QSM V6.0: MODULE 2 – QUALITY SYSTEM GENERAL REQUIREMENTS: IMPLEMENTATION AND UPDATES

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QSM 6.0 Goals

What, why and how the team approached this revision

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QSM 6.0 Goals

- Primary
  - –Align language with ISO/IEC 17025:2017 and
  - –Where appropriate, TNI 2016
- Additional
  - –Clarify difficult to interpret language
  - –Allow laboratories some additional flexibility in how they achieve compliance

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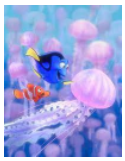
QSM 6.0 Goals

- Additional
  - –Eliminate duplicative requirements, internal references, unnecessary guidance, and "squishy" clauses
  - –Maintain or improve data quality and defensibility



Bad Squishy! Shoo, shoo, shoo!
Bad Squishy! Shoo, get away!

4

Significant, but Superficial Changes

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QSM 6.0 Superficial Changes

- Reorganization of requirements to fit the ISO/IEC 17025:2017 order of requirements
  - –"The last version of ISO/IEC 17025 was published in 2005 and, since then, market conditions and technology have changed. The new version covers technical changes, vocabulary and developments in IT techniques. It also takes into consideration the latest version of ISO 9001." (https://www.iso.org/ISO-IEC-17025-testing-and-calibration-laboratories.html)

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Reorganization of Module 2

QSM 5.4

4.0 MANAGEMENT REQUIREMENTS

- 4.1 Organization
- 4.2 Management
- 4.3 Document Control
- 4.4 Review of Requests, Tenders and Contracts
- 4.5 Subcontracting of Environmental Tests
- 4.6 Purchasing Services and Supplies
- 4.7 Service to the Client
- 4.8 Complaints

QSM 6.0

- 4.0 General Requirements
  - 4.1 Impartiality
  - 4.2 Confidentiality
- 5.0 Structural Requirements
- 6.0 Resource Requirements
  - 6.1 General
  - 6.2 Personnel
  - 6.3 Facilities and Environmental Conditions
  - 6.4 Equipment

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Reorganization of Module 2

QSM 5.4

- 4.9 Control of Nonconforming Environmental Testing Work
- 4.10 Improvement
- 4.11 Corrective Action
- 4.12 Preventive Action
- 4.13 Control of Records
- 4.14 Internal Audits
- 4.15 Management Reviews
- 4.16 Data Integrity Investigations

QSM 6.0

- 6.5 Metrological Traceability
- 6.6 Externally Provided Products and Services
- 7.0 Process Requirements
  - 7.1 Review of Requests, Tenders and Contracts
  - 7.2 Selection, Verification and Validation of Methods
  - 7.3 Sampling
  - 7.4 Handling of Test or Calibration Items

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Reorganization of Module 2

QSM 5.4

5.0 TECHNICAL REQUIREMENTS

- 5.1 General
- 5.2 Personnel
- 5.3 Accommodation and Environmental Conditions
- 5.4 Environmental Methods and Method Validation
- 5.5 Calibration Requirements
- 5.6 Measurement Traceability
- 5.7 Collection of Samples

QSM 6.0

- 7.5 Technical Records
- 7.6 Evaluation of Measurement Uncertainty
- 7.7 Ensuring the Validity of Results
- 7.8 Reporting of Results
- 7.9 Complaints
- 7.10 Nonconforming Work
- 7.11 Control of Data and Information Management

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Reorganization of Module 2

QSM 5.4

- 5.8 Handling Samples and Test Items
- 5.9 Quality Assurance (QA) of Environmental Testing
- 5.10 Reporting the Results
- 6.0 HAZARDOUS AND RADIOACTIVE MATERIALS MANAGEMENT AND HEALTH AND SAFETY PRACTICES (Section 6: DOE Only)

QSM 6.0

- 8.0 Management System Requirements
  - 8.1 Options
  - 8.2 Management System Documentation
  - 8.3 Control of Management System Documents
  - 8.4 Control of Records
  - 8.5 Actions to Address Risks and Opportunities
  - 8.6 Improvement
  - 8.7 Corrective Actions

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Reorganization of Module 2

QSM 5.4

QSM 6.0

- 8.8 Internal Audits
- 8.9 Management Reviews
- 9.0 (DOE-Only Requirement) Hazardous and Radioactive Materials Management and Health and Safety Practices

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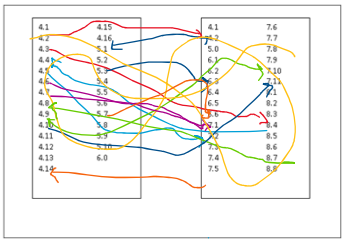
Gaaahhhhh!



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Significant Changes

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ISO/IEC 17025:2017 Changes

- Risk-based approach
- More latitude for how laboratories address quality system requirements
- Focus on outcomes rather than prescriptive requirements

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Module 2 Changes – 8.5 Risks and Opportunities

- This represents a very significant change in paradigm for the QSM
  - –From “we know the problems that occur in laboratories, and we’ll tell you how to avoid them”
  - –To “laboratories know better than we do what their customers need, how the laboratory may be challenged to meet the need, and what to do to fix it”

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Module 2 Changes – 8.5 Risks and Opportunities

- ISO 17025:2017 removed all language regarding “Preventive Actions”, but replaced it with requirements for consideration of risks and opportunities, which is structured to accomplish the same goals

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Module 2 Changes – 8.5 Risks and Opportunities

- In addition to the general requirements to consider risks and opportunities, QSM 6.0 contains a substantial list of specific topics to be considered from a risk standpoint annually to
  - –Ensure management system achieves its intended results
  - –Prevent, or reduce, undesired impacts and failures
  - –Achieve improvement

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Module 2 Changes – 8.5 Risks and Opportunities

- Additional requirements for risk include
  - –Identified risks and any mitigation plans shall be reviewed annually and updated as applicable
  - –Records of the annual review of risks and mitigation plans shall be maintained
  - –A list of activities for which laboratories are required to consider and address their risks

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Module 2 Changes – 8.5 Risks and Opportunities

The laboratory shall consider and plan mitigation for the risks and opportunities associated with the following laboratory activities:

- New method modifications/ in-house methods
- Externally provided products and services (including subcontract labs)
- Likely contaminants
- Minimum qualifications for personnel
- Designation/authorization of alternate personnel for key roles
- Use of electronic signatures

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Module 2 Changes – 8.5 Risks and Opportunities

- Determining version of methods to best suit customer needs
- Determining whether LOD/LOQ verifications will be performed quarterly or with each batch
- Metrological traceability procedures
- Determining acceptance criteria for auxiliary equipment verification and calibration
- Determining when correction factors are appropriate and how they are applied when they differ across a range of values

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Module 2 Changes – 8.5 Risks and Opportunities

- Determining frequency and content of technical reviews
- Determining frequency and content of quality record reviews to ensure data integrity
- Determining need for additional procedures not already required for accreditation

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Module 2 Changes – 4.1 Data Integrity

- Changed “Data Integrity” to “a documented program to detect and deter inappropriate or prohibited actions”
- Clarified language required for the program
- Listed prohibited actions

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Module 2 Changes – 5.2 Technical and Quality Manager

- 17025:2017 eliminated the requirement for a named Technical Manager and Quality Manager

Whaaaat??

No Way!

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Module 2 Changes – 5.2 Technical and Quality Manager

- Instead, it requires laboratory to:
  - Identify management that has overall responsibility for the laboratory
  - Specify the responsibility, authority and interrelationship of personnel who manage, perform or verify work
  - Have personnel with the authority and resources needed for implementation, maintenance and improvement of the management system

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Module 2 Changes – 5.2 Technical and Quality Manager

- Department of Defense embraced allowing laboratories to identify and authorize individuals to manage various portions of the quality system without requiring specific individuals to be named to particular roles
- Therefore, there are no additional 5.2 requirements that apply to DoD. The DoD follows the ISO language
- Department of Energy chose to maintain the named roles but rewrote duties and responsibilities to be more directly applicable to laboratories doing DOE work
- Therefore, the additional 5.2 requirements in Module 2 are DOE only

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Module 2 Changes – 5.2 Technical and Quality Manager

- **DOE-Only Requirement**- Technical Manager(s) duties and responsibilities shall include:
  - Ensuring the quality of data, review of QA and QC records, reviewing data packages, authorizing reports
  - Defining required qualifications and skills for all personnel
  - Oversight of demonstrations of capability and training for technical staff
  - Ensuring adequate supervision
  - Appointing someone to perform these functions when absent

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Module 2 Changes – 5.2 Technical and Quality Manager

- **DOE-Only Requirement**– Requirements for Quality Manager
  - Ensure that the management system related to quality is implemented
  - Have direct access to management
  - Be focal point for QA and QC, review and maintain Quality Manual
  - Function independently and without management influence

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Module 2 Changes – 5.2 Technical and Quality Manager

- **DOE-Only Requirement**– Requirements for Quality Manager (continued)
  - Arrange for or conduct internal audits and meet management schedule
  - Notify laboratory management of deficiencies in the quality system
- The requirement for the Quality Manager to inspect the Laboratory Information Management System (LIMS) annually was removed

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Module 2 Changes – 5.2 Technical and Quality Manager

- The requirement was essentially redundant with the requirements in QSM 5.4, previously M2 5.4.7.2, now M2 7.11 Information Management including:
  - Procedures for LIMS changes and validations
  - Detailed records for LIMS validations
  - Instructions and training for users
  - Electronic data security measures
  - Checks of data transfers and calculations

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Module 2 Changes – 6.2 Personnel

- ISO/IEC 17025:2017 contains more specific requirements for training compared to the previous revision:
  - 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

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Module 2 Changes – 6.2 Personnel

- The laboratory shall document the competence requirements **for each function influencing the results of laboratory activities**, including requirements for education, qualification, training, **technical knowledge**, skills and experience
- The laboratory shall ensure that the personnel have the competence to perform laboratory activities for which they are responsible and **to evaluate the significance of deviations**

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Module 2 Changes – 6.2 Personnel

- The laboratory shall have procedure(s) and retain records for:
  - Determining the competence requirements
  - Selection of personnel
  - Training of personnel

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Module 2 Changes – 6.2 Personnel

- Required procedures and training records (continued)
  - Supervision of personnel
  - Authorization of personnel
  - Monitoring competence of personnel

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Module 2 Changes – 6.2 Personnel

- The laboratory shall authorize personnel to perform specific laboratory activities, including but not limited to, the following:
  - **Development, modification, verification and validation of methods**
  - Analysis of results, including statements of conformity or opinions and interpretations
  - Report, review and authorization of results

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Module 2 Changes – 6.2 Personnel

- In the 17025:2017 revision, the requirement to have Job Descriptions was changed to "The management of the laboratory shall communicate to personnel their duties, responsibilities and authorities"
- QSM 6.0 adds "Records of these communications shall be maintained"

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Module 2 Changes – 6.2 Personnel

- **DOE-Only Requirement**-"Technical Manager educational and experience qualifications will be developed, required, and documented by the laboratory management"
- **DOE-Only Requirement-** "The Quality Manager, however named, shall have records of training and/or experience in QA and QC procedures and the laboratory's quality system, and have a general knowledge of the analytical methods for which data review is performed"

