Welcome to the 2025 DoD Environmental Monitoring and Data Quality Workshop

Session II: EDQW Training

June 25, 2025

Data Review and Implementation Training

Phase Two: Implementation

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Goal of Phase Two

- Understand the Project Chemists Role in Data Review
  - Data Validation Report
  - Laboratory Data Package
- Walk through a practical 1633A scenario that illustrates:
  - Common issues flagged during validation
  - How chemists can collaborate with validators early to resolve discrepancies
  - Strategies for improving data defensibility

Understanding Data Review

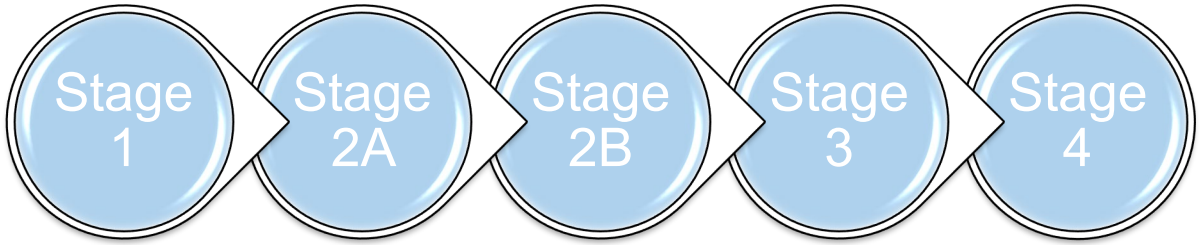
- Understanding the level of detail expected from the review.
- The importance of knowing the stage of data validation.
- Understanding the report to be included in the laboratory data package based on the review level.

Ideally these are well documented on QAPP WS #34, 35, and 36

QAPP WS #34, 35, 36

Understanding the Stage of Data Validation

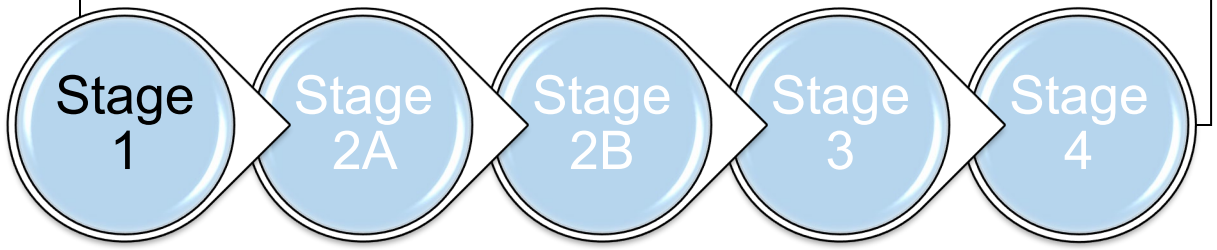
- Required for all stages:
  - Cover Sheet
  - Table of Contents
  - Laboratory Case Narrative
  - Sample Results Summary Form



Stage 1 Review

Overall Scheme DoD DVG

- Sample Summary Result Form
- Chain of Custody
- Sample Receipt (Checklist)
- Holding Time
- Field Quality Control (QC) Data



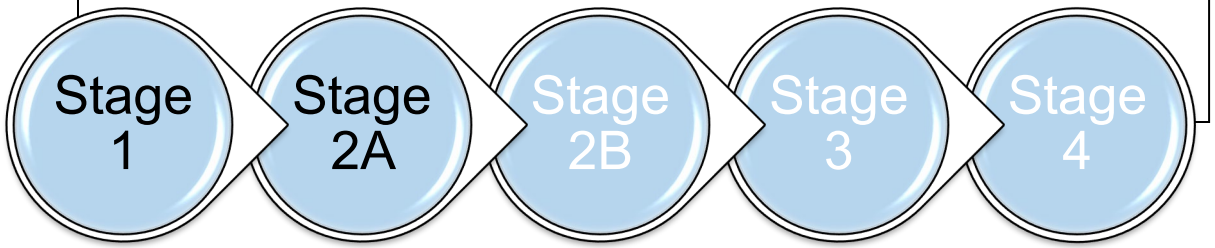
DoD DVG Module 6-PFAS

- Holding Time
  - EPA Method 1633 **Sample Collection, Preservation, Storage, and Holding Times** Section
  - EDQW Memo: Recommendation to Address Shorter Holding Times for Specific Per- and Polyfluoroalkyl substances (PFAS) When Using EPA Method 1633 for PFAS Investigations

Stage 2A Review

Overall Scheme DoD DVG

- Preparation Specific QC Data & Sample Specific Parameters Summaries:
  - Method Blanks (MB)
  - Laboratory Control Samples (LCS) and LCS Duplicates (LCSD)
  - Matrix Spikes (MS), MS Duplicates (MSD), and Matrix Duplicate (MD)
  - Sample Dilution/Reanalysis



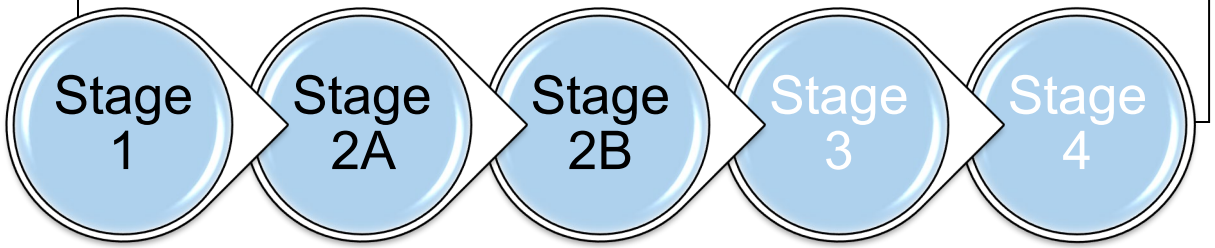
DoD DVG Module 6-PFAS

- QC Data Summaries:
  - Ion Abundance Ratio (IAR)
  - Extracted & Non-Extracted Internal Standard (EIS & NIS) Recovery
  - Retention Time
  - Low Level LCS (LLLCS)
  - Bile Salt Check
  - Qualitative Identification Standard

Stage 2B Review

Overall Scheme DoD DVG

- Instrument Specific QC Data:
  - Sequence & Preparation Logs
  - Instrument Performance/Sensitivity Checks (ISC)
  - Initial & Continued Calibration Summary
  - Internal Standard Summary



