAS Sampling & Analysis Initiatives

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<https://www.serdp-estcp.org>

1

1

SERDP ESTCP

DoD's Environmental Technology Programs

SERDP
Science and Technology

- Statutory program established 1991
- DoD, DOE, EPA partnership
 - Advanced technology development to address near-term needs
 - Fundamental research to impact real world environmental management

ESTCP
Demonstration and Validation

- Demonstrate innovative cost-effective environmental and energy technologies
 - Transition technology out of the lab
 - Establish cost and performance
 - Partner with end user and regulator
 - Technology transfer
 - Accelerate commercialization or broader adoption
 - Direct technology insertion

2

2

SERDP ESTCP

Focus Areas

- PFAS and AFFF
- Installation restoration
- Installation resilience
- Munitions remediation
- Sustainable energetics
- Corrosion and repair technologies
- Electrical infrastructure and planning



3

3

SERDP ESTCP

PFAS & AFFF Subtopics

- ❖ **Management of PFAS in the Environment**
 - ✓ Occurrence, Fate, & Transport
 - ✓ Treatment of PFAS-Impacted Matrices
 - ✓ Sampling & Analysis
 - ✓ Ecotoxicity of PFAS
- ❖ **PFAS-Free Firefighting Formulations**
 - ✓ Ecotoxicity of PFAS-Free Formulations
 - ✓ Development of New Replacements
 - ✓ Field Demonstrations
 - ✓ Aircraft Rescue & Firefighting Cleanup

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SERDP FY19 SON

Develop improved analytical and environmental sampling techniques for PFAS.

- Development of sampling techniques to evaluate soil and water columns, including consideration of potential biases associated with sampling supplies and equipment, and decontamination procedures for use at both minimally and highly impacted sites.
- Evaluation of potential media to be used for passive samplers and their performance.

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SERDP ESTCP

SERDP FY19 SON

- Assessment of subsampling techniques to determine the process by which the subsample provides results that are most representative of the entire sample collected.
- Development of procedures to assess the total organofluorine in environmental waters, soil, and sediment.
- Development of rapid field screening procedures for PFAS.
- Development of extraction techniques to produce the most accurate and precise quantitation.
- Evaluation of techniques to eliminate matrix interference.

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SERDP FY19 SON

- Evaluation of techniques to achieve the lowest limit of quantitation possible when analyzing AFFF formulations and samples containing high concentrations of PFAS while achieving the required precision and accuracy.
- Evaluation of techniques that could be used to ensure precision and accuracy of total PFAS analytical procedures.

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SERDP FY19 Funded Projects

- *Bench-Scale Assessment of Nuclear Magnetic Resonance (NMR) and Complex Resistivity (CR) Screening Technologies for Rapid Assessment of PFAS in Soils and Sediments*, Lee Slater, Rutgers University Newark
- *Developing PIGE into a Rapid Field-Screening Test for PFAS*, Graham Peaslee, University of Notre Dame
- *Development and Validation of Analytical Methods for Comprehensive Profiling of PFAS in Firefighting Foam Impacted Environmental Matrices*, Jinxia Liu, McGill University

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SERDP FY19 Funded Projects

- *Assessing and Mitigating Bias in PFAS Levels during Ground and Surface Water Sampling*, Jennifer Field, Oregon State University
- *Rapid Site Profiling of Organofluorine: Quantification of PFAS by Combustion Gas Analysis*, David Hanigan, University of Nevada, Reno

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ESTCP FY19 Funded Project

- *Validation of Streamlined Mobile Lab-Based Real-Time PFAS Analytical Methods*, Joseph Quinnan, Arcadis

Demonstrate the application of three real-time mobile laboratory methods for PFAS including a DoD LC/MS/MS method, an accelerated LC/MS/MS method for quantitative screening, and methylene blue active substances (MBAS) assay for semi-quantitative screening at source areas.

Publication: Quinnan, J., M. Rossi, P. Curry, M. Lupo, M. Miller, H. Korb, C. Orth, and K. Hasbrouck. 2021. *Application of PFAS-Mobile Lab to Support Adaptive Characterization and Flux-Based Conceptual Site Models at AFFF Releases*. *Remediation*, 2021:1-20

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SERDP FY20 SON

Develop improved forensic methods and tools for source tracking and allocation of PFAS.

- Evaluation of conventional or novel analytical techniques or methodologies to differentiate PFAS from AFFF versus non-AFFF sources.
- Develop spectral libraries of PFAS to include both AFFF-derived PFAS as well as PFAS derived from other sources.
- Improved analytical methods and/or validated models to predict changes to AFFF mixtures over time, including chemical pathways to the most toxic compounds.

