QMS Quick Learning Activity

Test Methods, Validation, & Verification

ISO/IEC 17025:2017

www.aphl.org

Objectives: At the end of this course participants will be able to…

- Understand that only the most current, approved and appropriate method should be used.
- Comprehend the requirements for validating a non-standard or in-house method.

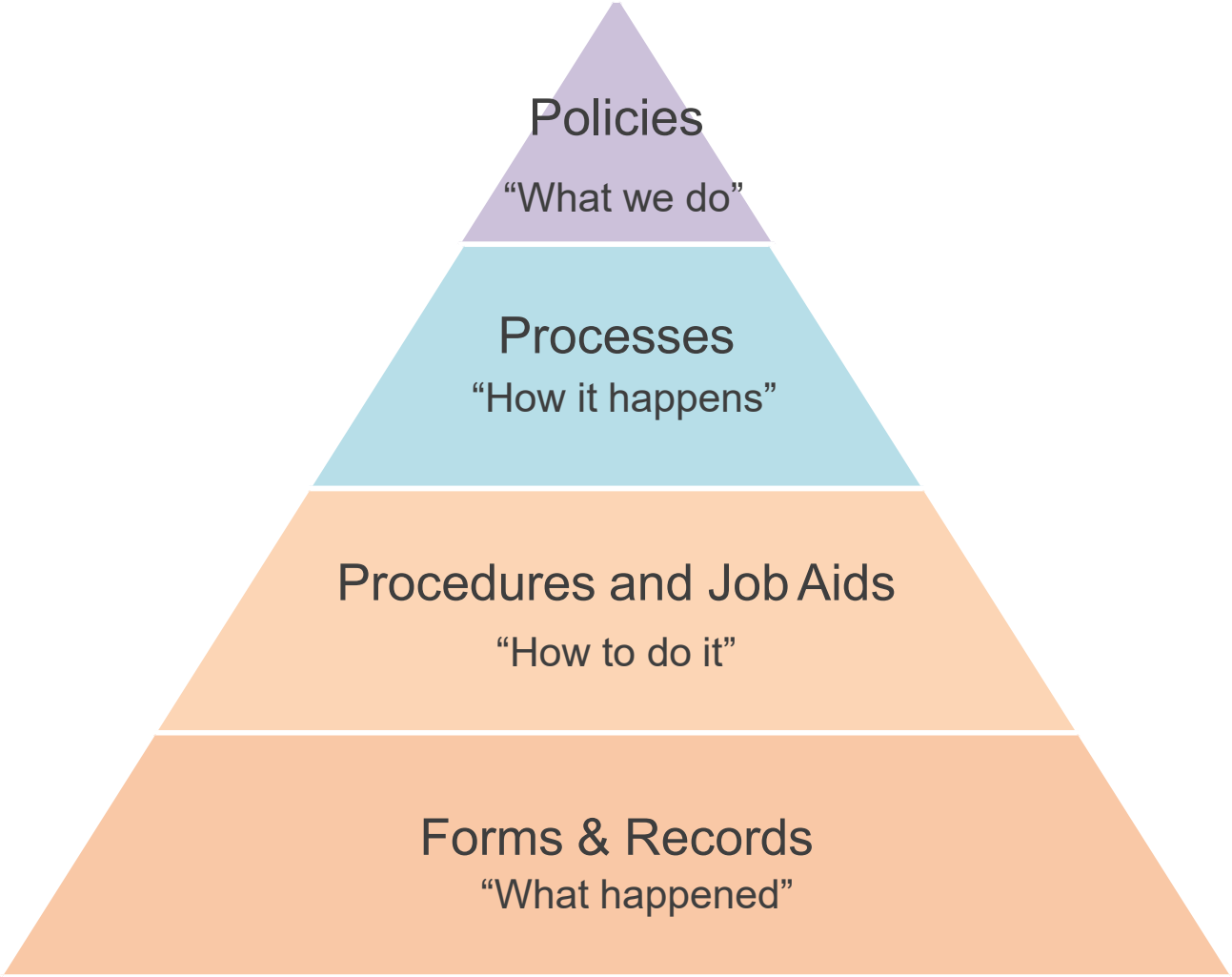
Abbreviations & Acronyms

- **Accuracy**-(Method Bias) nearness to the true value; can be determined by one of the following techniques:
  - Use of certified reference materials;
  - Use of reference method of known uncertainty
  - Use of recovery from spiked samples
  - Reference material, if available, should be carried through entire procedure with each batch of test samples. If fortified or spiked samples are used, the method of fortification should be described.
- **Bias**-systematic error manifested as a consistent unidirectional deviation from the true value
- **Reference Material-**sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties
- **Certified Reference Material** (CRM)-reference material that has documentation by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities

Abbreviations & Acronyms

- **Calibration-**the comparison of two measurement devices or systems (one of known uncertainty [your standard] and one of unknown uncertainty [your test equipment])
- **Laboratory developed methods-**methods that are developed using “in-house” procedures, or by changing an established reference procedure.
- **Reference method**-methods that have been published or documented by an authoritative body
- **SOP**-Standard Operating Procedure
- **Validation**-the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled
- **Verification**-the laboratory verifies, by an established process, that it is capable of using a validated method for its intended use

Some distinctions



Methods

7.2.1.1 “The laboratory shall use appropriate methods and procedures for all ~~laboratory~~ activities…”.
In addition to testing, this includes:

- Sampling
- Handling
- Transport
- Storage
- Sample Preparation
- Estimation of measurement uncertainty
- Statistical techniques for analysis

When Selecting a Method

Unless specified by a customer, the method should be approved or published in

- International, regional or national standards
- Reputable technical organizations
- Relevant scientific texts or journals
- Specified by manufacturer of equipment

Use the latest valid edition unless it is not appropriate or possible to do so

- Supplement with additional details when necessary to ensure consistent application
- All methods, procedures and supporting documentation shall be kept up to date and readily available

When Selecting a Method

Deviations shall occur only if the deviation has been

- Documented
- Technically justified
- Authorized, and accepted by the customer

Appropriate for the test

Laboratory must verify that it can properly operate the method before introducing the test. If the method changes, the verification must be repeated.