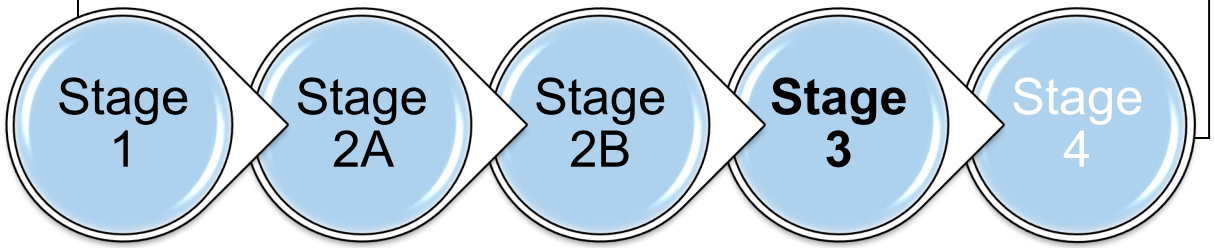
DoD DVG Module 6-PFAS

- Initial & Continued Calibration Summary
  - EIS Responses
  - Analyte and EIS Concentrations
  - Response Ratios (RRs) or Response Factors (RFs)
  - RR or RF Relative Standard Deviation or Relative Standard Error

Stage 3 **Recalculate/Re-quantify**

Overall Scheme DoD DVG

- Instrument Quantitation Forms (Raw Data)
  - Sample Results
  - Method & Instrument QC
- Standard Traceability



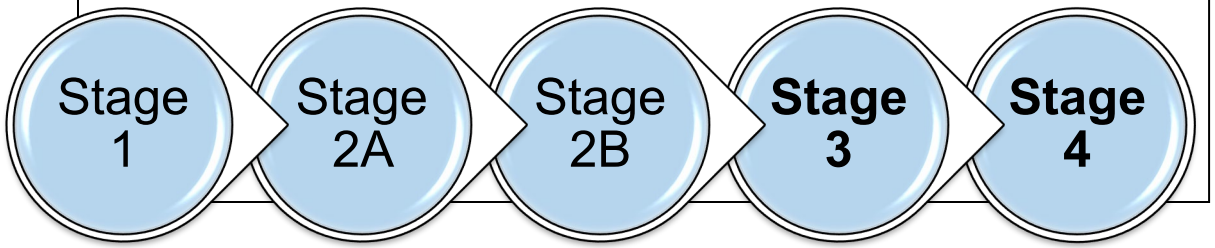
DoD DVG Module 6-PFAS

- Method & Instrument QC
  - EIS & NIS Recovery
  - Retention Time
  - LCD, LCSD, LLLCS Recovery
  - MS, MSD, MD
  - ISC
  - Calibration Verifications
  - RR and RF

Stage 4 **Qualitative** Review

Overall Scheme DoD DVG

- Data Manipulation
- Target Analyte Identification
- Manual Integration
- Signal-to-Noise (S/N)



DoD DVG Module 6-PFAS

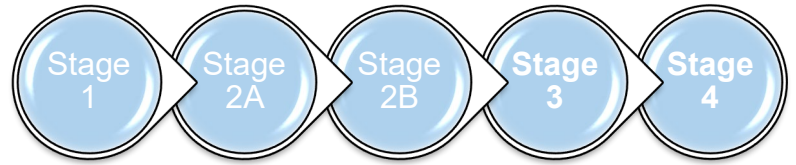
- Target Analyte Identification
  - EPA Method 1633 **Qualitative Peak Identification** Section

Slide Orientation

General

- Requirement
- Guidance

Scenario

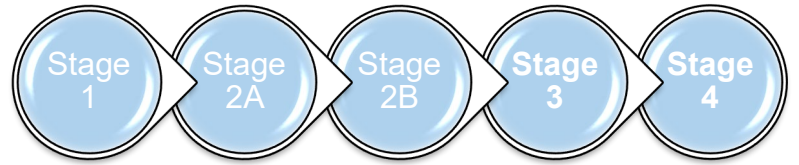


- PFAS analysis by Method 1633A*
  - Definitive Data
- Laboratory is accredited to DoD/DOE QSM v6.0

*The EDQW does not require laboratory accreditation to any particular version of EPA Method 1633.

  - Citation-EDQW Memo: EPA Method 1633 Clarification Update

What is the Level of Review?



General

- Laboratory Data Package support the intended stage of Data Validation?
- Data Validation Report (DVR) reflect intended review?
- QAPP WS 34, 35, 36 well defined?

Scenario

- Requested
  - Stage 4 DVR
    - 10% of the samples undergo Stage 4
  - Level 4 Laboratory Data Package

Poll Question

Does the statement provide enough information for you to understand the data validation process, or do you need more details?

“10% of samples undergo Stage 4 data validation”

A. Yes, enough information!

B. More details please!

Poll Question

Does the statement provide enough information for you to understand the process, or do you need more details?

"10% of samples per sample delivery group undergo Stage 4 data validation, the remaining samples undergo Stage 2B validation."

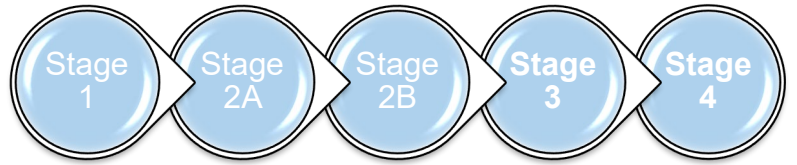
A. Yes, enough information!

B. More details please!

QAPP WS #34

- If you select B, please enter a suggestion into the chat!

Stage of Validation and the Package Level



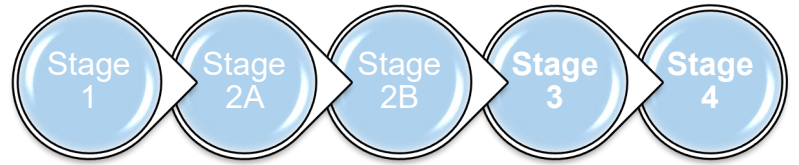
General

- Ensure the data validation report matches the required stage for the specific project.
- Verify that the laboratory data package aligns with the Stage of data validation.

Scenario

- Confirmed:
  - Stage 2B/4 DVR
    - 10% per the sample delivery group undergo a Stage 4 data validation
  - Level 4 Data Package

Where to Start the Review Process?



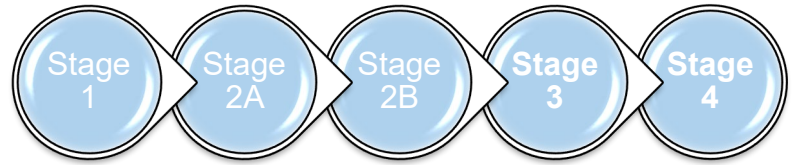
General

- Confirm DoD Environmental Laboratories Accreditation Program status
- DENIX
  - EDQW
    - Accredited Lab Search

Scenario

- Confirmed:
  - Good Standing!
- Cannot Confirm:
  - Check the Laboratory Data Package
  - Contact the Laboratory*
  - Contact EDQW*
- Unconfirmed:
  - Exclusion

Analyzing the Data Validation Report



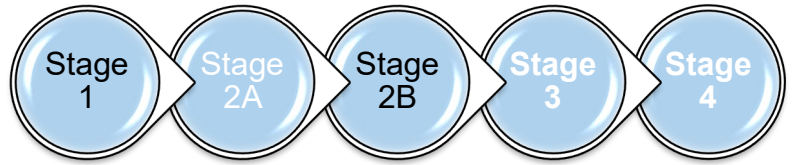
General

- Use the data validation report to guide your review of the laboratory data package.

