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CRMs and How They Relate to ISO 17025 Accreditation Outcomes

Agenda

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- Specific Technical Requirements of ISO 17025 (Section 5)
- How CRMs Help Meet ISO 17025 Technical Requirements
- Basics of Quality Control
- Use of CRMs in Quality Control

Requirements

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ISO 17025 requires laboratories to:

- Establish, operate, and maintain a quality system consistent with ISO 9001
- Be technically competent to operate the test systems for which they are accredited
- Be able to generate technically valid results for the test systems for which they are accredited

Personnel

- 5.2.1 - Laboratory management shall ensure the competence of all who operate specific equipment, perform tests and/or calibrations, evaluate results, and sign test reports and calibration certificates.
  - NOTE 1 – In some technical areas it may be required that the personnel performing certain tasks hold personnel certification. The requirements for personnel certification may be regulatory or required by the customer.
- 5.2.2 – The management of the laboratory shall formulate the goals with respect to education, training, and skills of the laboratory personnel. The effectiveness of the training actions shall be evaluated.

Methods and Method Validation

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- 5.4.1 – The laboratory shall use appropriate methods and procedures for all tests and/or calibrations within its scope. This includes, where appropriate, an estimation of the measurement uncertainty.
- 5.4.4 – Non standard methods shall be validated appropriately before use. Procedures for non-standard methods should include reference standards and reference materials required; checks that the equipment is working properly, calibration and adjustment of the equipment; uncertainty or procedure for estimating uncertainty.
- 5.4.5.2 – The techniques used for the determination of the performance of a method should be one or a combination of; calibration using reference standards, interlaboraotry comparisons, assessment of the uncertainty of results.

Estimation of Uncertainty of Measurement

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- 5.4.6.2 – Testing laboratories shall have and apply procedures for estimating uncertainty of measurement.
- 5.4.6.3 – When estimating the uncertainty of measurement, all uncertainty components which are of importance shall be taken into account.

Equipment

- 5.5.2 – Equipment used for testing shall be capable of achieving the accuracy required. Before being placed into use equipment shall be calibrated and checked.
- 5.5.3 – Equipment shall be operated by authorized personnel.

Measurement Traceability

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- 5.6.2.1.1 – Calibrations and measurements made by the laboratory must be traceable to SI Units.
- 5.6.2.1.2 – Certain calibrations/measurements cannot be strictly made in SI Units. Confidence in measurements in these cases can be made with the use of Certified Reference Materials provided by a competent supplier.
  - Participation in a suitable program of interlaboratory comparisons is required where possible.

Reference Standards and Reference Materials

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- 5.6.3.1 – Reference standards shall be calibrated by a body that can provide traceability as described in 5.6.2.1
- 5.6.3.2 – Reference materials shall, where possible, be traceable to SI units of measurement, or to Certified Reference Materials.

Assuring the Quality of Test Results

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- 5.9.1 – The laboratory shall have quality control procedures for monitoring the validity of tests undertaken. The resulting data shall be recorded so trends are detectable. The monitoring may include
  - Regular use of Certified Reference Materials.
  - Participation in Proficiency Testing Programs

WHAT DOES ALL THIS MEAN TO ME

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TRACEABILITY

QUALITY CONTROL

Traceability

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ISO 17025 Requires Traceability

Traceability is a property of the result of a measurement or the value of a standard where by it can be related to the stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.

Traceability/Quality Control

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ISO 17025 laboratories need to:

- Use traceable calibration standards whenever they are available.
- If traceable standards are not available, an ISO Guide 34 accredited producer of calibration materials should be selected to assure the best possible standards
- Verify calibrations against independent quality control standards from a ISO Guide 34 accredited supplier

Traceability/Quality Control

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- I do not have the capability or adequate resources to produce my own traceable quality control standards . What can I do?

Use Certified Reference Materials

Certified Reference Materials (CRMs)

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- A Certified Reference Material is defined in ISO Guide 34 as a reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of a specified property, its associated uncertainty, and a statement of metrological traceability.
- CRM producers go through rigorous quality control requirements per ISO Guide 34 in order to be accredited.