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Module 2 Changes – 6.2 Personnel

- Prescribed training requirements for Data Integrity/Deterring Improper Practices were rewritten to clarify the material to be included and the records of training required
- Moved the list of prohibited practices to section 4.1

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Module 2 Changes – 6.4 Equipment

- Changed the language from “support equipment” to “auxiliary equipment”
- Moved many of the requirements for auxiliary equipment from numbered paragraphs into the Table 6-1 so that each requirement occurs only once
- Adjusted some verification requirements in Table 6-1 to better match the use of the equipment:

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Module 2 Changes – 6.4 Equipment

Performance Check	Frequency	Acceptance Criteria
Verification of working standard masses (*i.e.*, masses used for daily balance verification) Option 1: comparison to calibrated reference weights not in daily use **Option 2: check on balance immediately (same day) after required balance calibration from accredited calibration provider**	Annually	± 0.1% or ± 0.2 mg, whichever is greater

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Module 2 Changes – 6.4 Equipment

Monitoring of refrigerator/freezer temperatures **Metrological Traceability not required for sample and standard storage**	Daily (i.e., 7 days per week) When personnel or an automated system are not available to record daily, use MIN/MAX thermometers or data loggers to monitor. Evaluate the data from devices upon return to the laboratory. The laboratory shall enact its nonconforming work procedures within 24 hours of detecting any excursion noted on MIN/MAX thermometers or data loggers with longer than 2 hours between measurements. **For data loggers recording more frequently, action shall be taken for any excursion of > 2 ˚C or any excursion > 2 hours.**	Refrigerators: 0 ˚C to 6 ˚C Freezers: ≤ -10 ˚C

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Module 2 Changes – 6.4 Equipment

Thermometer verification check Use a calibrated reference thermometer (**not required for sample and standard storage thermometers**) Perform **multiple measurements** at each of two temperatures that bracket the target temperature(s); **if the range of use is ≤ 10 ˚C** (e.g., 10 to 20 ˚C), **verification may be at a single temperature** within the range of use	Liquid in glass and electronic: Before first use and annually Hand-held infrared: Before first use and quarterly	Apply correction factors or replace thermometer

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Module 2 Changes – 6.4 Equipment

- For several types of equipment, QSM now states that metrological traceability is required when it impacts the validity of the results of the testing procedure (thus not required if the equipment does not impact the validity of the results of the testing), for example:

Timer **Metrological traceability is required when it impacts the validity of the result.**	Annually	Per laboratory procedure, ensure timer is fit for propose.

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Module 2 Changes – 6.5 Metrological Traceability

- Tightened the requirements for using reference materials within the expiration date stated on the certificate
- Expiration dates may only be extended when manufacturer provides a certificate with an extended date, and only if material is still in the original sealed packaging

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Module 2 Changes – 6.5 Metrological Traceability

- Now requires laboratories to use Certified Reference Materials (CRM) from a reference material producer accredited to 17034 for calibration standards, if available
- When CRMs from an accredited producer are used for calibration, second source calibration verification is no longer required (this will be addressed further in Module 4 and 8 presentations)

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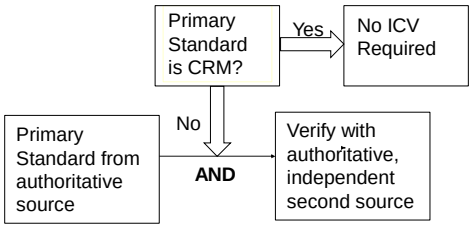
Module 2 Changes – 6.5 Metrological Traceability

- If no CRM from an accredited producer is available:



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Module 2 Changes – 6.6 Externally Provided Products/Services

- Previously called Purchasing Services and Supplies
- ISO 17025:2017 rolled requirements for subcontracting of testing into the section now called “Externally Provided Products and Services”
- Thus, QSM requirements for subcontracting have also been moved to section 6.6
- Other than change of location, there were no substantive changes to the subcontracting and purchasing requirements

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Module 2 Changes – 7.1 Requests, Tenders and Contracts

- Changed the requirements for waivers to require laboratories to obtain the waiver in writing from the “customer-identified technical point of contact” rather than from the “DoD or DOE Chemist or Contractor Project Chemist”
- Added a requirement that the record of approval of the waiver be included in all affected data packages

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Module 2 Changes – 7.2
Test Method Documents

- Test Method Documents ("SOPs")
  - –Reduced the number of topics which are required to always be addressed within a test method document to only those that are key to performance of the method
  - –Items no longer required in each test method document are still required to be included somewhere in the management system (e.g., in the quality manual)
  - –Indicated that the list of topics does NOT imply a specific format for the document

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Module 2 Changes – 7.2
Test Method Document Required Elements

- –Method identification
- –Any method modifications
- –Applicable matrix
- –Scope and application
- –Summary
- –Interferences
- –Safety specific to method
- –Equipment and supplies
- –Reagents and standards
- –Sampling, preservation, shipment, storage
- –Quality controls including preparation
- –Calibration type and instructions
- –Prescribed techniques and steps
- –Data analysis and calculations
- –References

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Module 2 Changes – 7.2
Test Method Documents

- Elements which shall be addressed but may be included by reference to other documents:
  - –Definitions
  - –LOD and LOQ
  - –Calibration evaluation and acceptance criteria
  - –Data assessment and acceptance criteria for QC
  - –Actions for handling unacceptable data
  - –General laboratory safety
  - –(DOE Only Requirement) cleaning labware
  - –(DOE Only Requirement) approaches to address background corrections

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Module 2 Changes – 7.2
Test Method Documents

- These items are optional
  - –Additional information on method performance
  - –Tables, diagrams, flow charts
  - –Method validation data
- Lab gets to decide when it is appropriate to include them

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Module 2 Changes – 7.4
Handling Test Items

- Requirements for sample acceptance procedures were rewritten to improve clarity
  - –For example, "proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink"
  - –Became "proper sample labeling to ensure readability and unique identity"

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Module 2 Changes – 7.4
Handling Test Items

- Legal Chain of Custody
  - –Some guidance ("should") statements were removed
  - –Laboratory is now required to have the procedure to be used for Legal CoC approved by the customer prior to accepting samples
  - –Ideally the customer will define the procedures
  - –There is still a basic set of requirements if there are no regulatory or customer requirements

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Don't forget the keys!

55

Module 2 Changes – 7.5 Technical Records

- New specific requirement to track alterations to the original version of automated instrument outputs (e.g., "Q delete") to ensure these alterations are reviewed by a technically qualified supervisor
- The list of records considered to be critical for historical reconstruction of analysis was rewritten to improve clarity

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Module 2 Changes – 7.5 Technical Records

- Changed TNI defined minimum retention of records from "5 years from last entry to the record" to "5 years after last use of the record"
- Defined that records are in use when it supports laboratory activities
- Expanded definition of preparation start and stop to include digestion in addition to extraction

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Module 2 Changes – 7.6 Measurement Uncertainty

- Removed the additional guidance for measurement uncertainty that had been in QSM 5.4 section 5.4
  - –Removing guidance where unnecessary was one of our goals
  - –The guidance is contained in the ISO17025-2017 notes for this section

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Module 2 Changes – 7.7 Ensuring the Validity of Results

- Previously called Quality Assurance for Environmental Testing
- ISO/IEC 17025:2017 greatly expanded requirements for ensuring the validity of results
- "This monitoring shall be planned and reviewed and shall include, **where appropriate**, but not be limited to: …

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Module 2 Changes – 7.7 Ensuring the Validity of Results

- –Use of reference materials or quality control materials
- –Use of alternative instrumentation that has been calibrated to provide traceable results;
- –Functional check(s) of measuring and testing equipment;
- –Use of check or working standards with control charts, where applicable
- –Intermediate checks on measuring equipment

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Module 2 Changes – 7.7 Ensuring the Validity of Results

–Replicate tests or calibrations using the same or different methods

–Retesting or recalibration of retained items

–Correlation of results for different characteristics of an item

–Review of reported results

–Intralaboratory comparisons

–Testing of blind sample(s)"

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Module 2 Changes – 7.7 Ensuring the Validity of Results

- Added clarifying language to define when any of these items is **appropriate**:
  - "All QC activities described within the reference method, as well as all QC requirements defined in the technical modules are applicable. **Items listed in** [ISO/IEC 17025:2017] **7.7.1 not specifically required by reference method, technical module, or Appendix B are not considered applicable for the purposes of the QSM"**

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Module 2 Changes – 7.8 Reporting of Results

- There was a major overhaul of reporting requirements
- Appendix A requirements were incorporated into Module 2 section 7.8
- These changes will be presented by Ms. Nancy Cooper

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Module 2 Changes – 8.2 Management System

- Management System Quality Manual
  - –ISO 17025:2017 eliminated the requirement for quality manual, but continues to require management to establish, document and maintain policies and objectives to fulfill the requirements contained in the standard
  - –**HOWEVER**…. QSM 6.0 kept the requirement for a quality manual, though it eliminated a couple of the requirements associated with it

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Module 2 Changes – 8.2 Management System

- Quality Manual changes
  - –Most policies required in the previous revision of the QSM have been eliminated in favor of required procedures
    - •No Quality Policy Statement,
    - •No Purchasing Policy,
    - •No Complaint Policy,
    - •No Nonconforming Work Policy…

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Module 2 Changes – 8.2 Management System

- The only policies still required are those found in ISO 17025:2017 to address:
  - –Competence
  - –Impartiality and
  - –Consistent operation of the laboratory

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Module 2 Changes – 8.2 Management System

- Quality Manual changes
  - –Other requirements eliminated include a document title, the name and address of the laboratory, the name and address and telephone number for the individual responsible for the laboratory
  - –Signed and dated concurrence of the quality manager, technical manager and agent in charge of all laboratory activities still remains a DOE-Only Requirement, but is not required for DoD

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Module 2 Changes – 8.2 Management System

- Quality Manual change justifications
  - –All major requirements from QSM 5.4 are still required topics in the quality manual
  - –All documents, including the quality manual, are reviewed and approved for adequacy prior to issue by authorized personnel
  - –All personnel involved in laboratory activities shall have access to all documents applicable to their responsibilities

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Module 2 Changes – 8.3 Document Control

- Eliminated the option for hand amendments
- Added a requirement to maintain an archived copy of retired/revised versions of documents for 5 years after retirement/revision

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Module 2 Changes – 8.8 Internal Audits

- Changed description of audit schedule to allow some flexibility within the annual internal audit requirements, but ensure all areas are evaluated within a period not to exceed 18 months
- This allows laboratories who perform internal audits throughout the year to change the order of the areas being audited while keeping the maximum time between audit activities limited

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Module 2 Changes – 9.0 HRMM

- Mr. Steve Clark will be presenting on Section 9.0 - 9.6 (DOE-Only Requirement) Hazardous and Radioactive Materials Management and Health and Safety Practices

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Module 2 Changes – Conclusion

- Much of the language in QSM 6.0 Module 2 is either the same as or very similar to QSM 5.4
- Substantive changes included incorporation of ISO 17025:2017 language and a complete rearrangement of paragraphs and organization of topics to match, added requirements to address risk specific to environmental laboratories, a few tightened requirements, and a few requirements loosened enough to allow laboratories greater latitude to achieve the required result
- Language has been upgraded to be more clear, concise, consistent throughout and flow more logically

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There's only two things I don't like:
Change and the way things are.