Scenario

- MB had a detect for PFOS, all associated sample with PFOS detections were J-qualified.

Reviewing the Laboratory Data Package- HT



General

- Focus on verifying sample collect, receipt, preparation, and analysis dates and time
- Chain of Custody
  - Number of samples and collect date
  - Notes
- Sample Receipt Checklist
- Calculate Holding Time (HT)
- Look for discrepancies that need to be documented.

Scenario

- 7 groundwater & 2 soil samples
- Collected 3/10/2025
- Received 3/11/2025
- Prepared 4/5/2025
  - 26 days from collection – confirm
- Analyzed 4/15/2025
- Prep Note: The field sample collection bottle was overfilled during collection, removed portion to add spikes.

Poll Question

What do you think about the sample processing described in the prep note?

A. It makes sense; removing sample to add spikes is a valid step.

B. It seems unusual; removing sample could affect the results.

C. I'm not sure what the impact would be, but it sounds a bit off.

D. I didn't catch that detail—can you clarify?

Poll Question: Follow Up

The field sample collect bottle was overfilled during collection, removed portion to add spikes.

- 1633A Requirement:
  - **The requirement from the Planning Phase- Who remembers?**

QAPP WS #19 & 30

Poll Question: Follow Up

The field sample collect bottle was overfilled during collection, removed portion to add spikes.

- 1633A Requirement:
  - Aqueous: Do not fill the bottle past the shoulder
  - Solids: Fill no more than ¾ full
- Lab communication
- Field communication

QAPP WS #19 & 30

Poll Question

What do you think about the sample processing described in the prep note?

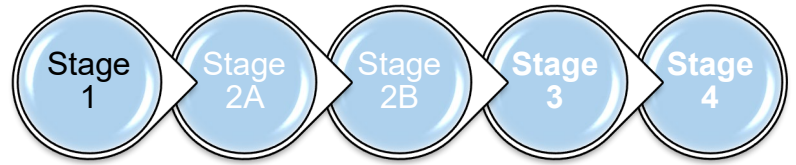
A. It makes sense; removing sample to add spikes is a valid step.

B. It seems unusual; removing sample could affect the results.

C. I'm not sure what the impact would be, but it sounds a bit off.

D. I didn't catch that detail—can you clarify?

The Case Narrative



General

- Look for anomalies identified by the laboratory and compare them with the data validation report.
- Ideally more detailed justification for anomalies.

Scenario

- Slides to follow and presented by Grace Nepomuceno.

Case Narrative - Laboratory

The low level LCS associated with Batch 1 for Sample XY failed high for almost all EIS and most target analytes failed low outside of acceptance criteria. Only the 125mL container remained for a re-extraction, resulting in elevated LOQs and DLs.

Case Narrative – Error

- Sample ID from case narrative: Sample XY
- Sample ID from Sample Summary Result Form: Sample 0XY

Case Narrative - Revisions

- When to Request a Revision:
  - Chemists must assess vague or erroneous case narratives.
    - Consider the nature of the project.
  - Revisions may not be necessary if the issue is minor and doesn't affect data usability.
    - If revisions are not needed, capturing the clarification in the DVR is another option to address minor issues without revising the Case Narrative.
  - For major concerns or unclear information:
    - Request clarification or correction from the laboratory.

Poll Question

- Sample ID from case narrative: Sample XY
- Sample ID from Sample Summary Report Form: Sample 0XY

Should a revision or clarification be requested from the laboratory?

A. Yes

B. No

Poll Question

- Sample ID from case narrative: Sample XY
- Sample ID from Sample Summary Report Form: Sample 0XY

Should a revision or clarification be requested from the laboratory?

A. Yes

B. No

Revised Case Narrative - Laboratory

The **Low Level LCS associated with Batch 1 for Sample 0XY** failed high for almost all EIS and most target analytes failed low outside of acceptance criteria. Only the 125mL container remained for a re-extraction, resulting in elevated LOQs and DLs.

Vagueness

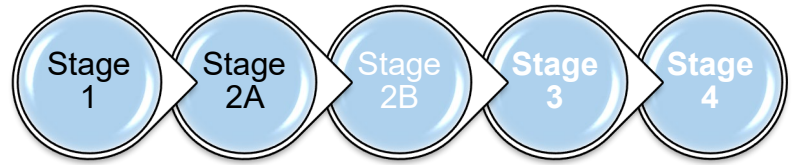
- Low Level LCS is a batch QC samples!
- What about the other samples in Batch 1?

- Big Picture Question- revise the laboratory case narrative or address in the DVR?

Case Narrative- DVR

Batch 1, Low Level LCS: nearly all EIS recoveries were greater than their respective Upper Control Limits, and nearly all spike recoveries were less than their respective Lower Control Limits. All samples associated with this batch were re-extracted, and results reported from the re-extract **Batch 2.** However, Sample 0XY was re-extracted at a lesser volume due to insufficient remaining volume, resulting in elevated reporting limits. This did not affect the usability of the results as the analytes of concern were not reported as non-detect at a reporting limit greater than the lower screening limit of 35 ng/L.

2A Summary Reports



General

- Reviewing detects, percent recovery, and relative percent difference, dilutions or reanalysis, IAR, Bile Salt Check, and Qualitative Identification Standard.

Scenario

- Few target analytes had laboratory qualifiers on the IAR Summary Sheet*
- Bile Salt Check
- Qualitative ID Standard

*Not always included, may only be included in the raw data.

Bile Salt Retention Time

The laboratory did not meet the requirement for bile salts to elute at least one minute from the PFOS window (including all its isomers).

- EPA Method 1633A Clause 10.2.2.5
- This is a critical issue as it could impact the method's accuracy and precision.

Required Bile Salts

Did not include all the required bile salts as specified by the method when using a mobile phase other than acetonitrile.

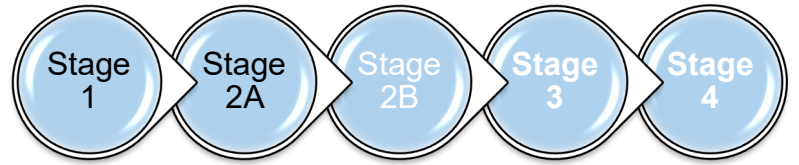
- EPA Method 1633A Clause 7.5
- Evaluate potential interferences-accuracy and precision.

Bile Salt Technical Justification

No technical justification for these deviations, and such deviations are prohibited because they compromise the precision and accuracy of the method.

- EPA Method 1633A Section 9.1.2.2
- Only modifications that meet or improve the quality of the data are allowed according to the method's standards and shall be documented.