What are CRMs: Characteristics

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- Characteristics of Certified Reference Materials:
  - A standard with known concentrations (assigned values) of specified analytes
  - The standard has a known uncertainty, homogeneity and stability
  - The assigned values are traceable to an independent reference
  - A certificate of analysis is included with the CRM
  - The certificate of analysis includes reference to the characteristics of the certified reference materials

Certificate of Analysis

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Reference Materials

▪ Certificate of Analysis ▪

Product: WatR™ Pollution Demand
Catalog Number: 516
Lot No. P222-516
Certificate Issue Date: November 13, 2013
Expiration Date: October 31, 2016
Revision Number: Original

CERTIFICATION

Parameter	Certified Value[1]	Uncertainty[2]	QC Performance Acceptance Limits[3]	PT Performance Acceptance Limits[4]
	mg/L	%	mg/L	mg/L
BOD	122	0.412	81.5 - 163	66.0 - 179
CBOD	111	0.412	72.1 - 161	51.9 - 170
COD	199	0.434	162 - 229	161 - 230
TOC	78.7	3.30	69.0 - 88.1	66.0 - 90.5

ANALYTICAL VERIFICATION

Parameter	Certified Value[1]	Proficiency Testing Study			NIST Traceability	
		Mean	Recovery[5]	n	SRM Number	Recovery
	mg/L	mg/L	%			%
BOD	122	123	101	261	-	-
CBOD	111	114	102	224	-	-
COD	199	199	99.9	221	917a	103
TOC	78.7	78.6	99.9	155	185h	94.0

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ERA
A Waters Company

Reference Materials

▪ Certificate of Analysis ▪

1. The **Certified Values** are the actual "made-to" concentrations confirmed by ERA analytical verification. The certified values for BOD and CBOD are the predicted interlaboratory means calculated from the USEPA/NELAC regression equations used for evaluating proficiency testing study data. The certified values are monitored and purchasers will be notified of any significant changes resulting in recertification or withdrawal of this certified reference material during the period of validity of this certificate.

2. The stated **Uncertainty** is the total propagated uncertainty at the 95% confidence interval. The uncertainty is based on the preparation and internal analytical verification of the product by ERA, multiplied by a coverage factor. The uncertainty applies to the product as supplied and does not take into account any required or optional dilution and/or preparations the laboratory may perform while using this product.

3. The **QC Performance Acceptance Limits (QC PALs™)** are based on actual historical data collected in ERA's Proficiency Testing program. The QC PALs™ reflect any inherent biases in the methods used to establish the limits and closely approximate a 95% confidence interval of the performance that experienced laboratories should achieve using accepted environmental methods. Use the QC PALs™ to realistically evaluate your performance against your peers.

4. The **PT Performance Acceptance Limits (PT PALs™)** are calculated using the regression equations and fixed acceptance criteria specified in the NELAC proficiency testing requirements. Use the PT PALs™ when analyzing this QC standard alongside USEPA and NELAC compliant PT standards. Please note that many PT study acceptance limits are concentration dependent (some non-linearly) and, therefore, the acceptance limits of this QC standard and any PT standard may differ relative to their difference in concentrations.

5. The **PT Data/Traceability** data include the mean value, percent recovery and number of data points reported by the laboratories in our Proficiency Testing study compared to the Certified Values. In addition, where NIST Standard Reference Materials (SRMs) are available, each analyte has been analytically traced to the NIST SRM listed. This product is traceable to the lot numbers of its starting materials. All gravimetric and volumetric measurements related to its manufacture are traceable to NIST through an unbroken chain of comparisons.
Traceability Recovery (%) = [(% recovery certified standard)/(% recovery NIST SRM)]*100
The traceability data shown were compiled by analyzing the ERA standards or their associated stock solutions against the applicable NIST SRMs.

6. For additional information on this product such as intended use, instructions for use, level of homogeneity, and safety information, please refer to the provided Instruction Sheet

If you have any questions or need technical assistance, please call ERA technical assistance at 1-800-372-0122 or send an email to info@eraqc.com.

