



QMS Quick Learning Activity

Test Methods, Validation & Verification

ISO/IEC 17025:2005

Objectives; At the end of this course participants will be able to...

- Participant will understand that only the most current, approved and appropriate method should be used.
- Participant will 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
- **Calibration**-the comparison of two measurement devices or systems; one of known uncertainty (your standard) and one of unknown uncertainty (your test equipment)
- **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
- **Laboratory developed methods**-methods that are developed using “in-house” procedures, or by changing an established reference procedure.

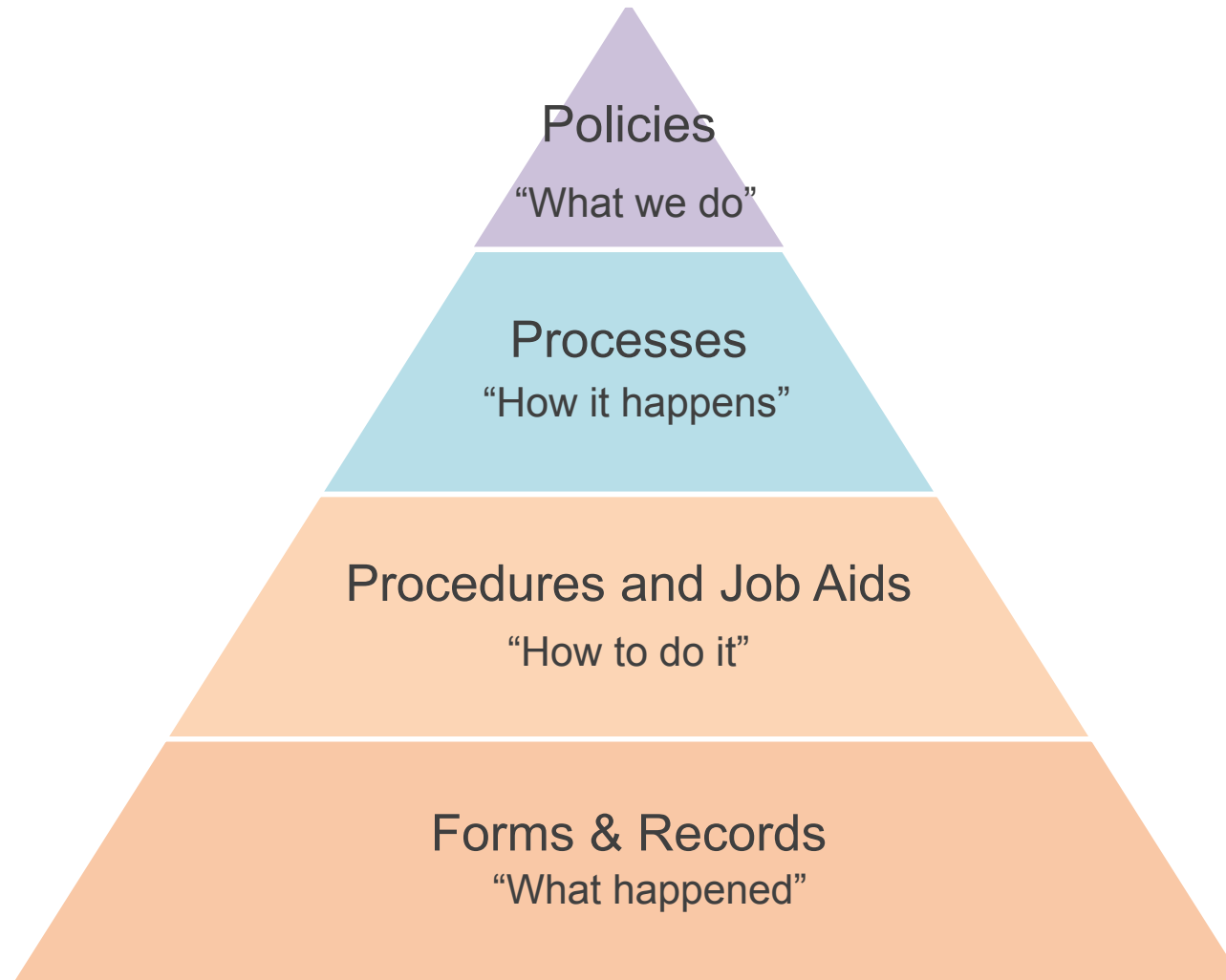


Abbreviations & Acronyms

- **Reference method**-methods that have been published or documented by an authorizing 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**-laboratory verifies by an established process that they are capable of using a validated method for its intended use