Bile Salt Check: Variations in Laboratory Approaches



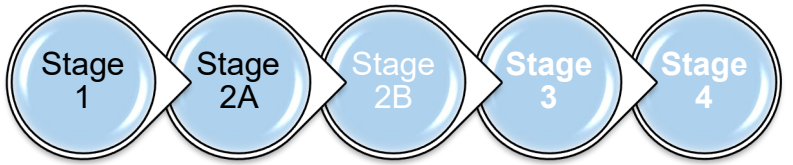
- Laboratories may perform Bile Salt Checks using slightly different methods and may label/name them differently.
- **Recommendation-**If the Bile Salt Check is unclear:
  - Review the laboratory's SOP for clarification.
  - Contact the laboratory (following proper channels) to ensure the analysis method is understood and aligned with project requirements*.

Review Lab SOP & QAPP WS # 24

* Ideally, addressed during the planning phase—another reason to thoroughly review the laboratory's procedure during QAPP development.

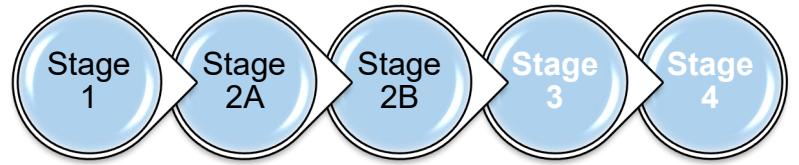
Qualitative Identification Standard

QAPP WS # 24



- Daily Check when standard become available
  - At the time of this writing, the DoD EDQW has not identified any required qualitative identification standards.
  - DoD EDQW is tracking availability

2B Summary Reports



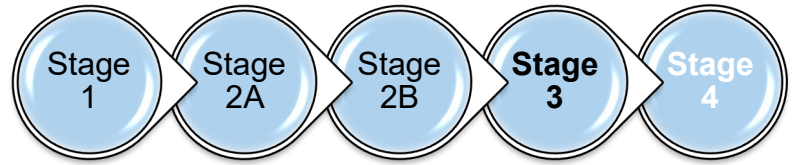
General

- Sequence and Preparation Logs
- Initial Calibration
- Continued Calibration
- Sensitivity Check
- Instrument Blank

Scenario

- New EDQW Memo: EPA Method 1633 Sequence Requirements
  - –June 17, 2025

Stage 3 **Recalculate/Re-quantify**



General

- Recalculate the initial calibration, and instrument and method QC elements
- Understand how the instrument behaves and how the peaks are integrated.
- Standard Traceability

Scenario

- Evaluating the Initial Calibration Data

Evaluating the Initial Calibration

- Recalculate initial calibration independently
- Recognize how EIS and NIS fit into calculations
  - EIS is equally as important as the integration of the target analyte
- Check standard traceability and response patterns

Validator Approach vs. Chemist Approach

- Validators may review only one target analyte
  - Defined in the QAPP?
- Contractor and **Government**-Project Chemists should review all target analytes chromatograms

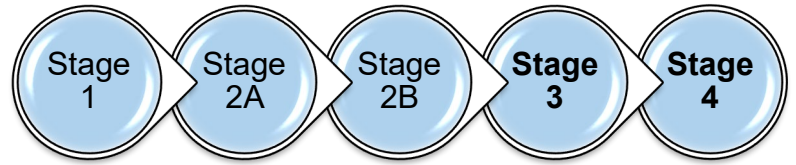
Benefits of Full Chromatogram Review

- Understand laboratory's integration behavior
- Spot trends in manual integrations
- Predict possible issues in field sample integration
- Strengthen defensibility of final data package

Comparing Calibration to Field Samples

- Examine lowest standard response areas
- Consider integration flags:
  - Manual integrations
  - Signal-to-Noise warnings
  - Abundance ratio failures
- Compare to field sample responses *Stage 4*

Stage 4 Raw Data



General

- Target Compound Identification
  - Peak Integration
  - Manual Integration
  - S/N
  - IAR