Certifying Officer
Tom Widera

Quality Officer
Kristina Sanchez

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Types of CRMs

- Calibration standards
  - Individual, high concentration solutions
  - Multi-analyte, high concentration mixtures
  - Low concentration, ready-to-use, solutions
- Calibration check standards
  - Simple matrix
  - Similar to calibration standards
- Matrix quality control standards
  - Real or synthetic matrix
  - Various analytes, various concentrations
  - May include interferences
  - Designed to mimic a real sample

Use of CRMs:
Basics of Quality Control

Basics of Quality Control

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- Quality control generally applies to four basic functions of the laboratory
  - Method development
  - Staff training
  - Ongoing performance
  - Corrective action

Method Development

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- Precision
  - Run several times in a row to determine repeatability
  - Run several times over days or weeks to determine reproducibility
- Accuracy
  - Analyze single-blind samples
    - CRMs or PTs
    - Best to use real matrix samples
- Detection Limit
  - How low can you accurately measure?
    - Analysis of blanks
- Reporting Limit
  - How low should you report?
    - Stock CRMs
    - Diluted CRMs
    - Custom-made standards
- Method ruggedness
  - Analyze QC samples
    - Controlled conditions
    - Variable conditions
    - Imagine as many variables that could change, and test them
    - Record variations and outcomes

Basics of Quality Control

Staff Training

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- When hiring new staff or giving them a new method to perform, it is important to train them and ensure training was effective.
  - Use CRMs as hands-on training samples
  - Document the analyst's performance versus the assigned value
  - Proof of competency to perform the analysis

- It is also important to regularly document that the analyst is still performing with the necessary accuracy and precision
  - Ongoing performance verification

Staff Training

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- Gaining experience
  - Analyze samples under direct supervision
    - Real world samples
    - Certified reference materials
  - First analysis by themselves
    - Include extra QC
- Ongoing performance verification
  - Review of standard method QC (CRMs)
  - Periodic single-blind QC (lab knows it is a test)
    - PT study for each analyst
  - Inclusion of double-blind PT (lab does not know it is a test)
    - Provides a better test of the analyst's performance

Basics of Quality Control

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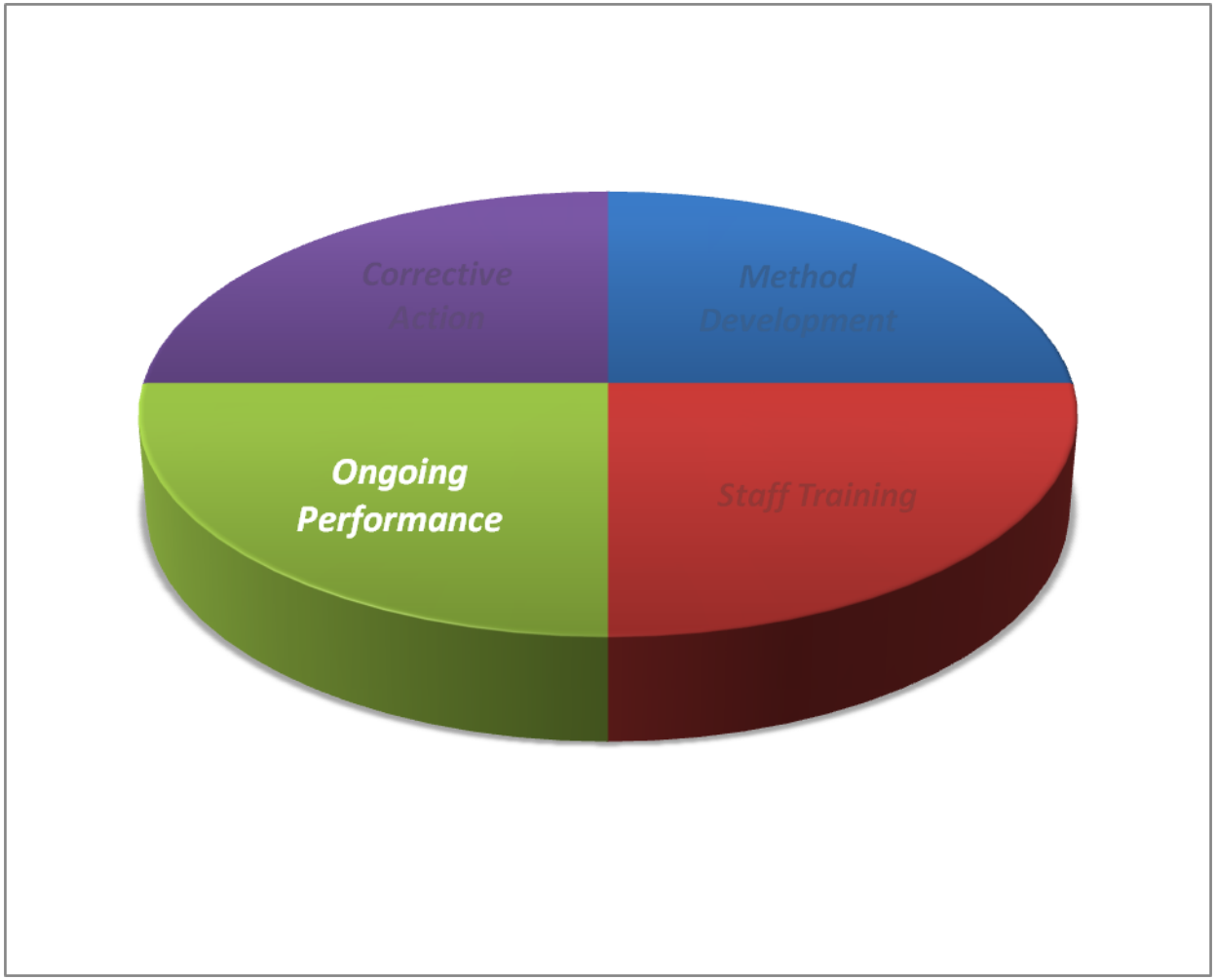