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SERDP FY20 Funded Projects

- *Improving Access and Utility of Analytical Data for the Confident Discovery, Identification, and Source-Attribution of PFAS in Environmental Matrices*, Benjamin Place, National Institute of Standards and Technology
- *Comprehensive Forensic Approach for Source Allocation of PFAS*, Lead Investigator: Christopher Higgins, Colorado School of Mines
- *Establishing an Approach to PFAS Forensics and a PFAS Source Materials Forensic Library*, Lead Investigator: Mark Benotti, NewFields Government Services

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SERDP FY20 Funded Projects

- *Machine Learning Pattern Recognition for Forensic Analysis of Detected PFAS in Environmental Samples*, Lead Investigator: Tohren Kibbey, University of Oklahoma
- *Ultrahigh-Resolution Fourier-Transform Ion Cyclotron Resonance Mass Spectrometry for Fingerprinting, Source Tracking, and Allocation of PFAS*, Lead Investigators: Jens Blotevogel, CSIRO
- *A Simple and Robust Forensic Technique for Differentiating PFAS Associated with AFFF from other PFAS Sources*, Lead Investigator: David Sedlak, University of California, Berkeley

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SERDP FY20 SON

Develop standard operating protocols to assess the potential for leaching and mobility of PFAS from solids, soils, and sediments.

- Development of a standardized method, similar to the Synthetic Precipitation Leaching Procedure (SW-846 Method 1312), to assess the leachability and mobility of PFAS from solid matrices.
- Development of new or improved methods to evaluate sorption/desorption affinity of PFAS for specific solid materials of concern to the DoD.

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SERDP FY20 Funded Projects

Original Project:

- *Development and Validation of Novel Techniques to Assess Leaching and Mobility of PFAS in Impacted Media*, Jennifer Guelfo, Texas Tech University

Funded FY23 Follow-on Project:

- *Leaching and Mobility of PFAS from Concrete and Asphalt*, Jennifer Guelfo, Texas Tech University

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SERDP FY20 SON

Develop passive sampling methods to provide repeatable and environmentally relevant measures of PFAS.

- Develop passive sampling media competent to quantitatively “concentrate” the wide range of PFAS of interest from water.
- Establish physical-chemical properties, including sorbent/water partition coefficients, molecular diffusivities of PFAS in water and sorbent media.
- Establish the range of PFAS that can be quantifiably sampled using the sorbent(s).

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SERDP FY20 SON

- Characterize the impacts of co-occurring chemicals and various water quality and conditions.
- Develop a fundamental understanding of the natural solid-water sorption coefficients of PFAS as a function of sorbate properties, natural solids, and solution properties.
- Develop passive samplers that yield representative spatial and temporal interrogation of site chemicals of concern when deployed.
- Develop passive sampling methods/procedures that are capable of being efficiently deployed and retrieved in widely varying field applications.

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SERDP FY20 Funded Projects

- *Development of Novel Functionalized Polymeric Thin Films for Equilibrium Passive Sampling of PFAS Compounds in Surface and Groundwater*, Upal Ghosh, University of Maryland, Baltimore County
- *Development and Field-Testing of Advanced Passive Samplers for PFAS*, Rainer Lohmann, University of Rhode Island
- *Development of Passive Sampling Methodologies for PFAS*, Sarit Kaserzon, The University of Queensland

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SERDP FY20 Funded Projects

- *Development of a Novel PFAS Passive Sampler with Efficient Sorbent Media and Robust Membrane Barrier*, Yin Wang, University of Wisconsin, Milwaukee
- *Passive Samplers for PFAS with Innovative Sorbents*, Mei Sun, University of North Carolina at Charlotte
- *Ion Exchange Membranes and Fibers as Passive Samplers for Chemically-Diverse PFAS*, Lee Blaney, University of Maryland, Baltimore County

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SERDP FY20 Funded Projects

- *Development of a Diffusive Gradients in Thin-Films Passive Sampling Methodology for PFAS in Water*, Julian Fairey, University of Arkansas
- *Osorb Media Use in PFAS Samplers*, Craig Divine, Arcadis

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SERDP FY23 SON

Develop analytical methods to provide repeatable and environmentally relevant measures of total PFAS in PFAS-free firefighting formulations to demonstrate that they do not contain PFAS at concentrations above 1 part per billion (ppb).

- Develop robust sample preparation procedures that are implementable in a production laboratory environment.
- Develop methods for accounting for or eliminating inorganic fluorine from total PFAS quantitation.

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SERDP FY23 SON

- Develop methods to reduce the impact of other constituents on total PFAS quantitation.
- Minimize the potential for exclusion of PFAS, such as short-chain PFAS, from total PFAS quantitation.
- Validate the method in accordance with a validation study plan consisting of all elements require by the current version of the DoD QSM.
- Document the method in a format that is compatible with DoD QSM requirements and allows for easy implementation by production laboratories.

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SERDP FY23 Funded Projects

- *Method to Measure PFAS in ML-SPEC Firefighting Formulations by Extraction Using Osorb and Advanced Sorbents with Organofluorine Analysis*, Paul Edmiston, College of Wooster
- *Development of Two New Total Organic Fluorine Methods to Determine Total PFAS in PFAS-Free Firefighting Formulations at Trace Levels*, Susan Richardson, University of South Carolina
- *Surface-Enhanced Raman Spectroscopic Analysis of PFAS in Firefighting Formulations*, Haoran Wei, University of Wisconsin-Madison

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SERDP FY23 Funded Projects

- *Coupling Foam Fractionation with High-Resolution Molecular Absorption Spectrometry Graphite Furnace to Quantify Total PFAS in PFAS-Free Firefighting Formulations*, Young Jeong Choi, Purdue University
- *A Standard Operating Procedure to Quantify Total PFAS in PFAS-Free Firefighting Formulations*, Kyle Doudrick, University of Notre Dame

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SERDP ESTCP

SERDP FY24 SON

Develop improved sampling and analytical methodologies to measure PFAS in the environment.