Scenario

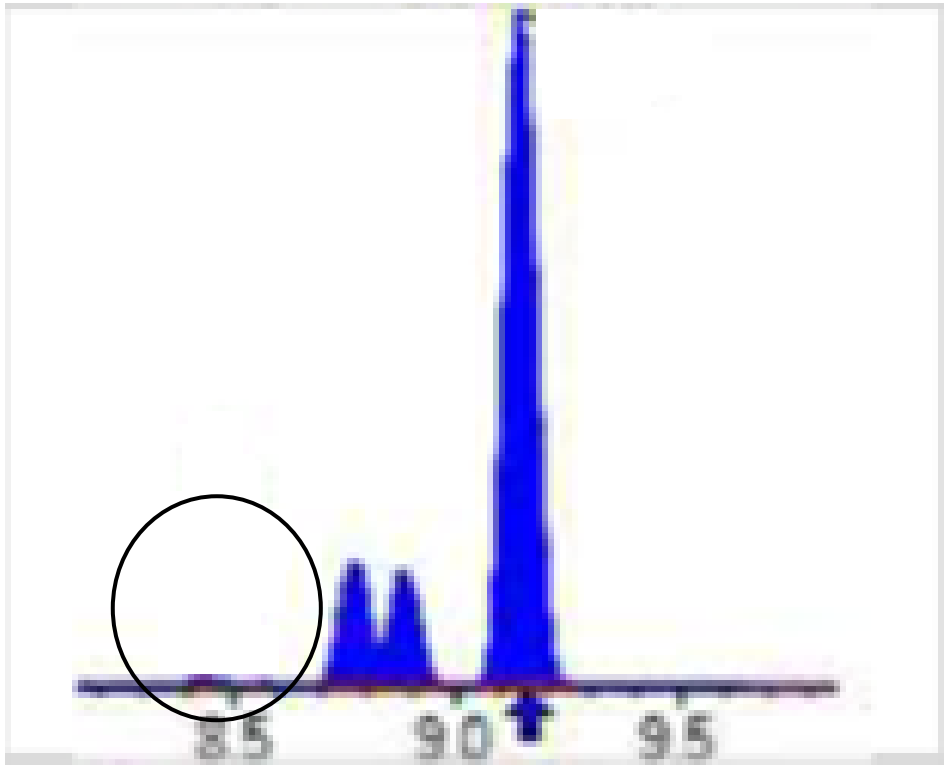
- Peak Identification

Instrument QC Informs Field Sample Review

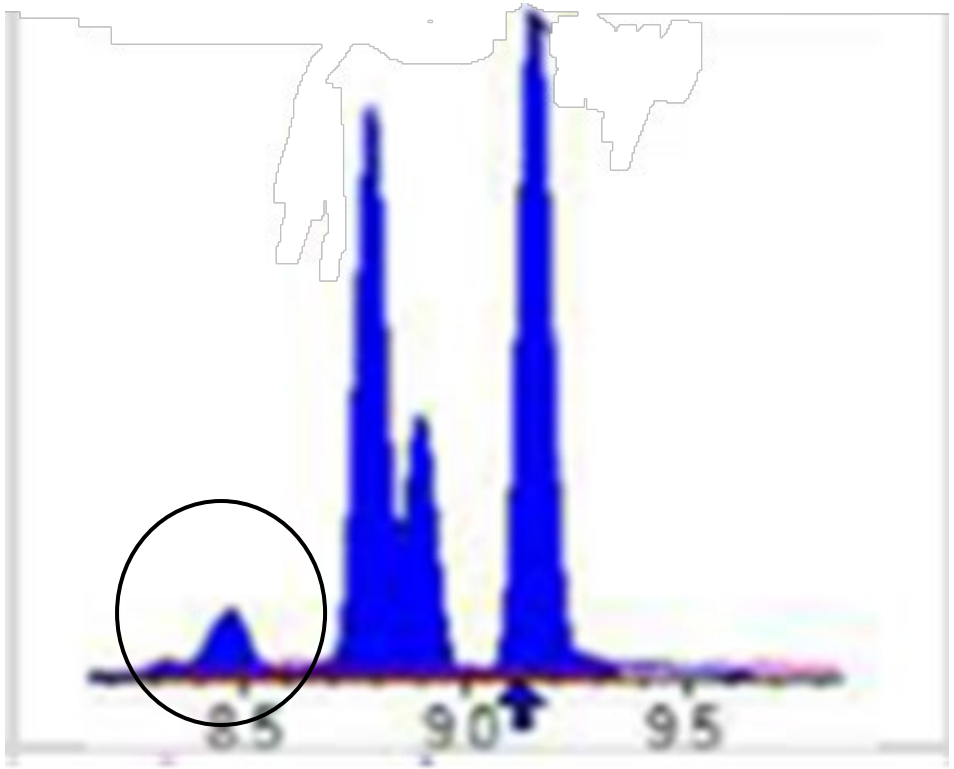
- Integration techniques used for QC must match field samples
- Any deviation should raise concerns
- Key Question: Is the field data being treated differently?

Example: Calibration vs. Field Sample Peaks

Mid-Level Calibration Standard

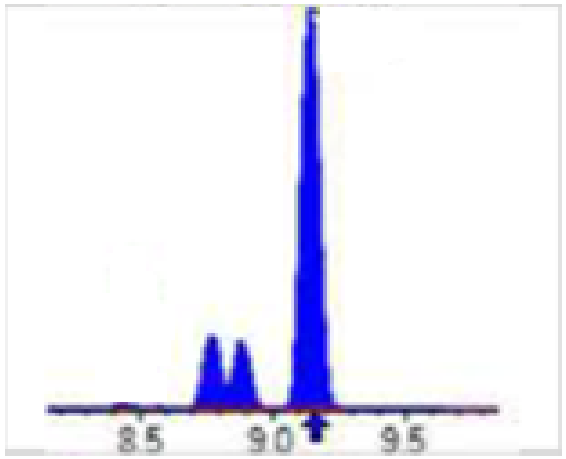


Field Sample- Similar Concentration

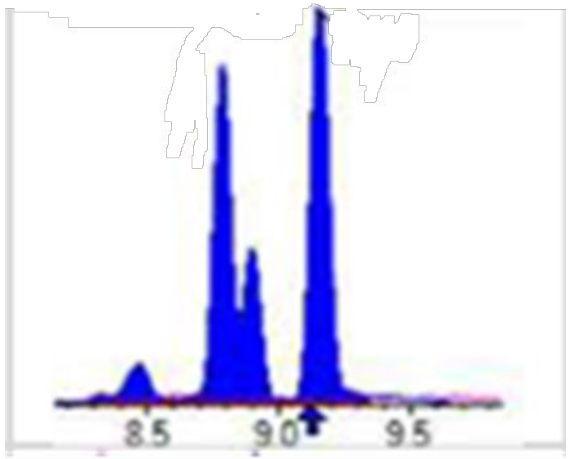


Poll Question

Mid-Level Calibration Standard



Field Sample- Similar Concentration



Does it appear the Field Sample is being treated the same as the Calibration Standard?

A. Yes

B. No

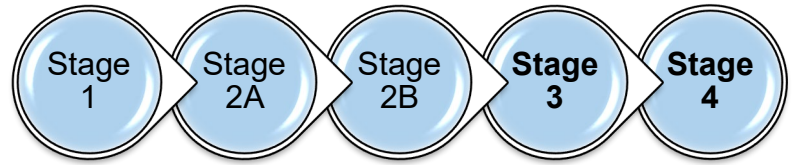
Poll Question Follow Up

Notice I said “appear” it is important to use caution and ask for clarification when a concern arises.

Request that the laboratory confirm whether the integration of the field samples aligns with their established procedures.

If any discrepancies are found, request any generated corrective action reports be provided.

Raw Field and QC Sample Data



General

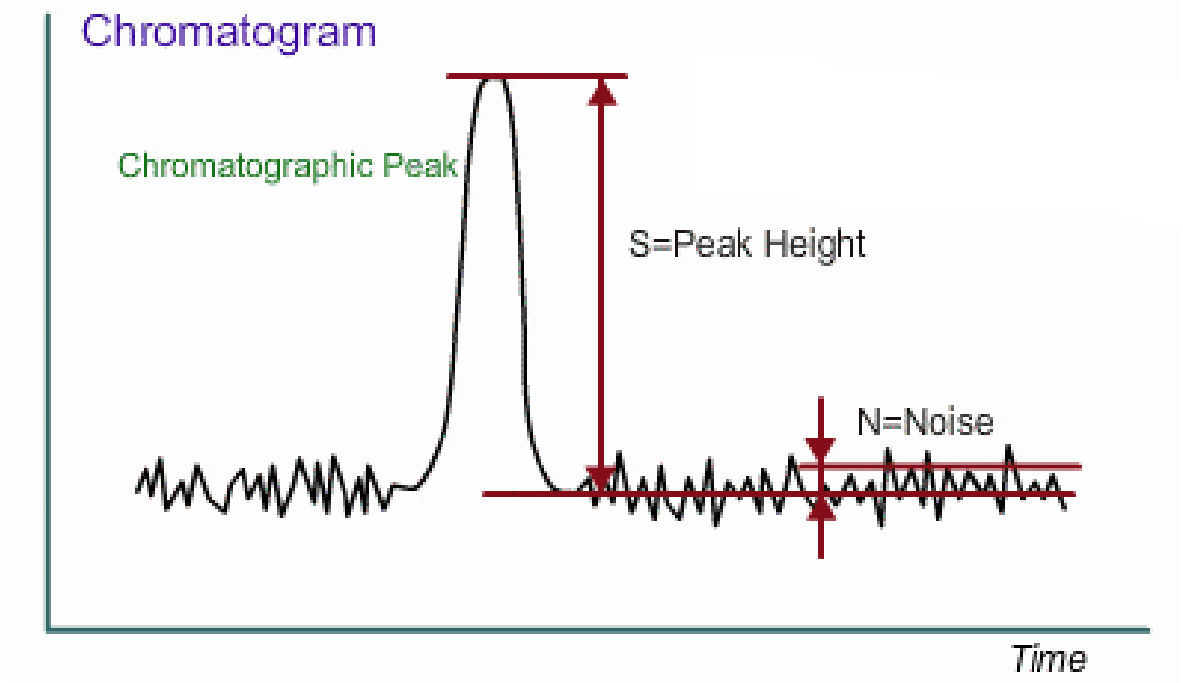
- Target Compound Identification
  - Peak Integration
  - Manual Integration
  - S/N
  - IAR