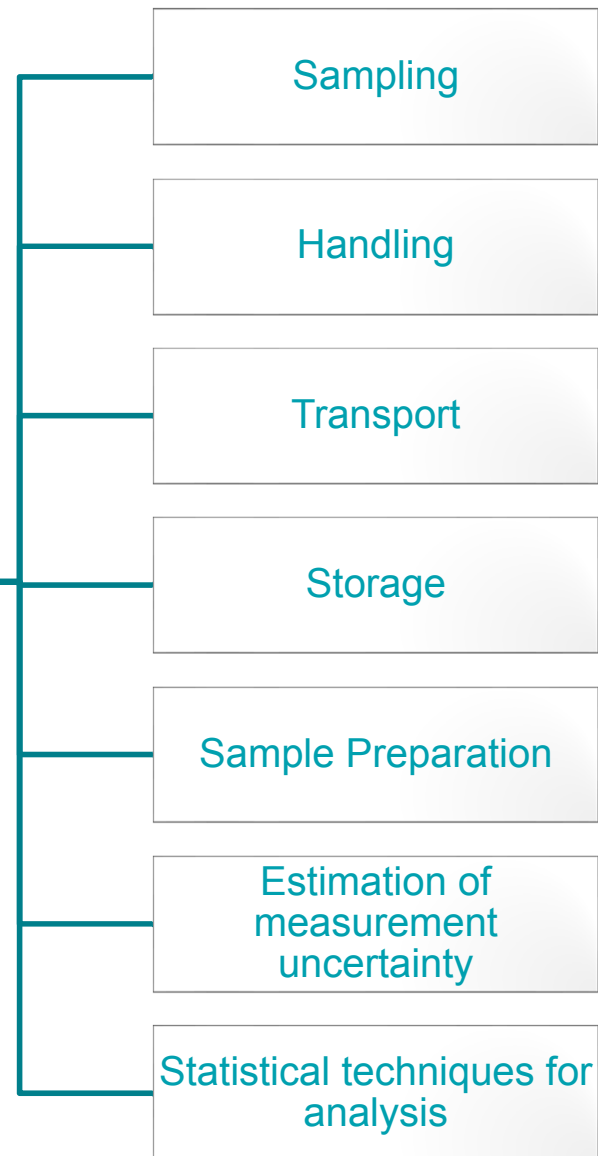


Some distinctions



Methods

The laboratory shall use appropriate methods that meet the needs of the customer and use procedures for all tests that should include these items:



When Selecting a Method

It should be approved or published in

- International, regional or national standards
- Reputable technical organizations
- Relevant scientific 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
- The laboratory must confirm they can properly operate any standard method



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 confirm that it can properly operate the method before introducing the test. If the method changes, the confirmation 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 (any amplifications or modifications)

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 the *(Insert name here) 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



Validation

Techniques used should include one or more of the following:



Calibration using reference standards or materials



Comparison of results with other methods



Interlaboratory comparisons



Systematic assessment of the factors influencing the result



Assessment of the uncertainty of the results based on scientific understanding of the theoretical principles of the method

New Test Methods Should Contain:

Appropriate identification

Scope

Description of type of item to be tested

Parameters of quantities and ranges to be determined

Apparatus and equipment, including technical performance requirements

Reference standards and material 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
- Relevant 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



Results Must be Reported

- Accurately
- Clearly
- Unambiguously
- In accordance with specific test instructions
- With information requested by customer & necessary to interpretation



AAFCO
Association of American Feed Control Officials



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 address of customer
- Identification of method used
- Description, condition, and identification of item being tested
- Date of receipt
- Date test was performed
- Reference to sampling plan/procedures used, where relevant
- Test results with units of measurement
- Name, function, and signature of person(s) authorizing the test report
- A statement to the effect that the results relate only to the items tested



Test reports shall contain, where necessary to interpretation of results

- Deviations from, additions to, or exclusions from the test method, and information on specific test conditions, such as environmental conditions
- A statement of compliance/non-compliance with requirements and/or specifications
- 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



References

- ISO/IEC 17025:2005
 - <https://www.iso.org/standard/39883.html>