- Develop field methods designed to rapidly screen or monitor PFAS.
- Develop better laboratory preparation and analysis methods for the inclusion of understudied types of PFAS in various media types.
- Develop and validate laboratory preparation and analysis method for understudied media types.

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SERDP FY24 SON

- Develop laboratory preparation and analysis methods to monitor total PFAS that can achieve a limit of quantitation (LOQ) that is in the ppt to single digit ppb range.
- Develop and validate sampling methods relative to thermal treatment, stormwater sampling, surface water, and sediment sampling.

Projects will be awarded Fall 2023

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SERDP ESTCP

2023 DoD Energy and Environment Innovation Symposium

- November 28th – December 1st
- Arlington, VA

SERDP & ESTCP Webinar Series

- Every two weeks on Thursdays from 12:00 ET to 1330 ET)
- 30 minute presentations followed by interactive Q&A

ESTCP Podcast Series on PFAS Research & Remediation

- Launched with Arcadis
- (<https://soundcloud.com/arcadis-north-america/sets/pfas-research-and-remediation>)

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SERDP ESTCP

Thank you for your attention!

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QUALITY SYSTEMS MANUAL VERSION 6.0 OVERVIEW

John Gumper, ChemVal Consulting, Inc

1



Introduction

A Brief History of the Quality Systems Manual

2



Introduction



- The QSM began as a project of the Department of Defense (DoD) Environmental Data Working Group (EDQW)
- This workgroup's primary mission is to develop and recommend DoD policy pertaining to environmental sampling, laboratory testing operations, and data quality.
- Includes all four branches of DoD

3



Introduction



- These four branches had at least five sets of requirements for environmental laboratories to follow
- EDQW's mandate was to develop one standard for environmental laboratories to be used by all programs
- Rather than "reinvent the wheel", they built the Standard on documents already published
 - NELAC 2003, which was built on
 - ISO/IEC 17025-1999

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ISO



- The International Organization for Standardization is an independent, non-governmental organization, whose membership consists of different national standards bodies.
- ISO/IEC 17025: Internationally agreed upon minimum competencies that all testing laboratories need to have to ensure the results they report are reliable and traceable.
- Use of ISO Standards complies with Federal Government's policy to use public-sector Standards

5



ISO



- ISO Accreditation is provided through Internationally-recognized Third-Party Accreditation Bodies
- EDQW embraced the concept of Third-Party Accreditation
 - Relieves federal government of responsibility for assessing, and maintaining highly-skilled assessors
- Use of internationally-recognized ABs provides the program international standing

6

Introduction

QSM 4.1 – April 22, 2009

- Based on TNI 2003 Standard, ISO/IEC 17025-1999
 - “Grey Boxes” provided additional QSM requirements
- Basis for DoD ELAP using Third-Party, Internationally-Recognized, ISO/IEC 17025 Accreditation Bodies
 - Assessor Training, May 2009
 - First Assessment, June 2009

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Structure of Quality Systems Manual

- Published by EDQW, built upon ISO and TNI Standards
 - QSM: *DoD/DOE Laboratories performing environmental work for DoD/DOE
 - TNI: *Published by The NELAC Institute *Environmental Analysis
 - ISO 17025: *International Standard *Key Testing Laboratory
- Minimum quality requirements for laboratories performing environmental analysis for DoD/DOE
- Specific requirements should be defined by each customer to meet the data quality objectives

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Structure of TNI Standards (b. 2009)

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Introduction

QSM 5.0 (“QSM5”)

- Published in 2013
- Based on TNI 2009 Standard, ISO/IEC 17025-2005
- Added text “in addition to” or “in lieu” of TNI text
 - No “Grey Boxes”
- Then a really cool thing happened
- Basis for both DoD ELAP and DOE DOECAP-AP

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QSM 4.1-2009	QSM 5.0-2013	QSM 5.1-2017
Based on NELAC 2003, 17025-1999	Based on TNI 2009, 17025-2005	Based on TNI 2009, 17025-2005
Base Standards + Grey boxes	QSM Text in line or “in lieu of”	QSM Text in line or “in lieu of”
DoD Only	DoD and DOE Publication	DoD and DOE Publication

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Introduction

- DOECAP began using Internationally-Recognized ISO ABs in 2016
- Publication of QSM 5.1 in January 2017
 - Basis for both DoD ELAP and DOE DOECAP-AP
 - Contained some “DOE Only Requirements”
 - Quick revision to QSM 5.1.1
- We like this one and life is good
- Note: DOECAP-AP began using Internationally-Recognized ISO ABs in 2018

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Transition to QSM 6.0

But the only certainty in life is change...