Scenario

- S/N
- IAR

S/N Ratio Fundamentals

- The signal represents the detected chromatographic peak, while the noise represents random fluctuations in the baseline. The ratio is a measure of how well the peak stands out from the background noise.



$$S/N = \frac{Peak\ Height}{Noise\ Height}$$

- Automatically calculated by instrument software

S/N Ratio

- A high S/N - peaks are well separated from noise, identification clear and accurate.
- A low S/N - peaks are harder to discern, leading to potential errors in quantification.

1633A S/N Ratio Criteria

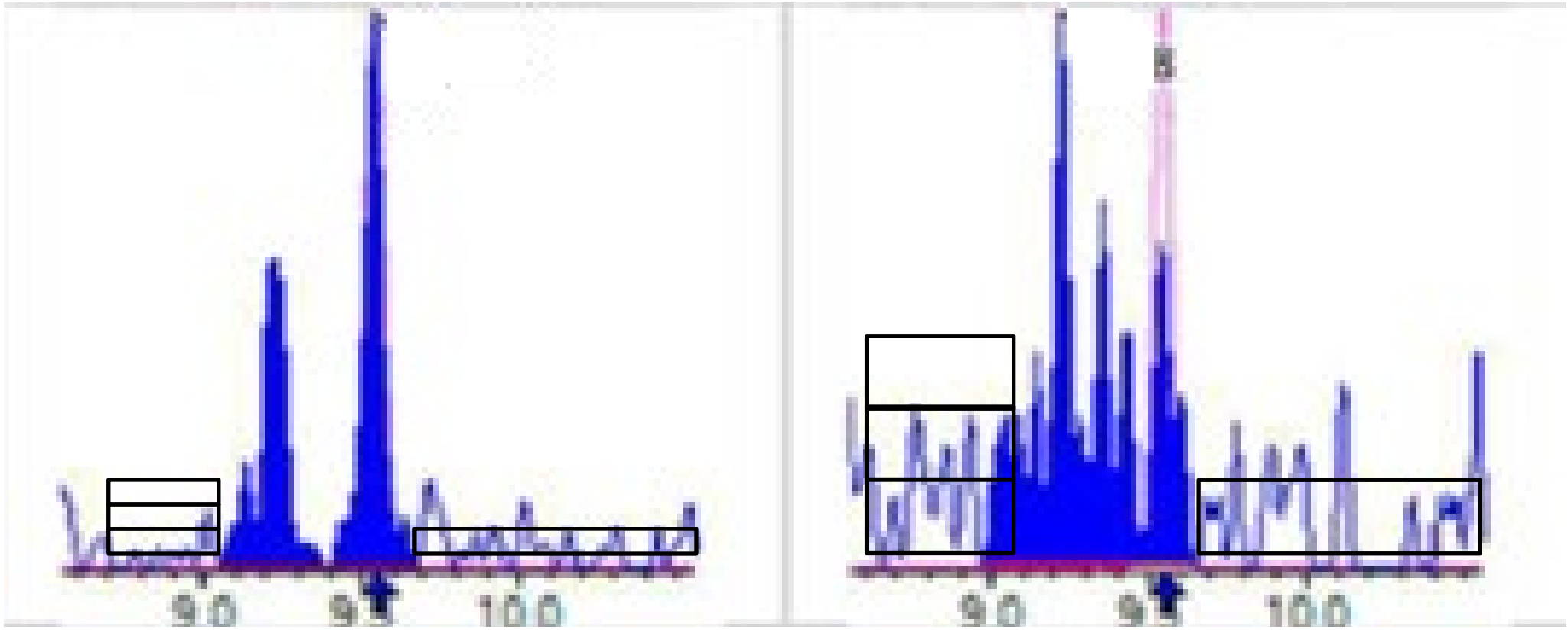
EPA Method 1633A, Section 15, Clause 15.1.1

"*For target analytes or EIS compounds to be identified, peak responses of the quantitation and confirmation ions must be at least three times the background noise level* ***(S/N 3:1).***

The quantitation ion must have a ***S/N ≥ 10:1*** *if there is no confirmation ion….*

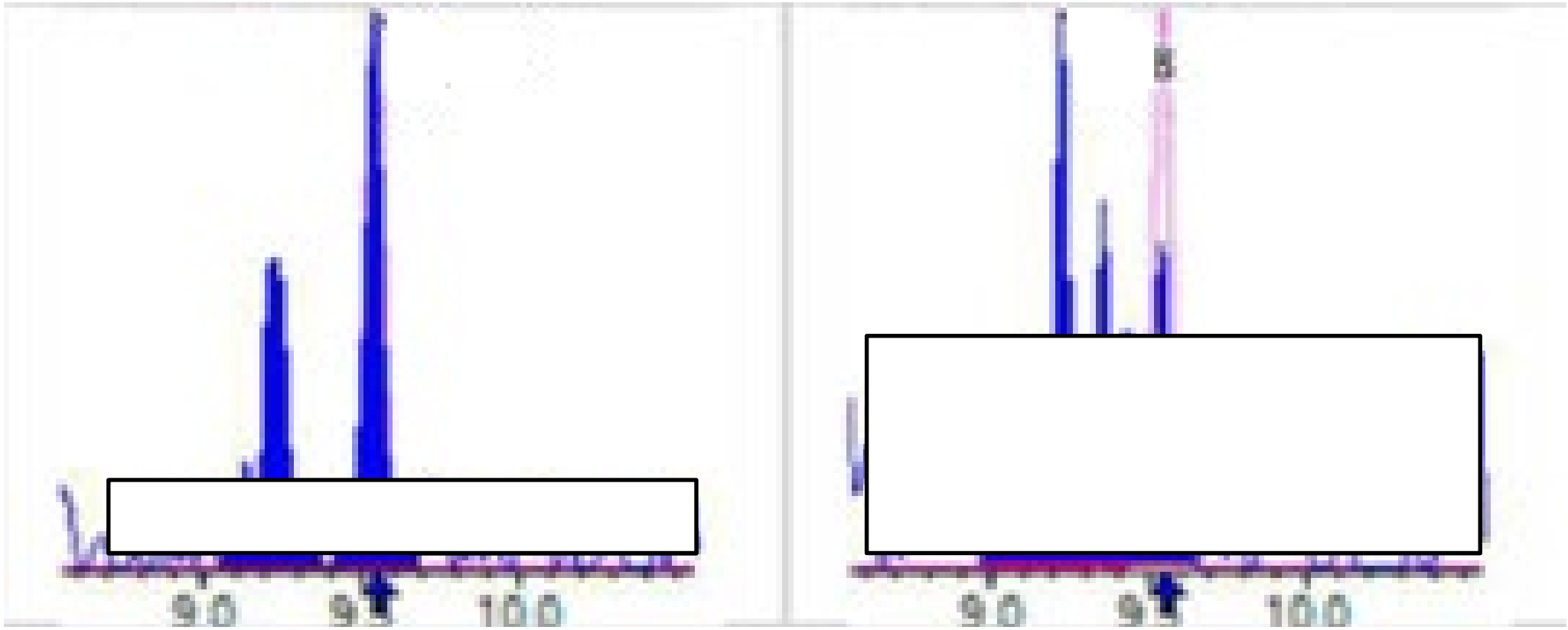
If the S/N ratio is not met but the background is low, then the analyte is to be considered a non-detect."