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QSM V6.0: V1M2 – HAZARDOUS AND RADIOACTIVE MATERIALS MANAGEMENT AND HEALTH AND SAFETY PRACTICES

Steve Clark
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Agenda

- DOE Concerns and Expectations
- Assessor Responsibility
- The DoD/DOE Quality Systems Manual, v6.0: V1M2, Section 9, Hazardous Radioactive Materials Management (HRMM)
- Prior Findings in HRMM

3

DOE Concerns and Expectations

DOE is committed to ensuring that environmental laboratories handling derived waste resulting from samples and analysis conduct operations in a manner that is protective of human health and the environment.

4

DOE Concerns and Expectations

DOE frequently sends samples with hazardous and/or radioactive constituents that require special handling to avoid worker, public, and environmental vulnerabilities and risks. The emphasis of DOE on general safety in the workplace is paramount. Therefore, DOE chooses to use only those analytical laboratories that can demonstrate effective management controls and reasonably diligent health and safety practices.

5

DOE Concerns and Expectations

- The laboratory shall determine its ability to safely receive and process the samples. The laboratory shall have the appropriate capabilities, procedures, and licenses to receive samples from a DOE site.
- Upon sample receipt, the laboratory shall assume the responsibility and liability for the safe and compliant management, storage, and disposal of the samples, and any associated analysis-derived wastes.
- Some DOE sites permit returning sample residues by pre-arrangement.
- The laboratory shall comply with all applicable federal and state regulations governing laboratory operations by developing, training, and implementing procedures.
- Plans shall be developed as they apply specifically to the laboratory's facility, staff, and DOE contractual requirements, deliverables, and obligations. The laboratory shall be assessed to these plans and its implementation on site.

6

DOE Concerns and Expectations

Failure to include in these plans/procedures specific federal, state, licensure, or permit requirements shall be identified as a finding requiring implementation of the nonconforming work process.

7

Assessor Responsibilities

- Assessors are not expected to be OSHA regulators.
- The expectation of the Assessor is to evaluate and/or confirm that the laboratories have the necessary procedures for the safe and accountable receipt, handling, and disposal of DOE samples and that the laboratories have implemented the requirements of their established procedures.

8

DOE Concerns and Expectations

We Defined DERIVED WASTE

Derived Waste: Derived waste is waste generated in the laboratory from samples, reagents, and residue from sample analysis. The derived waste can be non-hazardous, hazardous, and/or radiological (e.g., digestates, extracts, and aliquots).

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QSM v6.0: V1M2 – Hazardous and Radioactive Materials Management and Health and Safety Practices


Department of Defense
and
Department of Energy
Quality Systems Manual for
Environmental Laboratories
Version 6.0 – Draft
DRAFT

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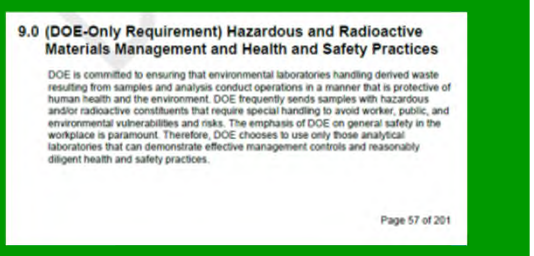
QSM v6.0: V1M2 – Hazardous and Radioactive Materials Management and Health and Safety Practices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Section 9: (DOE-Only Requirement) Hazardous and Radioactive Materials Management and Health and Safety Practices

Section 9:

DOE Only Hazardous and Radioactive Material Management

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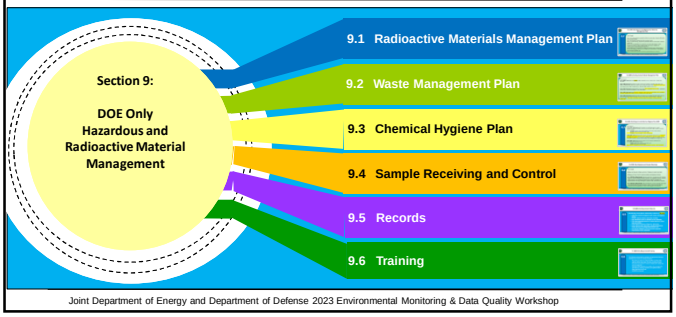
Section 9: (DOE-Only Requirement) Hazardous and Radioactive Materials Management and Health and Safety Practices



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9.1 (DOE-Only Requirement) Radioactive Materials Management Plan

9.1

What Changed:

9.1.3: A procedure will be documented to address how and when an alternate RSO will be necessary and available.
9.1.3: RSO Refresher Training no less than once every 3 years.
9.1.6.c at sample receiving, samples from potentially radioactive sites shall be screened to ensure that;
9.1.6.c.i customer identification of radioactivity (or lack of radioactivity) is correct;
9.1.6.c.ii the sample is properly categorized (per the laboratory's definition of radioactivity) for sample handling in the laboratory;
9.1.6.c.iii data input is obtained for the radioactive materials license tracking system in
9.1.6.c.iv the shipping container does not exhibit loose contamination or unacceptable external radiation readings; and
9.1.6.c.v that licensed material is secure from unauthorized access or removal.

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9.2 (DOE-Only Requirement) Waste Management Plan

What Changed:
9.2.1.i (DOE-Only Requirement) provide tracking of individual sample containers from receipt to final disposition;

9.2.2.b (DOE-Only Requirement) The laboratory shall develop criteria for evaluating waste brokers and TSDFs…DOECAP TSDF audits can be used in place of onsite visit requirements, provided other requirements not included in these audits are addressed (e.g., financial stability, liability insurance, etc.).

9.2.2.d (DOE-Only Requirement) Analytical process waste shall be segregated and removed to a designated storage area to minimize the potential for cross-contamination.

9.2.2.e (DOE-Only Requirement) Laboratory analysis for derived waste characterization shall be repeated at a frequency adequate to account for all known variations in the waste streams.

9.2.2.h (DOE-Only Requirement) The laboratory shall address how it manages the requirements for the pre-treatment requirements if disposal includes a Publicly Owned Treatment Works (POTW) or wastewater treatment system. The program shall address how the laboratory demonstrates compliance with these requirements.

15

9.3 (DOE-Only Requirement) Chemical Hygiene Plan (CHP)

9.3

What Changed:
9.3.12 (DOE-Only Requirement) If respirators are used during sample or waste handling/processing, the laboratory shall have an appropriate written respiratory protection program, including:
- **9.3.12.a** procedures governing the fit-testing of personnel using respirators;
- **9.3.12.b** selection and use of respirators; and
- **9.3.12.c** an annual evaluation to ensure effectiveness.

9.3.20 (DOE-Only Requirement) The laboratory shall establish and implement a procedure(s) for identifying hazardous and toxic chemicals located within the laboratory, locations stored, and training of personnel. The procedure(s) shall address precautions for handling and storing all hazardous and toxic chemicals used to include proper identification of storage areas.

9.3.21 (DOE-Only Requirement) All hazardous or toxic chemical cabinets shall be appropriately labeled with the following:
- **9.3.21.a** identity of the hazardous chemical(s); and
- **9.3.21.b** Appropriate hazard warnings.

(Removed Name and address of the chemical manufacturer, importer, or other responsible party.

16

9.4 (DOE-Only Requirement) Sample Receiving

9.4

What Changed:

9.4.2.e recording and notifying customers of shipping or sample anomalies;

9.4.2.g use of fume hoods for opening samples and shipping containers;

9.4.3 (DOE-Only Requirement) Materials submitted to the laboratory for industrial hygiene or asbestos analyses shall be opened in an established manner to prevent worker exposure, and sample-receiving practices shall be developed and implemented for the receipt of beryllium, beryllium oxide, and asbestos materials.

9.4.6 (DOE-Only Requirement) When the laboratory receives samples, there shall be an internal chain of custody procedure in place. Internal custody shall be maintained until final disposition or return of the sample to the customer.

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9.5 (DOE-Only Requirement) Records

9.5

- **The following records shall be maintained for a minimum of five years:**
  - **radioactive material management audit, review, and inspection reports;**
  - **records of airborne release of hazardous materials;**
  - **daily operational checks of radiological survey equipment;**
  - **TSDF waste brokering evaluation or review reports and a list of approved facilities;**
  - **waste disposal certificates of disposal or destruction;**
  - **waste characterization information, including analytical test results and process knowledge determinations; and**
  - **semi-annual ventilation hood and protective equipment contamination control verifications.**

18

9.6 (DOE-Only Requirement) Training

9.6

The following training shall be provided to all appropriate laboratory employees and records of training maintained:

- RSO training for both the designated RSO and backup RSO;
- radiation general awareness, security, and safety for those laboratory personnel and contractors dealing with radioactive waste management;
- Hazardous Waste Satellite Accumulation Area management;
- spill detection, cleanup procedures, and spill kit location;
- safety training (annual); and
- HAZWOPR, as required.

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Examples of "Section 9" Findings

20

Examples of "Section 9" Findings: Gas Cylinder Safety

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Examples of "Section 9" Findings: Gas Cylinder Safety

Gas Cylinder Safety and Monitoring:

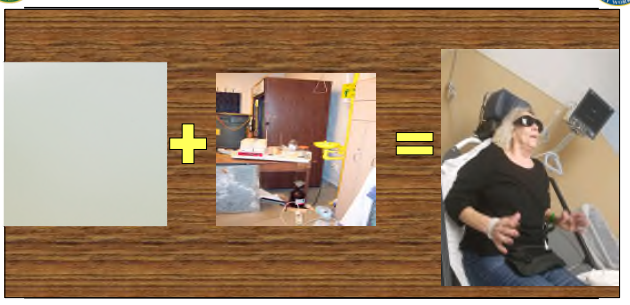
Indian Institute of Science (IISc) in Bengaluru (2018)
Gas Cylinder Explosion- Kills one, Injures 12. "IISC, which employs about 450 scientists on a sprawling campus, has "a very good safety record," says biochemist and former IISc Director Padmanabhan Balaram, who says this is likely the first death because of a research-related accident in the institute's 110-year history. "

22

Examples of "Section 9" Findings: Eye Wash and Shower Station Access:

23

Examples of "Section 9" Findings: Eye Wash and Shower Station Access:



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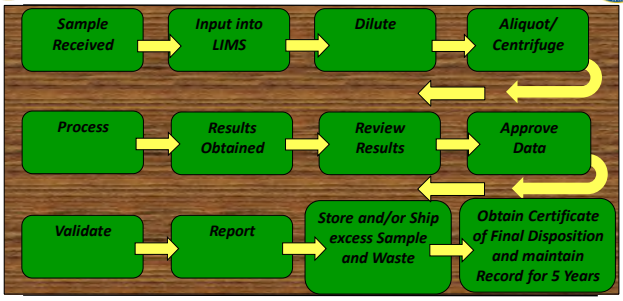
Examples of "Section 9" Findings

Incompatible Chemical Storage:

25

Examples of "Section 9" Findings: Waste Tracking and Disposition



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Today's Presenter

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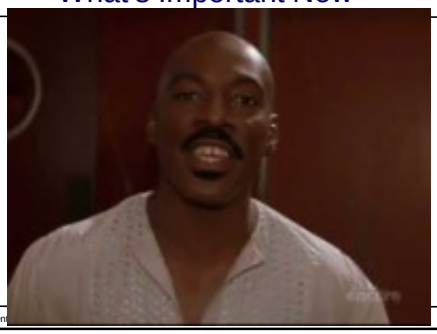
What's Important Now



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Questions and Comments Are Always Welcome

29

QSM V6.0: MODULE 6 – RADIOCHEMISTRY

William J. (Bill) Rogers, Ph.D., United Cleanup Oak Ridge LLC

1

Caveat

- QSM 5.4 was based on TNI 2009
  - The TNI 2016 update made significant changes to their VOLUME 1, MODULE 6 Quality Systems for Radiochemical Testing
- QSM 6.0 is based on TNI 2016
  - The update to TNI was significant which makes simple side-by-side comparison of 5.4 to 6.0 very difficult

2

Broad changes

- Directly incorporated text from TNI 2016
  - (updated from 2009)
- Renumbered / Repositioned
- New Section 3.2 Exclusions and Exceptions

3

New: 3.2 Exclusions and Exceptions

The elements of this module apply to techniques used for the purpose of measuring or monitoring radioactivity, or techniques used to demonstrate compliance with regulations pertaining to radioactivity. The laboratory shall comply with the requirements of Module 4 in cases where technique-specific QA/QC is not defined in Module 6 (e.g., Mass Spectrometry [ICP-MS, TIMS] or Kinetic Phosphorimetry) or by the respective reference method (e.g., calibrations, calibration verifications, determinations of detection statistics, or method-specific QCs). The laboratory shall identify in its Quality System how and when it is complying with the requirements and elements of Module 4, and Module 6, as applicable.

4

Other Broad Changes

- Moved equations from 1.3 closer to point of use without changes
  - We hope to have clarified detection limit / critical level
- Added Safe Drinking Water Act detection limit rule – (this is not in MARLAP)
- Moved definition-like" text to the definitions section

5

Deletions / Modifications

- Material balance checks on alpha calibration standards (old 1.7.1.a)
- Long count – energy calibration check before batch (old 1.7.1.b)
- Counting efficiency redetermination when check source fails (old 1.7.1.b)
- Monthly efficiency for radon scintillation detectors (Rev. 6.0 changed to annual) (old 1.7.1.b)
- Successive long backgrounds in lieu of shorter checks (old 1.7.1.c)
- Extension of background check frequency for long counts (old 1.7.1.c)
- For alpha spec, weekly short Instrument Contamination Check (ICC) allows reporting without ICC bracketing (old 1.7.1.c)

(many thanks to Richard Weiss for this slide and the ones which follow)

6

Deletions / Modifications

- Old 1.7.2.2a
  - LCS must contain one sample-specific analyte 5-20 times RL (Rev. 6.0 LCS at minimum 1/3rd of acceptance criteria)
  - Gross Alpha/Beta – LCS uses the same analytes (Rev. 6.0 use "appropriate" analyte)
  - LCS traceable to NIST (Rev. 6.0 only specifies meet reference standard criteria)
- Old 1.7.2.3a
  - Matrix spike recovery 60-140% if spiking > 5 times sample activity (Rev. 6.0 "as specified by method)
  - Elevated sample activity, MS counted for equal duration as sample
- Old 1.7.2.3b
  - No averaging of sample/duplicate results for reporting

7

Deletions / Modifications

- Old 1.8.1 (Alpha Spec)
  - No blank correction except as customer specified
  - FWHM criteria, Tracer <100 keV within 50 keV of reference energy
  - Slope < 15 keV per channel
  - Calibration range 3 to 6 Mev
  - Peaks within 40 keV of reference
  - Efficiency calibration >3000 net counts
  - Check source >2000 net counts

8

Deletions / Modifications

- Old 1.8.2 (Lucas)
  - Added reference to EPA Method 903.1
- Old 1.8.3 (Liquid Scintillation)
  - Calibration and detector response sections added
- Old 1.8.4 (GFPC)
  - Efficiency calibration >10000 net counts
  - Check source >5000 net counts
  - Check source and background run after gas bottle changes before samples
- Old 1.8.5 (gamma)
  - None

9

"Reasonable" Modifications

- To give you the flavor of our intent, a bit more detail on changes to LCS and MS
- Changes are consistent with TNI 2016

10

"Reasonable" Modifications – LCS Selection and Level

- Old
  - The LCS shall be counted for a sufficient time to quantify the activity level of the LCS (dropped).
  - The LCS matrix shall be the same as the samples, or as close as can be reasonably achieved, and the matrix shall be documented in the Case Narrative.
- Changed to
  - The material used to create the LCS should be free of analytes of interest at levels that will interfere with the evaluation of the results. If an analyte-free matrix is not available, the laboratory may use a surrogate matrix to simulate the sample matrix. If analyte-free materials are not available for the LCS, the materials shall be characterized and documented for the analyte(s) of concern and accounted for in the evaluation of the LCS

11

LCS cont'd

- Old – Spike at 5-to-20x RL. Must be NIST-traceable Standard materials
- Changed to
  - The laboratory shall spike the LCS at a level such that the uncertainty of the analytical result is less than 1/3 of the acceptance criteria. . . . When practical, the LCS should be spiked at a level comparable to the action level if known; or that of routine samples if the activities are expected to exceed 10 times the Decision Level (Critical Value).
  - When available, the standard used to prepare the LCS shall meet the requirements for reference standards. The final prepared LCS need not be traceable to a national standard organization. The LCS shall include all of the radionuclide(s) being determined with the following exceptions:

12

LCS cont'd

- Changed - LCS for Gross Alpha/Beta "shall be" same isotopes as used in efficiency curves. Changed to read "This will generally be the radionuclide(s) used to calibrate the detector"
- Dropped - LCS shall contain "at least one analyte reported for samples by that analytical method (separation chemistry and decay mechanism) "
- Added - for multi-analyte alpha spec only one analyte is needed in the LCS.
- Added – clarifying text that a gamma isotope with similar energy may be used (e.g. Ba-133 for I-131)
- Added – low energy (Am-241) and high energy (Co-60) isotopes for gamma with mid-range Cs-137 noted as "commonly included." (previously "approximate energy region")

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"Reasonable" Modifications – Matrix Spike

- Rephrased / changed - "added as early in the sample preparation step as practical" to "prior to performing any processes that affect the analyte of interest . . "
- Rephrased to "MSs are not typically employed for non-destructive methods (e.g., gamma spectrometry or direct counting of samples for alpha or beta radioactivity), or for methods that employ a chemical yield tracer or carrier for each sample"
  - Dropped exclusion wherein MS was not required for "non-aqueous tritium"
- New language: Frequency of MS is per contract but "consistent with LCS"
- Changed spiking level from "at least 5 but not greater than 20x RL" to "greater than 5 times the MDA."

14

B-Tables

- Still in draft – but progress!
- Trying to make completely consistent among themselves
- Trying to avoid any conflict between body of Module 6 and the Tables

15

Revision finalized last week

- Throughout: Changed to "Decision level" from "critical value" or required detection reporting limit (RL)
- Validation procedures re-worded
- "error" changed to "uncertainty"
- "fail" to "fall outside"
- "Corrective action" changed to "non-conforming work procedure"
- "Subtraction background" changed to "Background Subtraction"

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More changes

- 5.2.1.f.i Blank Population - Added "Equations may need to be modified depending on the measurement technique in use"
- 5.4.4.f – Added: In the case of zero counts, the uncertainty of the count is assumed to be the square root of one count. The uncertainty of a net count would have to propagate the uncertainty of the sample and background. Thus the uncertainty for a zero count background and zero count sample is assumed to be 1.4 (square root of 2). – (also added to maintain records of customer acceptance.)
- 6.2.3 and 6.3.1 Methods where spiking is not viable – two places, removed the text "e.g. leaching procedures."

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More

- 7.1.1.b Initial set-up: Maintain records of customer acceptance if you are deviating from "manufacturer recommended specifications "
- 7.1.6a.1 – dropped short term background check "should be performed" paragraph.
- 7.2.1A – dropped paragraph describing why we run batch QC.
- 7.2.2.h - Dropped requirement for control charts when a reagent blank is used to correct results.
- 7.2.2.h.iii - Filter blanks should be supplied by the customer from same lot
- 7.2.4b.v Dropped paragraph regarding LCSD in lieu of Method Duplicate (MD)

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Even more

- 8.2.1.d – Gamma: 5,000 counts rather than 10,000 counts for initial calibration / 4 peaks instead of 6
- 8.2.4b – Dropped "shall have" seal and ingrowth by gamma method for Ra-226.
- 8.2.6b – clarified what is meant by an "unidentified peak"
- 8.3.1.f.iv – Crosstalk correction is not necessary when there is no net activity in the opposing channel as is the case when counting chemically separated radionuclides.

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- 8.4.2.e - liquid scintillation - ensure phase separation "minimized" changed to "no visual evidence of phase separation"
- 8.4.3 dropped mention of method of standard addition.
- 8.4.5.c – type of water for background clarified to contain (dead water, DI water, etc.)
- 8.5 – Lucas Cell - deviations from methods must be accepted by customer. Other expansions /additions of quality control sample descriptions. Please read carefully.

20

5.1.7 Validation procedures shall include~~The laboratory shall analyze for all methods,~~ whenever available, externally-produced quality control~~QC~~ samples obtained from a Reference Material Producer accredited to ISO 17034 or a national metrology institute. When such reference materials cannot be obtained, that laboratory may use materials from a Proficiency Testing Provider accredited to ISO/IEC 17043 or from another authoritative source.~~from a nationally- or internationally-recognized source (i.e., a national metrology institute, accredited TNI Proficiency Test (PT) Provider, an accredited ISO/IEC 17043 PT Provider, an accredited ISO Guide 34: 20096 reference material provider, or from an ANSI N42.227 compliant PT manufacturer).~~ The laboratory shall evaluate the results of these analyses to determine its ability to produce acceptable data.

~~Note: The use of non-TNI accredited PT Providers is strictly for method validation purposes, and not for laboratory accreditation.~~

21

7.1.4.a.vii The laboratory procedure shall specify what corrective actions are to be taken when performance check acceptance criteria are not met; and

~~Note: If a performance check result exceeds established limits, instrument performance may have changed since the initial calibration. The laboratory should verify that the change is not attributable to normal statistical variability of the check measurement prior to taking corrective action.~~

7.1.4.a.viii When results for instrument performance checks are not within the acceptance criteria (i.e., limit of a statistical or tolerance chart or other QC parameters), the ~~cause of the problem shall be investigated~~laboratory shall institute its non-conforming work procedures.

~~**7.1.4.a.viii.a** If a performance check fails, the laboratory can immediately analyze two~~

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Questions

- Questions?

23

MODULE 4 - CHEMISTRY

John Gumpper

ChemVal Consulting, Inc

1

(As my proposed epitaph will say)

"Well, THAT didn't go according to plan!"