- So, of course, ISO released its updated ISO/IEC 17025-2017 near the end of the same year
- TNI had published an update the previous year (2016) which would be implemented over the next several years
- And the transition begins

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Versioning Timeframes

ISO/IEC 17025	TNI (NELAC)	QSM
• 1999	• 2002/2003	• 4.1 – 2009
• 2005	• 2009	• 5.0 – 2013
• 2017	• 2016	• 5.2 – 2018
	• In process	• 6.0 – 2023


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QSM 6.0

I think I need a drum roll
please



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QSM 6.0

- Why do we need a revision now?
- Transition Versions result in a unwieldy QSM 5.4
- Guiding principles
- Structure of the new QSM
- Development Process

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Why do we need QSM 6.0?

- ISO Updated to 17025-2017
- Incorporation of Industrial Hygiene analyses
- TNI Updated to TNI 2016
- But, TNI 2016 is not based on 17025-2017
 - TNI does not yet have a standard based on 17025-2017

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TNI 2016

- TNI 2016 made minimal changes to Module 2 (Management Systems = “Quality System”)
- TNI 2016 made significant changes to Modules 4 (Chemistry), 5 (Microbiology), and 6 (Radiochemistry)
 - Including some of these module changes into QSM 6.0 was important

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ISO 17025-2017

- Significant changes in approach
- Significant changes in structure
 - Details will be coming in the presentation tomorrow on Module 2

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Incorporation of Industrial Hygiene

- Previously, IH was sparsely addressed and did not have its own section in the QSM
- Industrial Hygiene (IH) analyses use a somewhat different analytical approach
- Changes to the Chemistry testing requirements cause difficulties for IH
- Decision was to add IH Module (Module 8) and B-Tables to address IH analyses

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QSM 5.2-2018	QSM 5.3-2019	QSM 5.4-2021
Based on TNI 2009, 17025-2005	Based on TNI 2009, 17025-2005	Based on TNI 2009, 17025-2005
Add 17025-2017 in Grey boxes	17025-2017 in Grey Boxes	17025-2017 in Grey Boxes
QSM Text in line or "in lieu of"	QSM Text in line or "in lieu of"	QSM Text in line or "in lieu of"
	Mostly changes in PFAS: B-15	Mostly changes in PFAS: B-24

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QSM 5.2 to QSM 6.0

- So, all these things were added, but it is now an unwieldy document that needs to be revised
- Requires use of
 - QSM 5.4
 - 17025-2005
 - 17025-2017
 - TNI 2009

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QSM 5.4 Looks Like This

4.11 Corrective Action (ISO/IEC 17025/2005(E), Clause 4.11)

Box 13

The following shall be implemented in addition to ISO/IEC 17025:2005(E) Clause 4.11: ISO/IEC 17025:2017(E) sections 8.7.1.a, 8.7.1.b, 8.7.1.e, 8.7.1.f

The following shall be implemented in addition to ISO Clauses 4.11.1 through 4.11.5 and TNI sections 4.11.6 through 4.11.7:

4.11.8 DoD/DOE Requirement

The laboratory shall have and use a record system for tracking corrective actions to completion and for analyzing trends to prevent the recurrence of the nonconformance.

Corrective actions developed to address findings during DoD ELAP or DOECAP-AP assessments must be implemented. Any changes to reviewed corrective action plans must be reviewed by the DoD ELAP AB or the DOECAP-AP AB, as appropriate.

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Guiding Principles

The Subgroup developed some goals for the final product


- Elimination of duplicate language—say it only once!
- Remove guidance and examples, where possible
- Remove non-assessable language, e.g., “The laboratory shall consider...”

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Guiding Principles




More goals


- Remove requirements that are meant for other organizations than the laboratory, e.g., requiring the laboratory to record the date and time sampled when they don't perform the sampling
- Clarify language where the Subgroup is aware different ABs and assessors are coming to different interpretations

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Structure of QSM 6.0




- Using the same modular format as previous versions (Thanks, TNI!)
- Series of Modules with requirements for different areas
- Appendices with method- or technology-specific requirements
- “Volume 1” as a designation was removed because the QSM only has one volume

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Structure of QSM 6.0




- Module 1-Proficiency Testing requirements for labs
- Module 2-Quality System requirements
 - Formatted to reflect order and numbering of 17025-2017
 - Hazardous and Radioactive Material Management (HRMM) Section added at the end (Section 9)
- Module 3-Quality System for Asbestos Testing

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Structure of QSM 6.0




- Module 4-Quality Systems for Chemical Testing
- Module 5-Quality Systems for Microbiological Testing
- Module 6-Quality Systems for Radiochemical Testing
- Module 7-Quality Systems for Toxicity Testing
- Module 8-Quality Systems for Industrial Hygiene Testing (*NEW*)

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Structure of QSM 6.0




Numbering for technical modules (3 – 7) has been changed slightly


- TNI begins every numbered paragraph with a “1.”
 - None of the modules have a number that starts with “2.”
- QSM deleted the leading 1 when numbering the modules
 - And numbered every requirement!