Visualize Representation S/N



Example Chromatogram 1: Showing Quantitation Ion (Left) and Confirmation Ion (Right) Traces

Visualize Representation S/N



Example Chromatogram 1: Showing Quantitation Ion (Left) and Confirmation Ion (Right) Traces

Ion Abundance Ratio Criteria

EPA Method 1633A, Section 15, Clause 15.1.3

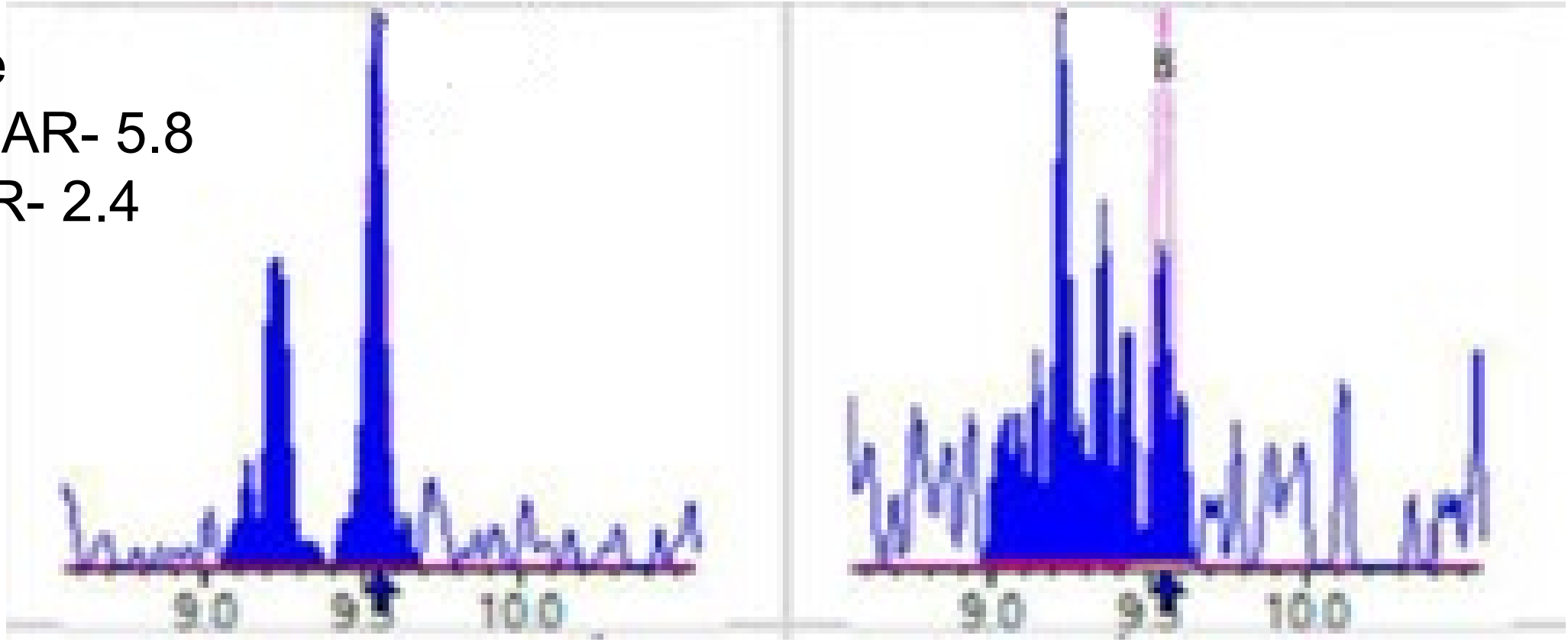
- Project-Specific OR

QAPP WS # 24

- ± 50% of the IAR observed in the mid-point initial calibration standard
  - Above and Below the LOQ

Ion Abundance Ratio Qualifier

IAR Failure
Observed IAR- 5.8
Sample IAR- 2.4
IAR= 41%



Example Chromatogram 1: Showing Quantitation Ion (Left) and Confirmation Ion (Right) Traces

Qualitative Peak Identification

EPA Method 1633A, Section 15, Clause 15.1.4

- Samples shall meet criteria of 15.1.1-15.1.4
  - Field Samples: expect qualification
  - QC or Standards: laboratory shall correct the issue

Poll Question

Example Chromatogram 1- failed IAR criteria, and the visual representation of the S/N has risen concerns. Should I expect my sample to be qualified?

A. Yes

B. No

Poll Question

Example Chromatogram 1- failed IAR criteria, and the visual representation of the S/N has risen concerns. Should I expect my sample to be qualified?

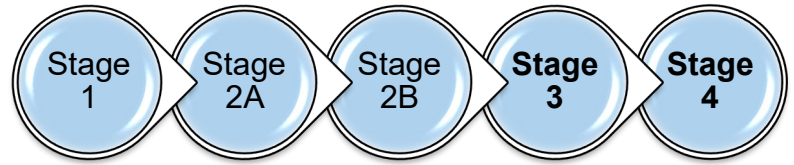
A. Yes

B. No

Trick Question

I never clarified if the Example Chromatogram 1 was a field sample or QC sample!