2

Introduction

- With Modules 3, 5, and 7, a few wording changes were all that was necessary
- Module 4 in the current QSM had a significant number of "in lieu of…" paragraphs
  - –TNI 2016 added some of what QAOS wanted
  - –Thought QAOS could make minor edits for the rest
  - –Ended up with significant deletions, edits and additions

3

Introduction

- This presentation will focus on the differences
  - –Changes from QSM 5.4
  - –Changes from TNI 2016
- This presentation will focus on bigger picture items for the whole community
- Will go through the Module in order
  - –DL/LOD/LOQ in another presentation

4

Method Validation

5

Method Validation

- DoD EDQW, DOECAP DQW, the assessor corps, and some Accreditation Bodies (ABs) have long been concerned about method validation
  - –Method modifications
  - –Laboratory-developed methods
- Language has been added to try to make clear the evaluation that is required

6

Method Validation

- Biggest Concerns
  - –Laboratories are modifying methods without thorough validation of the modifications
  - –Customers are not aware laboratories are using modified methods

7

Method Validation Requirements

- Why modify methods?
- Improve performance
  - –Solve sample interference issues
  - –Take advantage of new technologies
- Improve efficiencies

8

Method Validation Requirements

- 5.1: Prior to acceptance and institution of any method for which data will be reported, all methods shall be validated
- 5.1.1: Requirements in Module 2 are operative
- From Module 2:
  - –In cases where modifications to published reference methods have been made by the laboratory, these modifications shall be clearly identified and described in the method instructions. [M2, 7.2.1.2.b.i]

9

Method Validation Requirements

- Also from Module 2: Specific examples of improper, inappropriate or prohibited actions include
  - –Reporting data from a modified method without customer approval, including, but not limited to, changing the stoichiometry or detection system of a method, reducing the number of extractions, or reducing acid concentrations for digestions. [M2, 4.1.6.h.xxv]

10

Method Validation Requirements

Methods are referred to in two categories

- –Reference methods
  - •Published methods
- –Modified methods and non-reference methods
  - •Reference methods used outside their scope
  - •Reference methods modified by the laboratory
  - •Laboratory-developed methods

11

Method Validation Requirements

Validation of Reference Methods

- –Initial Determinations of
  - •DL (if required)
  - •LOD (if required)
  - •LOQ
- –Initial Demonstration of Capability

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Method Validation Requirements

- Everything required of Reference Methods, plus
- Use QA procedures and QC acceptance criteria consistent with similar reference methods or technologies

13

Method Validation Requirements

- While not a new requirement, it's worth a reminder that method validation shall be done according to a written plan
- Experiments and acceptance criteria shall be predetermined
- Records of the validation shall be maintained and include a statement the method is fit for purpose

14

Method Validation Requirements

Include the following in the validation plan

- –Scope
- –Calibration verification
- –Interferences and cross-contamination
- –Analyte identification

15

Method Validation Requirements

Include the following in the validation (continued)

- –Analyte quantitation
- –Selectivity
- –Sensitivity
- –Precision and bias

16

Method Validation Requirements

Methods shall be validated when substantive modifications are made to reference methods (e.g., stoichiometry, technology, mass tuning acceptance criteria, quantitation ions, compressing digestion or extraction timeframes, reducing reagent or solvent volumes, or changing solvents) [M4, 5.1.5]

17

Method Validation Requirements

Modifications to sample preparation steps

- –Shall include analysis of field samples
  - •In matrices of concern
  - •Represent a range of characteristics encountered or expected
- –Use parallel studies, where possible

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Method Validation Requirements

Examples of characteristics to consider

- –Organic matter content
- –Clay content
- –Moisture content
- –pH
- –Dissolved/suspended solids

19

Method Validation Requirements

Field samples used for demonstration

- –Shall contain target analytes
  - •Native
  - •Spiked
- –Multiple levels of target analyte concentrations
- –More than 45 target analytes, may use a subset
  - •Include all chemistries, bad actors

20

Method Validation Requirements

- One more note:
- Where modifications to **only the analytical portion** of the method are planned, the laboratory shall take into consideration any effects the matrix may have on the analysis as part of its risk assessment [M4, 5.1.8]

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Method Validation Requirements

- Thoughts for customers of laboratories
- Be aware of requests from laboratories to use modified methods
- Ensure method has been validated to be fit for project's purposes
  - –May have regulatory implications
  - –May want to include a government chemist in your discussions

22

Demonstrations of Capability

- Added a few clarifications [M4, 6.2.3]
- For methods where spiking is not a viable options (e.g., leaching methods) DOC shall include observation and evaluation of negative controls
- QS Matrix for DOCs shall be similar to samples
  - –For analysis of metals in solids, materials such as washed sand or non-reactive bead are acceptable

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Calibration

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Initial Calibration

Standards used for calibration shall be Certified Reference Materials specifically identified as such in an accompanying Certificate of Analysis from a Reference Material Producer (RMP) accredited to ISO 17034, when available [M4, 7.1.1.d]

- –Change in Standard, not in practice-ABs have required this previously, as noted by Ms. Gumpper in the Module 2 presentation

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Initial Calibration

- This is a big change
- When Certified Reference Materials are used for the initial calibration, the calibration is NOT required to be verified with a standard from a second source
  - –Reliability of preparation
  - –Dilution verifications

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Initial Calibration

If CRMs are not available

- –Use standard from an authoritative source

AND

- –Verify all initial calibrations with a standard from an authoritative independent source (“ICV”) [M4, 7.1.1.n]

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Initial Calibration

For regression or average response/calibration factor calibrations, minimum non-zero standards

Type of Calibration Curve	Minimum Number of Calibration Standards[b,c]
Threshold Testing[a]	1
Average Response	5
Linear Fit	5
Quadratic Fit	6

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Initial Calibration

Changes to the Footnotes [M4, 7.1.1.f]

- –a: The initial one-point calibration shall be at the project-specified threshold level and results shall be reported qualitatively with uncertainty, and in compliance with a decision rule

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Initial Calibration

- –b: Fewer calibration standards may be used only if equipment firmware or software cannot accommodate the specified number of standards. Records detailing that limitation shall be maintained by the laboratory
- –c: Ion-selective electrode analyses, e.g., pH, ammonia, are not covered by this table. The laboratory shall use the minimum number of standards as stated in the reference method or manufacturer’s instructions

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Initial Calibration

Results shall not be reported from responses above the high standard

- –Dilute and reanalyze within the calibrated range
- –If reanalysis not possible, report with appropriate data qualifiers

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Initial Calibration

- All target analytes require a multi-level calibration
- Surrogate compounds may be calibrated as a multi-point calibration at a single level
  - –What's this multi-level and multi-point thing? Aren't they saying the same thing?

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Multi-Level vs. Multi-point

New Definitions in the QSM

- –Multi-level Calibration: Calibration using standards with differing concentrations to determine an instrument response across a calibration range.
- –Multi-point Calibration: Calibration using standards with differing concentrations to determine an instrument response across a calibration range
- –Mid-Range: The concentration equal to 50% of the highest calibration standard concentration.

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Initial Calibration

- ICP Exceptions to range requirements
  - –Results above calibration range may be reported with high-level check standard
    - •Exceeds the sample concentration
    - •Within linear dynamic range
    - •"Passes" within 10% of true concentration

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Initial Calibration

Clarification: ICAL evaluation shall include both

- –Goodness of fit evaluation
  - •%RSD, or
  - •Correlation Coefficient or Coefficient of Determination, AND
- –Evaluation of error
  - •%RSE, or
  - •%RE for midpoint, lowest non-zero point

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Initial Calibration

Table B-2 Excerpt

ICAL for all analytes (including surrogates)	At instrument set-up and when needed based on QC results, prior to sample analysis.	Minimum 5 levels for when using evaluation by %RSD or linear regression and 6 levels for evaluation by quadratic regression. Each analyte shall meet one of the three options below: Option 1: %RSD for each analyte ≤ 20%, unless the specific method referenced has tighter criteria, in which case the method shall be followed; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$;	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

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Initial Calibration

Table B-2 Excerpt

Evaluation of relative error	Each ICAL using options 2 or 3 above shall also be evaluated for relative error either by determination of the %RE at or near the mid-range and low level of the ICAL or by the determination of the %RSE.	The laboratory shall meet the criteria listed in the reference method revision used for analysis and reporting. If no criteria are listed, the laboratory shall develop its own criteria; however, the maximum allowable %RE at or near the mid-range and low level of the calibration shall be 15% and 40%, respectively. The maximum allowable %RSE shall be 20%.	Correct problem, then repeat ICAL. Qualification of data is not appropriate.

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Initial Calibration

- Aroclor analysis
  - –ICAL shall be multi-level for a subset of Aroclors, e.g., 1016/1260
  - –Single-level allowed for pattern matching for others
  - –If others are identified, a multi-level shall be analyzed, and sample extracts re-analyzed for quantitation
- Not new, but now in the Standard

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Continuing Calibration Verification

Instrument calibration verification shall be performed at the beginning and end of each analytical batch, and at the frequency defined in the method

- –No exception for internal standard analyses
- –Using a LCS or a second-source ICV is an acceptable alternative if results meet CCV acceptance criteria

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Continuing Calibration Verification

- Previous QSM exception allowing two passing CCVs to negate one failing CCV has been removed
- Exception allowed for obvious gross failures, e.g., missed autosampler injection

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Continuing Calibration Verification

- Exception for “acceptance criteria for the CCV are exceeded high” allowing reporting of non-detects with qualification has been kept
  - –Results shall be reported with qualification
- Exception for “acceptance criteria for the CCV are exceeded low” that allowed reporting of values above regulatory limit has been removed

41

Quality Control Criteria

Required QCs and how to make them

42

Quality Control Criteria

The clarification of “blank quality system matrix” for metals is included in the sections for Method Blank and Laboratory Control Sample

- –“For analysis of metals in solids, materials such as washed sand or non-reactive beads are acceptable as a matrix”

43

Method Blank

M4 includes clarification on chromatographic analyses of blanks when analyzing a batch on multiple instruments

- –Method Blank is only required to be analyzed once
- –Other types of blanks may be used to show cleanliness on other instruments

44

Laboratory Control Sample

- Components to be spiked
  - –All reported (target) analytes, except Aroclors
  - –May require multiple LCS samples to avoid interferences
- Concentrations at or below mid-range of calibration
  - –MS may be used in place of LCS if acceptance criteria are as stringent

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Matrix Spikes

Each preparation batch shall contain an associated MS and MSD from the specific project

- –Unless exempted by method or B-Table
- –Some B-Tables allow MD instead of MSD

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Matrix Spikes

- If inadequate material amount for MS/MSD
  - –Note in Case Narrative
  - –Perform LCSD
- Spike with all reported analytes, except Aroclors

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Matrix Duplicates

- Each preparation batch when not containing a MSD
- In general
  - –Use MSD when target analytes are not expected
  - –Use MD when target analytes are expected

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Selectivity

- Confirmation for non-Mass Spectrometry chromatography methods
  - –Required for all results greater than DL
- Confirmation techniques include
  - –Second, dissimilar column
  - –Second detector type, e.g., MS confirmation
  - –HPLC UV-Diode Array not allowed for UV detector

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Selectivity

When using second-column confirmation

- –Calibration and QC criteria for confirmation using the same detector type shall be the same as primary analysis
- –Laboratory shall identify the primary column for each target analyte
- –Results reported from the secondary column shall be discussed in the case narrative