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Structure of QSM 6.0-Appendices




Appendix A- “Reserved”

- Previously contained reporting requirements
 - Not really requirements
 - More like a validation wish list
- Requirements have been folded into Module 2 Reporting Requirements (Section 7.8)
 - Presentation on that section later in workshop

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Structure of QSM 6.0-Appendices

- Appendix B
- Tables of Method- or Technology-specific quality control requirements
 - Tables reformatted
 - Philosophy changed somewhat

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Structure of QSM 6.0-Appendices

- Appendix C-LCS Recovery Limits
 - Small changes to clarify the 8330 Tables and which are applicable when ISM is used
- Appendix D-Non-Destructive Assay Requirements
 - Dropped due to lack of use
- Appendix E-HASQARD Checklist
 - Incorporated into Module 2

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Module 2 Development

This was the big one

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Development of QSM 6.0

Tasks

- Rewrite Module 2 in the structure of ISO/IEC 17025-2017
 - Incorporate language from existing QSM
 - Incorporate language from both TNI Standards
- Determine requirements and write Module for PT (M1)
- Review Technical Modules for incorporation

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Development of QSM 6.0

Biggest task is Module 2

- ChemVal Consulting's first task was to come up with a way to
 - Consider all of the language from all the pertinent standards
 - Track every decision made by the group to include, discard, or edit the language

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Development of QSM 6.0

- Enter the "Mash Table"
 - Build a Table, ordered by ISO/IEC 17025-2017 and align all the requirements from all four documents
 - Remember, 17025-2009 had a different ordering
 - Color-code the different language to track the origin of each sentence
 - Here's an example from "Personnel"

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Mash Table

17025-2017	TNI 2009	TNI 2016	QSM
6.2 Personnel			
6.2.1 All personnel of the laboratory, either internal or external, that could influence the laboratory activities shall act impartially, be competent and work in accordance with the laboratory's management system.	5.2.3 The laboratory shall use personnel who are employed by, or under contract to, the laboratory. Where contracted and additional technical and key support personnel are used, the laboratory shall ensure that such personnel are supervised and competent and that they work in accordance with the laboratory's management system.	5.2.3 The laboratory shall use personnel who are employed by, or under contract to, the laboratory. Where contracted and additional technical and key support personnel are used, the laboratory shall ensure that such personnel are supervised and competent and that they work in accordance with the laboratory's management system.	
			5.2.3 DoD/DOE (Clarification of ISO Clause 5.2.3): The laboratory shall ensure that all personnel, including part-time, temporary, contracted, and administrative personnel, are trained in the basic laboratory quality assurance (QA) and health and safety programs.

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Development of QSM 6.0

First cut

- The Subgroup worked through the entirety of Module 2 comparing the requirements.
- Items which were identical in meaning were cut from the document
- Most of the TNI 2009 and 2016 Standards for Module 2 were identical and were combined at this point (and became red)

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Development of QSM 6.0

- **First cut**
 - Removed redundant language from the presentation, too!
- **Next, created working draft table**
 - Color-coded language was arranged sequentially in a new table
 - Categorized paragraphs for consideration

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Development of QSM 6.0

Key to abbreviations for references:

- N – New
- E – Exactly the same
- F – Functionally the same
- A – The same, but adds an additional requirement
- O – Obsolete; requirement from old standard changed or eliminated

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Working Table

F 5.2.4.4.1.5.k, 5.2.5	ISO/IEC 17025:2017	6.2.4 The management of the laboratory shall communicate to personnel their duties, responsibilities and authorities.
	EL-V1M2-ISO-2016-Rev2.1	5.2.4 The laboratory shall maintain current job descriptions for managerial, technical and key support personnel involved in tests and/or calibrations.
	EL-V1M2-ISO-2016-Rev2.1	5.2.4 NOTE: Job descriptions can be defined in many ways. As a minimum, the following should be defined: <ul style="list-style-type: none"> — the responsibilities with respect to performing tests and/or calibrations; — the responsibilities with respect to the planning of tests and/or calibrations and evaluation of results; — the responsibilities for reporting opinions and interpretations; — the responsibilities with respect to method modification and development and validation of new methods; — expertise and experience required; — qualifications and training programmes; — managerial duties.
	DoD/DOE QSM 5.3	5.2.4 DoD/DOE Requirement The following shall be implemented in addition to ISO Clause 5.2.4: The job description elements itemized in the note following ISO Clause 5.2.4 are minimum requirements.

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Working Table


F 5.2.4.5.2.5	ISO/IEC 17025:2017	6.2.5 The laboratory shall have procedure(s) and retain records for:
FS.2.2	ISO/IEC 17025:2017	6.2.5 a) determining the competence requirements;
N	ISO/IEC 17025:2017	6.2.5 b) selection of personnel;
FS.2.2, 5.2.5	ISO/IEC 17025:2017	6.2.5 c) training of personnel;
N	ISO/IEC 17025:2017	6.2.5 d) supervision of personnel;
	EL-V1M2-ISO-2016-Rev2.1	4.1.5.g) [The laboratory shall]... provide adequate supervision of testing and calibration staff, including trainees, by persons familiar with methods and procedures, purpose of each test and/or calibration, and with the assessment of the test or calibration results;
FS.2.5	ISO/IEC 17025:2017	6.2.5 e) authorization of personnel;
	EL-V1M2-ISO-2016-Rev2.1	4.1.5.j) appoint deputies for key managerial personnel (see Note); NOTE: Individuals may have more than one function and it may be impractical to appoint deputies for every function.