Ongoing Performance

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- Once a method is established in a laboratory, there are still many variables that can affect its performance:
  - Analysts
  - Instruments
  - Standards and reagents
  - Laboratory environment
  - Sample matrix
  - Random errors

Ongoing Performance

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- Routine QC
  - Calibration
    - Fresh
    - Trusted source
    - Traceable
  - CCV: continuing calibration verification
    - Same source as calibration
    - Checking for short-term drift
  - Linearity check
  - ICV: independent calibration verification
    - Independent source
      - Certified reference material
    - Generally mid-calibration range
    - Control chart the results of ICV analysis

Ongoing Performance

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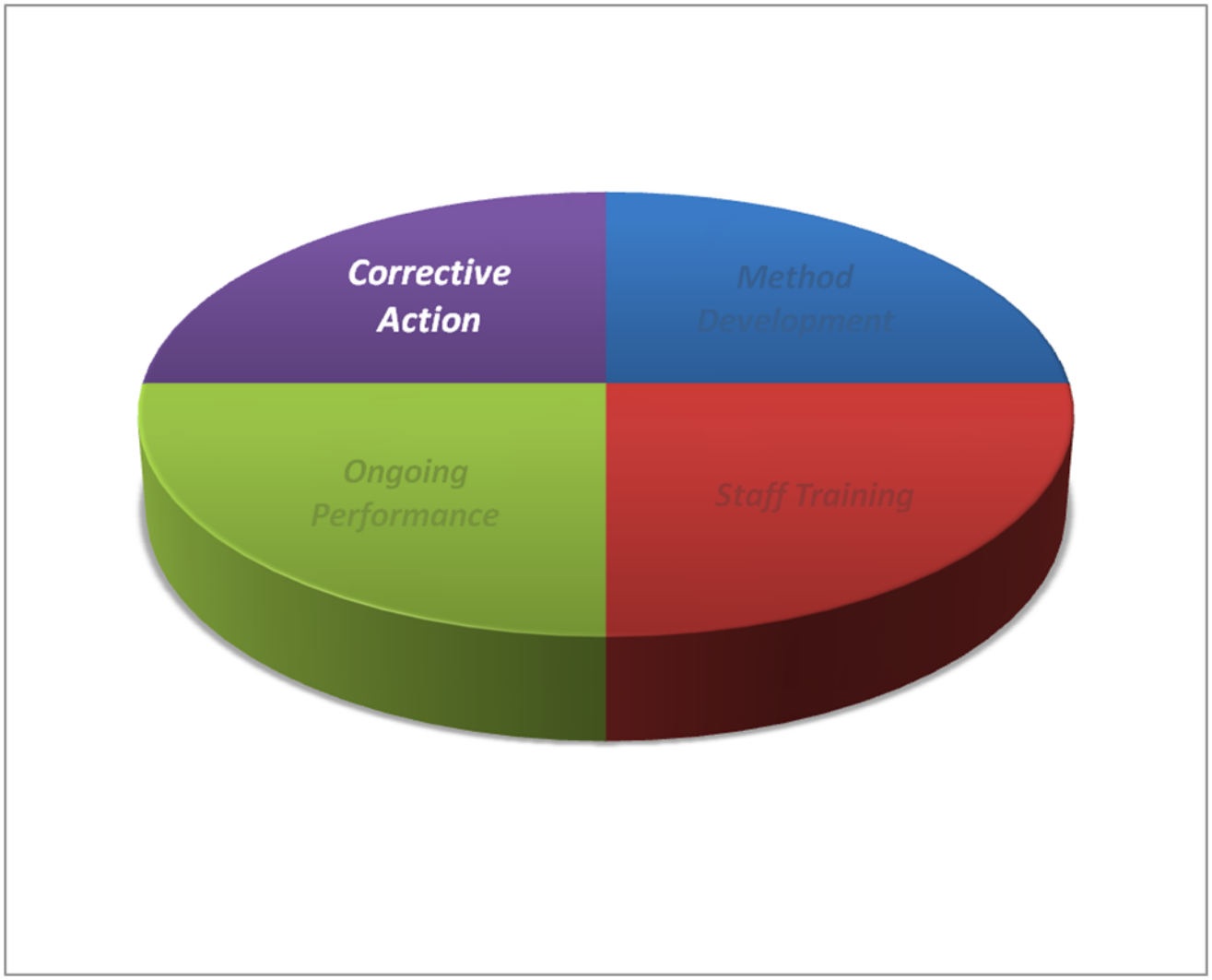
- Control charting is the process of plotting the percent recovery of a certified reference material, each time it is analyzed
- Ideally, the CRM is analyzed with each analytical batch, or at least once each day that the analysis is performed
- When possible, use the same lot number of CRM for your control charting for as long as possible
  - This ensures that any potential variations between CRMs are not affecting your control charts
  - If you consistently use CRMs from the same, accredited provider, you should not experience lot-to-lot variations

Basics of Quality Control



Corrective Action

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- Whenever quality criteria are not met:
  - Failed PT sample
  - Calibration linear regression does not fit
  - Outside of quality control limits
    - Control chart

- Corrective action:
  - Root cause analysis
  - Implement corrections
  - Test that corrections are appropriate
  - Monitor to ensure that corrections are effective

Corrective Action

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- CRMs are effective when conducting root cause analysis:
  - Run using same conditions as when problem identified
  - Make minor adjustments if necessary
  - Use to identify the source of the error (Man, Machine, Materials, Methods)
    - Analyst
    - Instrument
    - Standards
    - Procedure

Corrective Action

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- Once the problem is identified and a change has been implemented, CRMs are helpful to ensure effectiveness:
  - Was change appropriate
    - Verify new calibration standard, give QC standard to retrained analyst, etc.
  - Is the change effective long term
    - Routine use of CRMs will identify if the system is in control and may predict when it is trending out of control

Conclusion

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- CRMs can be used to satisfy all the technical requirements for ISO 17025 Accreditation.
- CRMs are critical in establishing and maintaining a quality control program. By incorporating CRMs into a QC program regularly, a lab can help ensure both reliability of results and successful PT performance.
- CRMs are certified by an outside source, giving you added confidence that your standard is made correctly.

THANK YOU