50

Selectivity

If a lab uses a mass spectrometer for confirmation for a non-mass spec chromatography method, the laboratory shall have a procedure that includes acceptance criteria for

- –Selectivity
- –Sensitivity

51

Selectivity

- Customer shall be informed of unconfirmed results
  - –Qualifiers
  - –Case Narrative
- Analyte presence shall only be reported as positive
  - –If original and confirmation signals are positive, or
  - –If confirmation signal cannot be discerned from interference (still requires notification)

52

Data Acceptance/Rejection Criteria

Evaluation of Quality Control Results

53

Method Blank

- Method blank-Goal is to have no detectable contaminants
- Considered contaminated if target analyte in blank
  - –Exceeds 1/2 the LOQ, or
  - –1/10th the amount measured in any associated sample
  - –Whichever is greater

54

Method Blank

If a method blank is contaminated

- –Reprepare and analyze all affected QC and field samples processed with the contaminated blank if sufficient material is available.
- –Samples are “affected” if sample result is greater than DL and less than 10X the amount in the MB
- –If cannot be reprepared, report with qualifier on specific analytes in all samples in batch

55

Laboratory Control Sample

Acceptance criteria

- –Provided by the customer, or, if not,
- –Laboratory-developed criteria (hold that thought)

56

Laboratory Control Sample

If results are outside acceptance criteria

- –Reprepare and analyze the LCS and all affected QC and field samples in the associated preparation batch for failed analytes, if sufficient material is available
- –If cannot reprepare, report results with data qualifier applied to specific analytes

57

Laboratory Control Sample

Exceptions

- –If acceptance criteria are exceeded high and associated samples are non-detect, report non-detects with a qualifier, not necessary to reprepare/analyze
- –There is no exception when criteria are exceeded low
  - •There used to be one; it’s been removed

58

Laboratory Control Sample

Laboratory shall develop acceptance criteria for all analytes on its scope

- –Statistically derived from lab’s historical data
- –Scientifically valid calculation procedures
- –Meet the limits of the reference method, if available
- –No wider than ± 3 SD from mean

59

Laboratory Control Sample

Acceptance criteria, continued

- –Updated at least annually or as stated in the reference methods, whichever is more frequent
- –Re-established after major changes
- –Based on at least 30 data points under same system
  - •Do not exclude “failed” data without valid reason
  - •Not outside ±3 SD of mean LCS recovery

60

Laboratory Control Sample

- Use laboratory-developed limits for trend and batch control
- Control charts shall be maintained
  - –Monitored at least quarterly for shifts in mean, changes in SD, trends
  - –May use representative compounds for trend analysis
    - •Procedure shall document selection criteria

61

Laboratory Control Sample

Marginal Exceedances

- –All the TNI language is there
- –Not allowed for target analytes, i.e., chemicals of concern identified by the customer, without customer approval

62

Matrix Spike, Matrix Spike Duplicate

- %R Acceptance criteria are the same as for the LCS
- RPD acceptance criteria
  - –Customer requirement, or, if none
  - –Applicable B-Table

63

Matrix Spike, Matrix Spike Duplicate

Results outside acceptance criteria

- –Evaluate for analytical error
- –If error and if sufficient sample, reprepare and analyze the affected QC samples
- –Otherwise, qualify specific analytes in the parent sample

64

Matrix Duplicate, LCS Duplicate

Evaluate the same way as just described for Matrix Spike Duplicates

- –Except Matrix Duplicate RPD is not evaluated if both results are < LOQ

65

Surrogate Spikes

- Acceptance criteria
  - –From customer, or, if not specified
  - –Appendix C limits, or, if not in App. C
  - –Laboratory developed from LCS data
- If outside acceptance criteria
  - –Check for analytical error, reprep/analyze if found
  - –If not reprepared, apply qualifier to associated analytes

66

Sample Handling

Temperature criteria were moved to M2

67

Preservation Checks

- Laboratory shall check preservation before or during preparation or analysis (generally done at receipt)
- Except for VOAs, which shall be checked after analysis.

68

Storage Blanks

- Stored with all VOA samples
- Analyze every 14 days at a minimum as samples
- If greater than 1/2 LOQ (Methylene chloride, Acetone, 2-Butanone greater than LOQ), implement nonconforming work procedure
- Laboratory shall have procedures and acceptance criteria

69

Any Questions?

Thank you!

70

MODULE 8-INDUSTRIAL HYGIENE

John Gumpper, ChemVal Consulting, Inc.

1

Contradiction

- From *The House at Pooh Corner*
- *-A. A. Milne*

2

IH and the QSM

- Industrial Hygiene (IH) analyses are important to some DoD and DOE programs using laboratories accredited to the QSM
- Previously, IH was sparsely addressed and did not have its own section in the QSM
- Chemical Testing samples and IH testing samples have significant differences

3

IH Testing vs. Environmental Testing

- Chemical Testing Samples
  - –Widely varying matrices
  - –Push for very low detection
- IH Testing Samples
  - –Well-characterized, well-behaved sorbent matrices
  - –Sensitivity is more a function of sampling time

4

IH Testing vs. Environmental Testing

Many requirements for chemical testing analyses (M4) are overkill for IH analysis

- –IH laboratories have struggled with navigating accreditation to the QSM
- –Some requirements don't apply
- –Applying the older QSM versions and Module 4 might be seen as an actual "Contradiction"
- –IH labs will better served with specific IH requirements

5

Development of Module 8

Initial thought

- –Change the PT requirements to reflect IH norms
- –Add technical manager requirements
- –Change requirements for LOD/LOQ to reflect IH norms
- –Add a few B-Tables
- –Voilá!

6

Development of Module 8

- However:
- As we tried to make requirements bend in one area after another to accommodate standard IH practices, it became clear that what we needed was a full module that mirrored Module 4
- So, that's what happened

7

Development of Module 8

- Final Product
  - –PT requirements, plus
  - –Full set of requirements similar to Module 4
  - –B-Tables for IH Technologies
- Module now aligns more closely to American Industrial Hygiene Association (AIHA) requirements

8

Module 8 Detail

Hitting the high points

9

PT Requirements

- Addressed in the Module 1/Module 8 PT presentation yesterday
- Includes Round-Robins and Internal PT programs
  - –Based on the AIHA Laboratory Accreditation Program

10

Method Selection

- No significant differences for IH
- Follow Module 2
- Additions of analytes to reference methods
  - –Meet all calibration and QC of method
  - –If none, use requirements for same technology
  - –Follow regulations to determine if the proposed change constitutes a method modification

11

Validation of Methods

In addition to Module 2 Requirements, reference methods validated by

- –Determination of Detection Limit, if required
- –Determination of Limit Of Detection, if required
- –Determination of Limit of Quantitation
- –Initial Demonstration of capability

12

Validation of Methods

Modified reference methods and non-reference methods require additional validation

- –Requirements as in Module 4
- –Definition of "Modification" as in Module 4
- –**Significant extra matrix evaluation for preparation modifications is only required for bulk samples, e.g., soil, paint chips**

13

Validation of Methods

Bulk sample validation requires analysis of field samples containing target analytes, either natively or through spiking.

- –Multiple levels of target analyte concentrations
- –Samples that are like or from specific sampling sites in which the method will be used

14

Validation of Methods

Where modifications to only the analytical portion of the method are planned, or modifications in methods using routine sorbents, **the laboratory shall take into consideration any effects the matrix may have** on the analysis as part of its risk assessment

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DL, LOD, LOQ-General

- The requirements for Determination of
  - –Detection Limit (DL)
  - –Limit of Detection (LOD), and
  - –Limit of Quantitation (LOQ) are different for IH
- In general, data is not reported below LOQ
- In general, low detection is not required

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DL, LOD, LOQ-General

- Determination of DL and LOD is not required for gravimetric analyses or asbestos
- Determination of DL and LOD is not required if results are not reported below LOQ
  - –Unless required by regulation or method

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DL, LOD, LOQ-General

- For each analyte in each field of testing, the laboratory shall have procedures for determining and verifying DL (when required), LOD (when required), and LOQ that reflect current operating conditions
- The laboratory shall determine a DL (when required), LOD (when required), and LOQ for each preparation method unless it falls within one of the stated exceptions

18

DL, LOD, LOQ-General

- The laboratory is not required to determine DL, LOD, and LOQ for every possible combination of preparation and cleanup techniques as long as it determines them using the combination of processes most likely to interfere with sensitivity
- DL, LOD, and LOQ shall be reported unless it is not applicable
- Records of all determinations shall be maintained

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Detection Limit (DL)

When required, the laboratory shall determine the DL using published methodologies from recognized entities, or based on historical data

- –USEPA
- –USDOE
- –ASMT
- –NIOSH

20

Limit of Detection (LOD)

- Initial Determinations-labs shall have a procedure
- Establish the LOD by spiking at a concentration greater than or equal to the DL. The LOD is equal to the concentration of the spike
  - –Apparent signal-to-noise shall be at least 3
  - –Results shall meet all identification requirements
  - –Same as M4-Stay tuned!

21

Limit of Detection

Ongoing Verification

- –Required (only) annually
- –Repeat the spike at a concentration
  - •1/2x LOD ≤ Ongoing LOD Spike ≤ 2x LOD
  - •But always ≥ DL
- –Same verification criteria

22

Limit of Quantitation (LOQ)

Initial Determination of the LOQ

- –For methods with multi-level calibration
- –LOQ ≥ LOD and lowest non-zero calibration standard, and;
- –LOQ ≤ 10X LOD

23

Limit of Quantitation

LOQ shall meet same criteria for acceptance as the LOD for:

- –Signal to noise
- –Identification criteria

24

Limit of Quantitation

- Laboratory acceptance criteria for recovery based on 3 SD from mean of historical data
  - –No wider than LCS acceptance criteria plus 20% allowance above and below
  - –Must be greater than 10% recovery
- Laboratory shall verify the LOQ annually, at a minimum

25

Precision and Bias

- The laboratory shall have a procedure for determining precision and bias
- Sample shall be processed through the entire measurement system for each analyte of interest
- Acceptance limits come from, order of preference
  - –The customer
  - –The method
  - –The lab

26

Selectivity

Lab shall evaluate selectivity by following the checks in the method

- –Mass spectral tuning
- –Second column confirmation
- –ICP interelement checks
- –Absorption or fluorescence profiles
- –Etc.