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Development of QSM 6.0




Second Cut


- Work through language that is functionally the same and language that has added requirements
 - Will “functionally the same” be good enough?
 - Are the added requirements necessary to produce good data?

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Development of QSM 6.0




- From here on out, the same tables were maintained so all changes could be recorded and tracked
- Third cut-Subgroup worked to evaluate whether
 - Additional language is beneficial
 - Language is properly located
 - Language accurately communicates the requirement

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Development of QSM 6.0




This Third cut resulted in


- A great deal of rewording
- Elimination of a significant number of paragraphs

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Development of QSM 6.0




Fourth Cut


- The last time through the Table, QAOS was reading for continuity and working to ensure
 - Requirements were clear-more rewording ensued
 - Requirements were properly located-more paragraphs were moved
 - Any additional duplication was removed
 - Internal references were eliminated or made generic

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Development of QSM 6.0




Other goals


- Standardize language: From ISO/IEC 17025-2017
- “shall” indicates a requirement;
- “should” indicates a recommendation;
- “may” indicates a permission;
- “can” indicates a possibility or a capability.

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Development of QSM 6.0



- More language issues
- Document vs. Record
 - ISO consistently discusses documenting instructions and maintain records of events
- Procedure vs. SOP
 - ISO uses “procedure” to mean a written set of instructions

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Development of QSM 6.0

- After fourth cut on the Table, the Draft Module 2 was generated and sent out for comment
- Comments were received (they were REALLY appreciated!) and adjudicated
- Comments from other Modules have required small changes to Module 2

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Development of QSM 6.0

The last step of the process was the application of “Global Edits”

- As QAOS went through the Standard, QAOS collected words and phrases that required standardization
- Each use of these terms was checked for compliance with the definitions

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Development of Other Modules

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Development of QSM 6.0

- Other technical modules (Mod 3-7)
 - Similar process, but made much easier by not needing to incorporate the ISO document
 - “Mash Tables” were prepared to compare versions
 - In general, the TNI 2016 language was used

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Development of QSM 6.0

Other technical modules, continued

- Some rearrangement of requirements within Module 4 was performed for clarity and consistency
- Edits were made for clarity
- Edits were made for consistent (global) language
- Edits were made where TNI requirements did not match QSM requirements

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Development of QSM 6.0


- The process was different for two of the modules
- Module 1 – Proficiency Testing
 - Significant additional requirements were added
- Module 8 – Industrial Hygiene Analyses
 - New Module-started as “in lieu of” requirements
 - Was written as an entire set of requirements

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Other Items to Note




- In the QSM 6.0, Definitions are considered requirements
 - They are assessable
- In practice, customer requirements supersede QSM requirements
 - However, they must come from the customer
 - Laboratories proposing changes to requirements shall use waiver system

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Other Items to Note




- To alleviate copyright issues, the QSM document must be used in conjunction with ISO/IEC 17025-2017
 - Module 2 contains the additional requirements
 - Where requirements are taken directly from the TNI 2016 Standard, the TNI reference is included
- Other modules follow the TNI structure, but the numbering as been changed
 - The leading “1” has been removed

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Example Text



The following shall be implemented in addition to ISO/IEC 17025:2017(E) Clause 7.4.3.


7.4.3.a The laboratory shall have a procedure for communicating to all affected laboratory personnel when samples that require non-routine analysis, additional sample preparation steps, or customer-required deviations are received. Records of these communications shall be maintained.

The following shall be implemented in addition to ISO/IEC 17025:2017(E) Section 7.4.


7.4.5 The laboratory shall have a documented system for uniquely identifying the samples to be tested to ensure that there can be no confusion regarding the identity of such samples at any time (i.e., a laboratory ID code). This system shall include identification for all samples, sub-samples, preservations, sample containers, tests, and subsequent extracts and/or digestates. (TNI 2016 V1M2 5.8.5.a)

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Conclusion




- Module 2 completely incorporates and follows the order of the updated ISO document
- QSM 6.0 will be a more clear, more concise document
- Requirements are more robust and more easily assessable
- Requirements will maintain or improve quality while adding some added flexibility

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Finishing up



- Questions?
- Note: Additional presentations are coming for all of the modules used routinely and some particular sections of those modules
- Questions may be deferred to those presentations

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MODULE 1 – PROFICIENCY TESTING UPDATES

John Gumper, ChemVal Consulting, Inc

1



Not just Module 1, but...

will also include Proficiency Testing (PT) information from Module 8

2



Introduction



- Module 1 of the QSM has been significantly rewritten to deal with gaps in the current program
 - Also to align with TNI to the degree possible
- Module 8 for Industrial Hygiene (IH) has a section for PT in the IH analyses
 - IH PT was written to mostly align with American Industrial Hygiene Association (AIHA) Proficiency Analytical Testing (PAT) requirements

3



Introduction



- General Requirements
 - Comparison to TNI
- Reporting Requirements
- Use of Accredited TNI PT Providers
- Use of other ISO/IEC 17043 Providers

4



Introduction



- Procedures when no Commercial PT studies are available
- PT Requirements of Module 8 – Industrial Hygiene

5



A Few Definitions



Field of Accreditation (FoA)