Raw Field and QC Sample Data



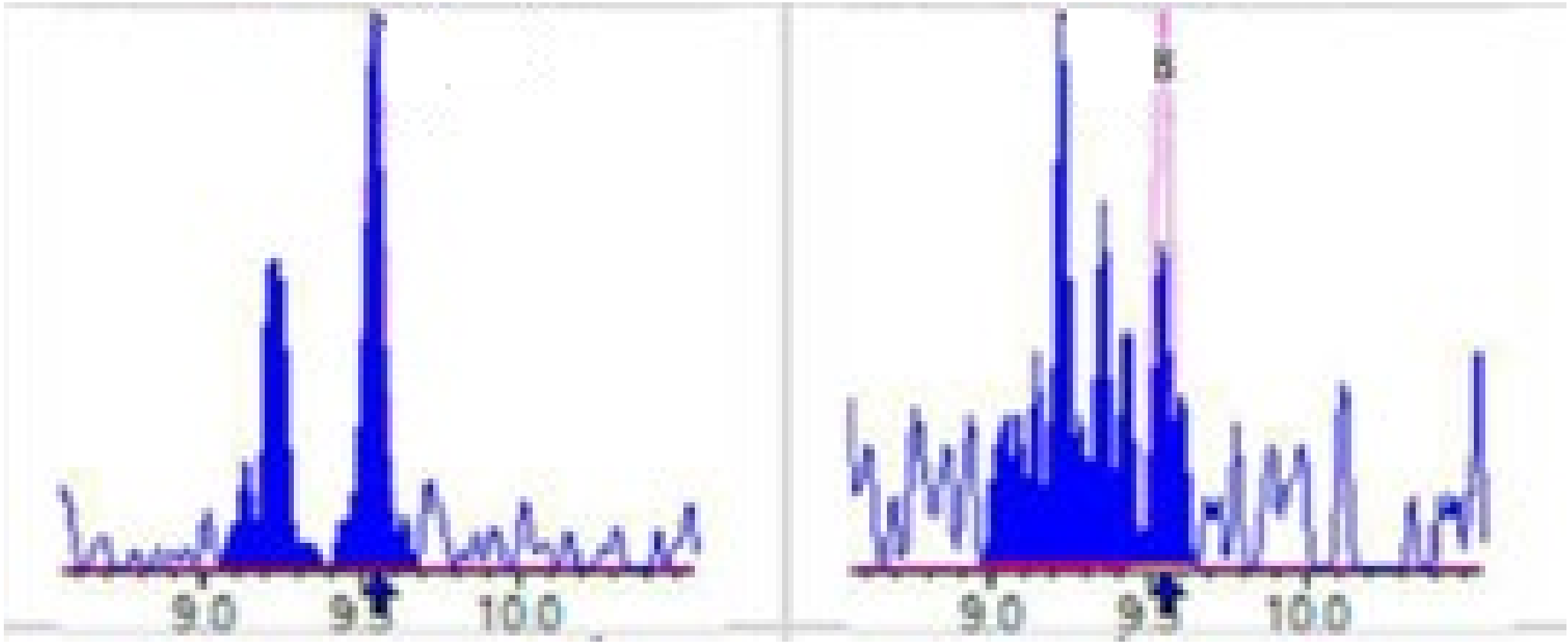
General

- Target Compound Identification
  - Peak Integration
  - Manual Integration
  - Signal to Noise
  - Ion Abundance Ratio

Scenario

- MB had a detect for PFOS, all associated sample with PFOS detections were J-qualified.

Method Blank



Example Chromatogram 1: Showing Quantitation Ion (Left) and Confirmation Ion (Right) Traces

Poll Question

Example Chromatogram 1- failed IAR criteria, and the visual representation of the S/N has risen concerns. Should I expect to my sample to be qualified?

A. Yes

B. No, because this is a QC sample and per EPA Method 1633A Clause 15.1.4 stated

"If the criteria listed above are not met for the standards, the laboratory must stop analysis of samples and correct the issue."

Data Quality

- Project-Specific consideration
  - How often was the issue observed?
    - Was it isolated or part of a broader pattern?
  - Is the analyte affected a target of interest, or less critical?
  - Were J qualifiers or other qualifiers already applied?
  - Did the deviation impact the key data points used in risk or decision making?
    - What is the end use of the data?

Final Steps in Data Review

- Follow a systematic approach for data validation.
- Verify and Cross-Check
- Track Discrepancies
- Review Everything

Training Session Conclusion

Proper Planning Sets the Foundation for Effective Implementation

- Communication is a critical part of the technical process.
- Document your decisions clearly and consistently.
- Clarity upfront reduces downstream workload
- Get the right people involved early.

Resources Phase 1 & 2

- DENIX- EDQW Home Page https://www.denix.osd.mil/edqw/
  - What's New-Updated Frequently!
- DENIX- EDQW Accreditation Page
  - DoD ELAP Fact Sheet- https://www.denix.osd.mil/edqw/featured-content/documents/dod-elap-fact-sheet/
- DENIX- EDQW Quality System Manuals Page
  - QSM Version 6.0- https://www.denix.osd.mil/edqw/denix-files/sites/43/2024/01/QSM-Version-6.0-FINAL-Dec-13-2023.pdf
- DENIX- EDQW Data Validation Guidelines Page
  - Module 6 Data Validation Guidelines- https://www.denix.osd.mil/edqw/denix-files/sites/43/2023/02/Module-6-Data-Validation-Guidelines-1633-PFAS-Final-1.pdf
    - Update under revision as of June 25, 2025

Resources Phase 1 & 2

- DENIX- EDQW Outreach and Guidance
  - Find EM/DQ Workshop Slides- https://www.denix.osd.mil/edqw/outreach/
  - EPA Method 1633 Clarification Update
  - Recommendation to Address Shorter Holding Times for Specific Per- and Polyfluoroalkyl substances (PFAS) When Using EPA Method 1633 for PFAS Investigations
  - EPA Method 1633 Sample Volume Modifications
  - EPA Method 1633 Sequence Requirements as of July 17, 2025
- EPA Method 1633
  - https://www.epa.gov/cwa-methods/cwa-analytical-methods-and-polyfluorinated-alkyl-substances-pfas
- Uniform Federal Policy for Quality Assurance Project Plans - Training Materials
  - https://www.epa.gov/fedfac/uniform-federal-policy-quality-assurance-project-plans-training-materials
- Optimized Uniform Federal Policy for Quality Assurance Project Plans Worksheets
  - https://www.epa.gov/fedfac/optimized-uniform-federal-policy-quality-assurance-project-plans-worksheets

Resources Phase 1 & 2

- Part 188 of Title 32-ELAP Requirement
  - https://www.ecfr.gov/current/title-32/subtitle-A/chapter-I/subchapter-L/part-188
- DoDI 4715.15
  - https://www.denix.osd.mil/international/policy/dodi/
- DoD PFAS Task Force Home Page https://www.acq.osd.mil/eie/eer/ecc/pfas/tf/policies.html
- ASD(E&IE) Memos
  - Establishing a Consistent Methodology for the Analysis of Per- and Polyfluoroalkyl Substances in Matrices Other than Drinking Water
    - Great to support screening data concept- “Screening samples to determine the presence or magnitude of PFAS concentration, but not to confirm absence”.
    - Recommends using a DoD ELAP accredited laboratory for screening methods.
  - Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program
    - Great to support considering additional target PFAS analytes not included in EPA Method 1633 with project-specific considerations.