27

Demonstrations of Capability

- Initial and on-going demonstrations of capability are performed in the same manner as in the Chemistry module (M4)

28

Calibration

29

Calibration

- Calibration criteria mostly mirror the criteria in the Chemistry module (M4)
- Some of the criteria may be changes from historical IH practices

30

Calibration Highlights

- Standards used for calibration shall be CRMs from an ISO 17034-accredited RMP, when available
  - –If no such producer in the USA or Canada, standards shall be obtained from an authoritative source
- Initial Calibration Verification (ICV) is required using a standard from a second source
  - –When using neat materials, an independent preparation is acceptable

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Calibration Highlights

For regression or average response/calibration factor calibrations, minimum non-zero standards

Type of Calibration Curve	Minimum Number of Calibration Standards[b,c]
Threshold Testing[a]	1
Average Response	5
Linear Fit	5
Quadratic Fit	6

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Calibration Highlights

- The lowest non-zero standard shall be at or below the LOQ
- The highest calibration standard shall be at or above the highest concentrations to be reported
  - –ICP may report above the highest standard with acceptable analysis of a high-level check standard that exceeds the sample concentration

33

Calibration Highlights

- ICP analyses may be calibrated with a single high point, a zero point and a LOQ check
- Aroclor analyses shall be quantitated from a multi-level calibration
  - –May use 1016/1260 for initial calibration and single points to pattern match other Aroclors
- CCV criteria same as in Chemistry module (M4)

34

Quality Control

35

Desorption Efficiency

- Requirements are still in flux
- Desorption efficiency (DE) is a measure of the recovery of the target analytes from the media used for collection
  - –It is media dependent and can be significantly media lot dependent
  - –It can be concentration dependent

36

Desorption Efficiency

- Best-case scenario: Laboratory has the same lot of media as the customer uses to sample. This happens when:
  - –Everyone in the country has the same lot
  - –Lab has same lot and provides to the customer, or;
  - –Customer provides enough media to perform initial calibration (ICAL)
- Laboratory performs ICAL using media standards

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Desorption Efficiency

- Second best: Laboratory is provided media by sampling team that matches samples
  - –Lab performs spikes to determine desorption efficiency
  - –Sample results are corrected for DE
- Third Best: Laboratory performs ICAL using the same type of media or determines DE but does not know the source or lot of the sampler's media.

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Method Blanks

Most Important: Media blanks take the place of method blanks in many IH analyses

- –Some media types have small amounts of contamination
- –Subtraction shall be applied as described in the reference method and/or the B-Tables
- –Analysis of a Reagent Blank may be indicated

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Method Blanks

Other requirements are as expected

- –One per preparation batch
  - •Unless the method requires additional MB
  - •Maximum batch size may be stated in the method
  - •Otherwise, there is a maximum of 20 field samples
- –Use same or similar matrix/media

40

Laboratory Control Sample

- Shall be spiked as specified by the reference method or requested by the customer
- Otherwise, shall contain all target analytes at a concentration at or below mid-range of calibration
- LCSD is required by the B-Tables, one per preparatory batch

41

Matrix Specific Control

- Matrix Spike
- Matrix Spike Duplicate
- Matrix Duplicate
- Instructions are included for their use along with the caveat they are usually not required in IH methods

42

QC Acceptance Criteria

- The acceptance criteria for IH quality controls are derived in the same manner as the Chemistry acceptance limits
  - Recovery criteria are laboratory developed if project-specific criteria or reference method limits are not specified
  - Duplicate evaluation criteria (RPD) are specified in the B-Tables

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Questions?

Thank you!

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Module 2 Section 7.8 Reporting of Results

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1

Overview

- Radical Difference
- Module 2 Section 7.8
  - Clarifications
  - Eliminated Requirements
  - New Requirements

2

Radical Difference

Appendix A

3

Radical Difference

DON'T PANIC

Clear Expectations	Assessment Easier	Project Specific Needs	Reduce Variability

4

Impact Chart

Laboratories (LAB)	Accreditation Bodies (AB)	Project Team (P) or Customer	Data Validators (DV)

5

Radical Difference

Appendix A

LAB
AB
P
DV

6

Quick Reference

Historical Appendix A	QSM 6.0 Section 7.8
• 1.0 Cover Sheet	• Within 7.8.2.1 of ISO
• 2.0 Table of Contents	• 7.8.2.1.r
• 3.0 Case Narrative	• 7.8.2.1.t
• 4.0 Analytical Results	• 7.8.2.1.hh
• 5.0 Sample Management Records	• Throughout 7.8.2.1
• 6.0 QC Information	• Throughout 7.8.2.1
• 7.0 Data Reports for Third Party Review or Validation	• Removed

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QSM 6.0 Section 7.8

- Implementing 7.8
- Reporting Requirements
- Waiver
- General Requirements 7.8.2.1 of ISO
- Case Narrative 7.8.2.1.t Requirements 1-7
- NEW Precision and Bias 7.8.2.1.u
- NEW Manual Integration 7.8.2.1.v
- NEW Preparation Batch 7.8.2.1.dd
- NEW Sample Result(s) Summary Form 7.8.2.1.hh Requirements 1-17

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Implementation

7.8 Requirement	Clarification
• Implemented in addition to ISO/IEC 17025:2017(E)	• QSM defined minimum requirements for accreditation

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QSM 6.0 Section 7.8

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Reporting Requirements-What

- Section 7.8 represents the minimum requirements to reconstruct a Stage 4 Data Package
- Appendix A Section 7.0 Data Reports describes Stage or Level of a Data Package
  - Removed the Stage descriptions from QSM 6.0
  - Referenced in the DOD EDQW Data Validation Guidelines

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Reporting Requirements-Why

- Section 7.8 requirements are not always necessary
- The Project Team should
  - Determine their minimum reporting requirements
    - Collaborate with their DV
    - Consider the type of project
    - Consult the laboratories during project-planning activities
    - Effectively communicate requirements

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QSM 6.0 Section 7.8

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Waiver

M2 7.1.5.a & 7.8.2.1.x

- Obtained in Writing
  - Technical Point of Contact
- Project-Specific Technical Justification
- Records Maintained
- Included in **ALL** Affected Data Packages

Caveat

- Record
  - Output of Implementation
  - Page(s)
- Approved QAPP is **NOT** a Waiver
- Laboratory Procedure is **NOT** a Waiver

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QSM 6.0 Section 7.8

- Implementing 7.8
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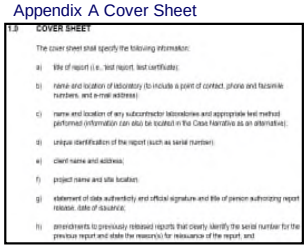
Less Prescriptive

Appendix A Cover Sheet


1.0 COVER SHEET

The cover sheet shall specify the following information:

a) title of report (i.e., test report, test certificate);

b) name and location of laboratory (to include a point of contact, phone and facsimile numbers, and e-mail address);

c) name and location of any subcontractor laboratories and appropriate test method performed (information can also be located in the Case Narrative as an alternative);

d) unique identification of the report (such as serial number);

e) client name and address;

f) project name and site location;

g) statement of data authenticity and official signature and title of person authorizing report release, date of issuance;

h) amendments to previously released reports that clearly identify the serial number for the previous report and state the reason(s) for reissuance of the report; and

Caveat

- Required to be included in the report by 7.8.2.1 of ISO
- Does not have to be on the Cover Sheet

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QSM 6.0 Section 7.8

- Implementing 7.8
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17

Case Narrative

Requirements

- 7.8.2.1.t
- Every Appendix B-Table contains the phrase “explain in the case narrative” at least 10X
- Module 2, 4, 6 & 8

Big Picture

- Useful information to understand data quality
- Unplanned deviations from the method
- Affecting quality of the results
- Sample QC failures

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Explain in the Case Narrative

- Thorough
- Well-Organized
- Communicate Uncertainties
- Identification is not an explanation (e.g., "The LCS exhibited recovery failures")

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Case Narrative Example

- **Issue:**
  - –The LCS had recoveries below the acceptance criteria for target analytes X and Y associated with Batch A.
- **Corrective Action:**
  - –Insufficient sample material to reprepare and analyze all samples associate with Batch A.

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Case Narrative Example Continued

- **Uncertainty or Limitation:**
  - –Samples results associated with low LCS recovery may be biased low.
  - –All samples with non-detects for target analytes X and Y are qualified UJ.
  - –All samples with detects for target analytes X and Y are J qualified.

Sidenote- the proposed QSM does not dictate laboratory qualifiers

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Case Narrative Example Continued

- **Thorough**:
  - –Although the LCS experienced failures, it is important to note the Matrix Spike and Matrix Spike Duplicate recoveries were within acceptance criteria for all target analytes and the precision of all associated samples are unaffected.

Sidenote- Opinions and interpretations expressed in reports shall be contained in the case narrative.

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Still Wondering

Should I include this in my Case Narrative?

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QSM 6.0 Section 7.8

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New Precision and Bias

7.8.2.1.u

- LOQ and associated precision and bias at the LOQ, where the determination of precision & bias at the LOQ is required

Why?

- Understand uncertainty for low-level concentrations of analytes when the LOQ is near the project action level

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Precision and Bias at the LOQ

- Comparing Different Analytical Methods & Laboratories
  - Fair and accurate comparison
  - Especially at low-levels and near the project action level
- Sample Analysis
  - Field samples around the LOQ
  - Informed decisions for those results

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Precision and Bias at the LOQ

- Confident Decision Making
  - Understanding uncertainty around the LOQ
    - Project-specific criteria like MS/MSD at the mid-range
    - LCS and CCV batch-specific at higher concentrations
- Understand Uncertainties Risk Assessment
  - Target analytes present at the LOQ
  - Impacts to the environment

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QSM 6.0 Section 7.8

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New Manual Integration

- 7.8.2.1.t.7 occurrence of analytes for which manual integration occurred shall be included in the **case narrative**.
- 7.8.2.1.v before and after chromatographs of analytes for which manual integration occurred including the **justification for the change**

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Provide Justification

Justification

- Thorough
- Organized
- Communicate Uncertainties
- Identification is not justification (e.g., "Split Peak")

Why?

- Manual integration can
  - Introduce subjectivity
  - Variability between analyst
  - Increase risk of error
  - Add validation challenges

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DV

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QSM 6.0 Section 7.8

- Implementing 7.8
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New Preparation Batch

7.8.2.1.dd

- Sample preparation records, including start time and date of the first and last sample;

Why?

- Definitions are requirements
  - **preparation batch** …. a maximum time between the start of processing of the first and last sample in the batch to be 24 hours.

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QSM 6.0 Section 7.8

- Implementing 7.8
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Sample Result(s) Summary Form

7.8.2.1.hh

- A sample result(s) summary form for each field sample reported by the laboratory that includes the following information;

Caveat

- Does not have to be on a single page
- Not dictating format or terminology

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Sample Result(s) Summary Form

- 7.8.2.1.hh.1 Field sample identification as written on custody form
  - Customer ID, Client ID, Description, etc.
- 7.8.2.1.hh.12 sample-specific factors (e.g., sample aliquot or weight of soil/sediment, final extraction volume, dilution factor, and percent moisture or percent solids);
  - Anywhere on the Form
  - Applicable to the analysis or N/A

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Sample Result(s) Summary Form

7.8.2.1.hh.5

- Date and time sample collected if the laboratory performs sampling or ***if provided by the customer.***

Historic Appendix A

- Date and time sample collected.

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DV

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QSM 5.4 Scenario

- Historically
  - If sample collection time was not provided, the laboratory would have to assume the most conservative time (12:00 a.m.).
- Impact
  - Inaccurate hold time calculated
  - Data unnecessarily qualified

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QSM 6.0 Scenario

- Proposed
  - If sample collection time was not provided, the laboratory has two options (M2 7.7.10).
- Option 1: Laboratory is encouraged to contact the customer
  - 7.8.2.1.bb records of communication shall be included in the report
- Option 2: Laboratory does not contact customer or unknown
  - Internal HT will be set at 12:00 a.m.
    - Laboratory does not report time
    - DV cannot verify without field records

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Sample Result(s) Summary Form

7.8.2.1.hh.9

- Method identification for all preparation, cleanup, and analytical methods including the version number.