- Matrix, technology/method, and analyte combination for which the accreditation body offers accreditation

Field of Proficiency Testing (FoPT)


- Matrix, technology/method, analyte combination for which the composition, spike concentration ranges, and acceptance criteria have been established by TNI's Proficiency Testing Program Executive Committee

FoPTs can be found on the TNI website:
<https://nelac-institute.org/content/NEPT/fopt.php>

6




General Requirements




<p>TNI 2016</p> <ul style="list-style-type: none"> Based on FoPT No additional requirements for scope items with no FoPT 	<p>Both</p> <ul style="list-style-type: none"> Require participation in commercially available PT from TNI approved PTP for all FoPT on scope When a regulatory program has additional PT requirements not covered by this Standard, then the laboratory shall follow those requirements 	<p>QSM 6.0</p> <ul style="list-style-type: none"> Based on FoA If no TNI PTP, use ISO/IEC 17043-accredited PTP If no 17043 PTP, do internal precision and bias studies
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General Requirements




- The QSM needs to be a bit different because
 - TNI FoPT tables do not include some analytes important to the QSM community, for example:
 - 1,4-Dioxane
 - Perfluoroalkyl substances (PFAS)
 - Uranium isotopes
- So, the QSM provides additional requirements

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General Requirements




- Regardless of whether there is a TNI FoPT, the QSM requires laboratories to perform PT for each matrix, technology-method, analyte for which they seek accreditation
- OK, not every analyte-there are a couple of exceptions

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General Requirements




- For methods where the laboratory analyzes a large suite of compounds (e.g., 8260 or 8270 compounds), and all the compounds are not included in the PT Studies, the requirements for the additional compounds shall be met by the successful analysis of a FoPT study for that method, unless there are separate PT studies specifically for the analytes not included in the PT studies
- There are two scenarios in this paragraph

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General Requirements




- Scenario 1:** For methods where the laboratory analyzes a large suite of compounds (e.g., 8260 or 8270 compounds), and all the compounds are not included in the PT Studies, **the requirements for the additional compounds shall be met by the successful analysis of a FoPT study for that method**

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General Requirements



- Example**
 - Benzo(e)pyrene is not included on the FoPT table and would not typically be included in a proficiency test study, however there are several other benzo-pyrene isomers that are included and would give evidence of the laboratory's capability

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- Scenario 2: For methods where the laboratory analyzes a large suite of compounds (e.g., 8260 or 8270 compounds), and all the compounds are not included in the PT Studies, the requirements for the additional compounds shall be met by the successful analysis of a FoPT study for that method, **unless there are separate PT studies specifically for the analytes not included in the PT studies**

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General Requirements

- Example
 - 1,4-dioxane is not included in the FoPT tables and is not included in the multi-analyte suite offered by PTPs, but there are single analyte PTs available, thus the lab must participate in one of those
- Additionally, if the laboratory analyzes PFAS
 - Aqueous PT is an acceptable substitute for the AFFF matrix

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Reporting Requirements

The laboratory shall, on or before the study closing date

- Report the results using the PT Provider's (PTP) reporting format
- Direct the PTP to report the results directly to the laboratory's AB

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Reporting Requirements

- Results used for **initial accreditation** shall be sent directly from the PTP to the AB
 - Lab may have previous PT reports that are needed to demonstrate compliance for initial accreditation
- Results shall be reported so there is a specific match between the PT results and the FoA
- Results reported by technology (not allowed for drinking water) apply to all methods using that technology

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Reporting Requirements

NEW QSM REQUIREMENT [M1 4.3.5]

- If a laboratory chooses to analyze and report a single method to represent a technology, and multiple combinations of preparation/analytical methods are used for analysis of field samples, the laboratory shall follow a documented schedule and rotate the combinations used for analysis of field samples each PT study. Every combination shall be used a minimum of once every three years for each matrix.

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When a FoPT is Available

FoPTs include ranges of analysis, "Proficiency Testing Reporting Limits" (PTRLs), and the TNI Standard has all sorts of rules for analyzing and reporting results


- QSM quotes the TNI Standard at this point
- All the rules apply

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Use of Other 17043 Providers




- When there is no TNI FoPT table available for a FoA, but there is a United States of America (USA) or Canada-based ISO/IEC 17043 accredited PT provider for that FoA, the laboratory shall procure, analyze, and report the PT sample(s) in accordance with criteria established by the PT provider [M1 4.6]
- The FoA needs to be included in the PTP's accreditation to meet this requirement

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Use of Other 17043 Providers




- Only required to use those in USA and Canada
 - May use others, if appropriate and lab wishes
- Requirements for reporting, frequency and all other requirements of Module 1 apply
 - If PT is not available at required frequency, shall be annually
- Note: Many PTPs provide PT studies under their 17043 accreditation for FoAs not included in the FoPTs

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No Commercial PT Available




- [...in the USA or Canada, ISO/IEC 17043] [M1 4.7.3]
- Applies to chemical/radiochemical analyses
- A lab may use an ISO/IEC 17043-accredited provider from elsewhere
 - All the same QSM requirements apply
- A lab may use a non-accredited PT provider with permission of the lab's AB
 - All the same QSM requirements apply