Laboratory Developed Methods

Must be appropriate for intended use

Customer must be informed

Laboratory shall validate these methods

- Non-standard
- Laboratory designed/developed
- Standard methods outside their intended scope or modified

Method is validated:

- Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
- It is a planned activity
- It is assigned to qualified personnel with adequate resources
- Follow your laboratory's *Validation and Verification SOP* (standard operating procedure)
- Record the results obtained, the procedure for the validation, and a statement as to whether the method is fit for the intended use
- Includes processes for sampling, handling, and transportation

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Validation

Techniques used should include one or more of the following:

Calibration using reference standards or materials

Comparison of results with other methods; testing method robustness through variation of controlled parameters

Interlaboratory comparisons

Systematic assessment of the factors influencing the result

Assessment of the uncertainty of the results based on understanding of the theoretical principles of the method and practical experience performing the method

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New Test Methods Should Contain:

- Appropriate identification
- Scope
- Description of type of item to be tested (e.g. matrices)
- Parameters of quantities and ranges to be determined
- Apparatus and equipment, including technical performance requirements
- Reference standards and reference materials required
- Environmental conditions required and any stabilization period needed

New Test Methods Should Contain:

Description of procedure

- Identification, handling, transporting, storage, and preparation of items
- Checks to be made before work is started
- Checks that equipment is working properly, required calibration and adjustments are made prior to each use
- Method of recording observations and results
- Safety measures to be observed

Criteria and requirements for approval/rejection

Data to be recorded and method of analysis and presentation

Uncertainty or the procedure for estimating uncertainty

Validation

Range and accuracy of the values obtained should include:

- Uncertainty of the results
- Detection limit
- Selectivity of the method
- Linearity
- Limit of repeatability and/or reproducibility
- Robustness against external influences and/or cross-sensitivity against interference from the sample matrix
- Assessed for the intended use
- Relevance to the customer

Validation, continued

When changes are made to a validated method, the influence of the changes should be documented, and if appropriate, a new validation carried out

These items shall be relevant to the customer's needs:

- Range and accuracy of the values (uncertainty)
- Detection limit
- Selectivity of the method
- Linearity
- Limit of repeatability and/or reproducibility
- Robustness against external influences
- And/or cross-sensitivity against interferences from the matrix of the sample

Validating Data/Software

- Calculations (manual and electronic) and data transfers shall be subject to appropriate checks in a systematic manner
- When computers or automated equipment are used for the acquisition, processing, recording, reporting, storage, or retrieval of data the laboratory shall ensure that:
  - Computer software developed by the user is documented in sufficient detail and is suitably validated as being adequate for use
    - Excel spreadsheets with calculations only (documents)—Lock the formula cells
    - Excel spreadsheets with results after calculations (records)—pdf the record so no changes can be made
  - Procedures are established and implemented for protection of data (integrity and confidentiality, storage, transmission, and processing)
    - Commercial software (e.g., word processing, database, statistical programming) is considered sufficiently validated
    - Laboratory software and modifications to commercial software must be validated before use

After review and authorization, results must be reported:

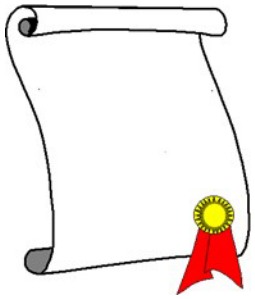
- Accurately
- Clearly
- Unambiguously
- Objectively
- Usually in a report
- With information requested by customer & necessary to interpretation
- All information required by the method used

Unless agreed upon with the customer for a simplified result, test reports shall contain:

- Title, e.g., “Test Report”
- Name and address of laboratory
- Unique identification of the test report and clear numbering system, e.g., page 1 of 3
- Name and contact information of the customer
- Identification of method used
- Description, condition, and identification of item being tested
- Date of receipt of test item
- Date test was performed
- Date report was issued
- Reference to sampling plan/procedures used, where relevant
- Statement that results relate only to the items tested
- Test results with units of measurement, where appropriate
- Additions to, deviations, or exclusions from the method
- Name, function, and signature of person(s) authorizing the test report
- Clear identification when results are from external providers
- With a simplified report, all the above information must be readily available.

Test reports shall contain, where necessary to interpretation of results:

- Information on specific test conditions, such as environmental conditions
- A statement of conformity with requirements and/or specifications, where relevant
- Opinions and interpretations, where appropriate
- A statement on the estimated uncertainty of measurement
  - Where relevant to validity or application of test results
  - When a customer requires it
  - When uncertainty affects compliance to a specification limit
- Additional information that may be required by the method or customers



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References

- ISO/IEC 17025:2017
  - https://www.iso.org/standard/66912.html