Impact

- Compliance
- Traceability
  - Laboratory
  - DOD and DOE

LAB
AB
P
DV

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Sample Result(s) Summary Form

7.8.2.1.hh.16

- The result for each target analyte from the lowest dilution that met all QC acceptance criteria.

Caveat

- Does not have to be on a single page
- Can be a single or multiple summary form

LAB
AB
P
DV

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Lowest Dilution

Single Form

- Target Analyte
  - A[1]
  - B[2]
  - C
  - D

Results reported from 1X except:

- (1) Reported from the 5X Dilution
- (2) Reported from the 10X Dilution

Multiple Forms

- Target Analyte 1X DF
  - C
  - D
- Target Analyte 5X DF
  - A
- Target Analyte 10X DF
  - B

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Thank you

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*Or like I tell my family; I am The Ruler of QAOS!

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DETECTION AND QUANTITATION: A HITCHHIKER'S GUIDE

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Just Remember:

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Big Picture

- Why do we have detection and quantitation limits?
  - Where can we be sure an analyte is present?
  - Where can we be sure an analyte is absent?
  - Where can we be sure how much analyte is there?
- Ultimately, we are determining how our data may or may not be appropriate for use.

"The story so far:

In the beginning the [LOD] was created.

This has made a lot of people very angry and been widely regarded as a bad move."

— Douglas Adams, The Restaurant at the End of the Universe

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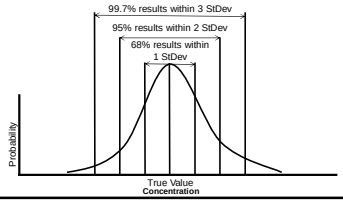
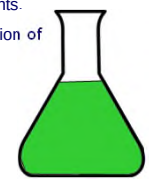
A Quick Review (oh no, not again)

- Remember, the result (data) obtained by the laboratory should really be considered one point from a distribution of points.
- A probability distribution is a plot of the relative distribution of those results.





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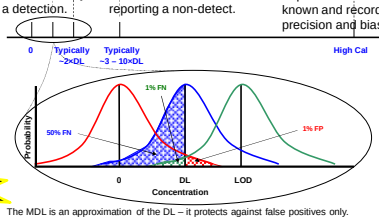
Defining Limits

Detection Limit (DL): False positive rate (Type I error) is 1%. Lowest concentration for reliably reporting a detection.

Limit of Detection (LOD): False negative rate (Type II error) is 1%. Lowest concentration for reliable reporting a non-detect.

Limit of Quantitation (LOQ): The smallest concentration that produces a quantitative result with known and recorded precision and bias.



What about MDL?

The MDL is an approximation of the DL – it protects against false positives only.

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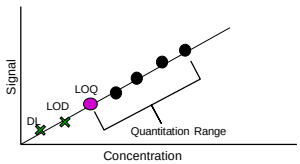
Defining Limits

- Quantitation Range: The range of values (concentrations) in a calibration curve between the LOQ and the highest successfully analyzed initial calibration standard. The quantitation range lies within the calibration range.



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QSM 6 – What is changing?

- The overall scheme is much the same
- Changes were made to:
  - Align with other published requirements and guidelines
  - Reduce the overhead burden on laboratories
  - Maintain a high data quality standard

We are Here

"I've come up with a set of rules that describe our reactions to technologies:

1. Anything that is in the world when you're born is normal and ordinary and is just a natural part of the way the world works.

2. Anything that's invented between when you're fifteen and thirty-five is new and exciting and revolutionary and you can probably get a career in it.

3. Anything invented after you're thirty-five is against the natural order of things."

— Douglas Adams, The Salmon of Doubt

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Side-By-Side – DL

	QSM 5.4	QSM 6
Frequency	• Each matrix-method-analyte combination • Initially • Whenever there is a change in the method	• Each matrix-method-analyte combination • Compliant with EPA MDL revision 2
Procedure	• The EPA MDL procedure is one example of an approach	• Compliant with EPA MDL revision 2
Acceptance Criteria	• Meet requirement for false positive rate (1%)	• As stated in EPA MDL revision 2 • Estimates a 1% false positive rate

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Side-By-Side – LOD

	QSM 5.4	QSM 6
Frequency	• Initial and quarterly verifications • Per batch basis for infrequently used methods	• Initially determined • Ongoing verifications quarterly • When method altered • Per batch basis for infrequently used methods
Procedure	• Spike LOD at 2-4x DL • Spike sets LOD until next spike	• Initial spike – sets LOD • Ongoing spikes ½ to 2X initial LOD (but ≥ DL)
Acceptance Criteria	• S/N ratio >3 • Meet all requirements for identification	• S/N ≥ 3 or Signal > MDLb (3 StDev above Method Blank) • Meet all requirements for identification

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Side-By-Side – LOQ

	QSM 5.4	QSM 6
Frequency	• Quarterly verifications • Per batch basis for infrequently used methods	• Verification quarterly at ½ to 2X LOQ (but ≥ LOD) • Per batch basis for infrequently used methods
Procedure	• Within calibration range, no lower than lowest calibration point • LOQ > DL, should be LOQ > LOD • Empirically demonstrate Precision and Bias	• Select LOQ such that: LOD ≤ LOQ ≤ Lowest Calibration Standard (or check standard) • Empirically demonstrate Precision and Bias
Acceptance Criteria	• Precision and bias meet client requirements	• S/N ≥ 3 or Signal > MDLb (3 StDev above Method Blank) • Fall within 3 StDev of mean historical data • No wider than LCS criteria ± 20% • ≥ 10% on lower end • Precision and bias meet client requirements

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Side-By-Side – Qualification of Data

Unchanged

- Results below the DL are reported as LOD U or < LOD
- Results between the DL and the LOQ are reported with an estimated "J" qualifier
- Results above the LOQ, up to the high end of the calibration range, are considered quantitative
- Precision and Bias at the LOQ must be determined

Changed

- Precision and Bias at the LOQ shall now be reported in every data package

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Question and Answer Time

- Wait, can the DL = LOD now?
- Why can I spike at ½ to 2X the values for verification?
- What does a detection and quantitation data package look like?
- I'm not a laboratory… what does this all mean for me?

"I refuse to answer that question on the grounds that I don't know the answer"
— Douglas Adams

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Can DL = LOD?

- Yes, if:
  - All requirements for LOD verification are met with spike at DL
- No, if:
  - Any requirement for LOD verification is not met with a spike at DL

Reminder of LOD Requirements:
Shall have S/N ≥ 3 or Signal > MDLb (3 StDev above Method Blank) and shall meet all requirements for identification

- Scenario 1:
  - All requirements are met
  - Spike at DL (MDL) produces signal meeting requirements of LOD
- Scenario 2:
  - All requirements are not met
  - Spike at DL (MDL) did not produce a signal meeting the requirements of LOD

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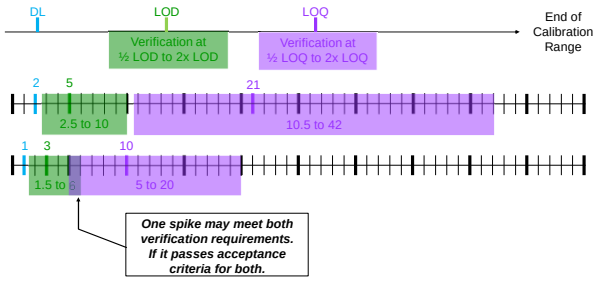
Verification Spikes ½ to 2x Limit



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Summary Package Requirements

- Who might request this package and why?
  - The AB to verify conformance with the QSM
  - A project to evaluate whether data are likely to meet project-specific DQOs for sensitivity
- Requirements specified in M4, Section 5.2.5

5.2.5 The laboratory shall provide the following when DL, LOD, and LOQ summary information is requested:
5.2.5.a indication of which analyte/matrix/prep method/analytical method and instrument used;
5.2.5.b DL;
5.2.5.c claimed LOD;
5.2.5.d concentration of initial LOD spike and verification spike, if different;
5.2.5.e statement of compliance with analyte identification requirements;
5.2.5.f signal-to-noise value or statement of compliance with requirements;
5.2.5.g claimed LOQ;
5.2.5.h concentration of LOQ spike;
5.2.5.i recovery or result of LOQ spike;
5.2.5.j calculated precision and bias at the LOQ that incorporates historic and current data;
5.2.5.k accuracy acceptance criteria at the LOQ;
5.2.5.l description of precision and bias calculations; and
5.2.5.m if specifically requested, raw data to support parameters reported.

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Project Team Consideration

- How does this change things for our DoD Project Teams?
  - The good news: not much changes!
  - The bad news: you still need to think about things.

"I'd take the awe of understanding over the awe of ignorance any day."

— Douglas Adams, The Salmon of Doubt

"You live and learn. At any rate, you live."

— Douglas Adams, Mostly Harmless

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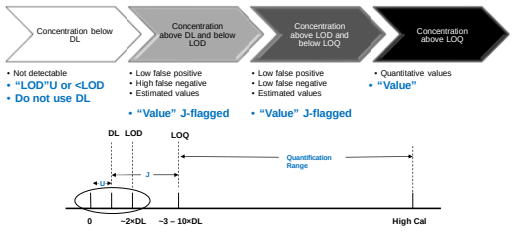
Data Reporting and Quality

- How will data be reported and qualified around these limits?



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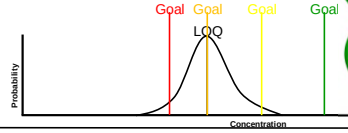
Should Project Goals = LOQ?

- Why Not?
  - Precision and bias are only defined at and above the LOQ
  - The laboratory's DL, LOD, and LOQ are typically defined in a clean matrix, and so are a lowest possible value.
  - The laboratory LOQ does not take into consideration any of the uncertainty derived from sampling procedures.
  - Remember, data is a distribution. Consider tolerable decision error along with measurement error when determining project goals.



Recommendation:
Projects should aim for a laboratory with LOQs 3 to 10 times below action or screening limits for analytes of concern.

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What Should Project Chemists Do?

- Review the limits of the laboratory analysis with respect to Project Action Limits (PALs) or Project Screening Limits (PSLs)
- Understand the inherent limitation of the selected analytical methods
- Consider potential or known matrix effects. Discuss these with the laboratory beforehand, if known.

LOQ << PALs	LOQ ~ PALs	LOQ >> PAL
OK – Sensitivity of analysis will likely meet project DQOs	Caution - Sensitivity of analysis may or may not meet project DQOs	STOP – Sensitivity of analysis will likely not meet project DQOs
Continue to plan project as previously	Evaluate DL, LOD, and LOQ summary information from laboratory and review for project requirements	Evaluate alternative analyses or revisit DQOs – consult appropriate project team members

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So long, and thanks for…

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"I may not have gone where I intended to go, but I think I have ended up where I needed to be."

— Douglas Adams, The Long Dark Tea-Time of the Soul

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The following are backup slides –

not to be presented

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Examples

Detected Value	DL	LOD	LOQ	Reported Value
20	1	2	10	20
9	1	2	10	9 J
1.5	1	2	10	1.5 J
0.5	1	2	10	2 U

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