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No Commercial PT Available




- **NEW REQUIREMENT!!**
- If no PT program is used, the laboratory shall determine Precision and Bias according to this Standard
 - Applies to Chemical and Radiochemical Testing Only
- The laboratory shall have procedures for performing these precision and bias studies [M1 4.7.3.b]

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Justification for the New Requirement




- Previous requirements were poorly defined
- Based on AB's policies
- Labs may demonstrate by participation in other quality control activities
 - Probably unevenly applied
 - Probably unevenly assessed

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Precision and Bias




- Labs shall submit list of parameters to AB for which no suitable PT is available (USA, Canada)
 - Each Analyte-Matrix-Method/Technology combination
 - DOE labs shall also submit this list to all affected customers

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Precision and Bias




- Studies shall be performed for each Analyte-Matrix-Method/Technology combination
- Studies performed twice per year
 - Subject to the same time constraints as PT studies
 - Samples processed through the entire measurement system
 - No fewer than eight quality control samples

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Precision and Bias




Sample concentrations shall be


- No greater than the concentration used for the LCS
- No less than the concentration used to verify the LOD
- John's note: Will work best with consistent concentration (not a requirement)

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Precision and Bias




Where standard solutions/low-level spiking solutions are not available and the laboratory uses a comparable compound for quality control (e.g., "cold" Selenium for Se-79), the laboratory shall collate the results of those analyses to meet this requirement [M1 4.7.3.e]

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Precision and Bias




Samples may be selected in the following ways:


- Eight or more replicates prepared and analyzed for the semiannual study
- The last eight quality control samples analyzed with routine samples
- Random selection of eight sample from those analyzed in the last six months, since the last study
 - Procedure shall define random selection algorithm

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Precision and Bias




- For the samples selected, calculate:
 - Average percent recovery
 - Percent relative standard deviation
- Compare to acceptance criteria

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Precision and Bias




- Acceptance criteria:
 - Use the criteria in the published method
 - If none, use the criteria from a similar method, or
 - Use laboratory-developed criteria

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Precision and Bias




- Precision and Accuracy results shall be reported to the laboratory's AB
- Results of the study shall be reported to any customer who requests them
- Unacceptable results require initiating lab's non-conforming work procedure in the same manner as those from an external PTP

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Other Items




- There are required time frames, numbers of PT required pre-accreditation, numbers of days between studies, etc.
- I'm not going into them-they haven't changed
 - Labs know them
 - ABs know them

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Other Items





Something to know

- In the version of Module 4 put out for comment, Solid and Chemical Material (SCM) PTs could be used to meet requirements for tissue PTs
- After comments from regulators, that was taken out of the Standard
 - Data from actual biological matrices is required

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Industrial Hygiene PTs


Module 8

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Industrial Hygiene PT




- Module 8 contains specific requirements for PT which supersede those in Module 1
 - Found in Section 4.0 of Module 8
- Requirements are based on AIHA requirements for laboratories
- There are 4 options for proficiency demonstration which are ordered by priority

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Industrial Hygiene PT – Four Tiers



External PT Provider Program	• 1 st Choice
Round Robin Program	• 2 nd Choice
Internal PT Program	• 3 rd Choice
Internal QC Program	• 4 th Choice

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Industrial Hygiene PT – Four Tiers

External PT Provider Program • 1st Choice

- External PT Program
 - Meet requirements of American Industrial Hygiene Association Laboratory Accreditation Program (AIHA LAP)
 - Use providers accredited to ISO/IEC 17043, if available
 - Use non-accredited providers with approval of AB
- When external PT is not available, the laboratory shall list all scope items affected and provide that list to its Accreditation Body

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Industrial Hygiene PT – Four Tiers

Round Robin Program • 2nd Choice

- Round Robin
 - Where available, participate in round-robin studies meeting the requirements of the AIHA LAP
- Laboratories shall have procedures for participation in round robins
 - Frequency
 - Acceptance criteria
 - Unacceptable results are non-conforming work

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Industrial Hygiene PT

Internal PT Program • 3rd Choice

- Internal PT Program
 - When no external PT and round robins are prohibited, proprietary or impractical
 - Compliant with AIHA LAP section on PT

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Industrial Hygiene PT

- Internal PT Program shall have a procedure addressing
 - Spiking procedures
 - Frequency
 - Responsibility for implementation
 - Statistical treatment of resultant data
 - Acceptance criteria
 - Actions to be taken in the event of an unacceptable result

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Industrial Hygiene PT

Internal QC Program • 4th Choice

- Internal Quality Control Program
 - When no external PT and round robins are prohibited, proprietary or impractical and internal PT program is impractical
 - Compliant with AIHA LAP section on PT
 - Approval from AB

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Industrial Hygiene PT

- Internal QC program requires a procedure
 - Schedule and frequency of evaluation
 - Identification of QC samples evaluated
 - Acceptance criteria
 - Actions to be taken in the event of an unacceptable result

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Conclusion



- Chemical testing and Industrial Hygiene testing PT programs have parallel but different requirements
- The biggest change in this version fo the QSM is the addition of the requirement to determine Precision and Bias for chemical and radiochemical analyses

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Thank you!



- Questions?

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