

The background features a light blue color scheme with various chemical structures, including hexagonal and pentagonal rings, and several translucent blue spheres of varying sizes. The text is centered in a bold, dark blue font.

METHOD VALIDATION: AN ESSENTIAL COMPONENT OF THE MEASUREMENT PROCESS

Presented by :

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Source Material

- ❑ The NELAC Institute, Volume 1, Module 2, **Quality Systems General Requirements**, 2016
- ❑ Keith, L.H., et. al, *Principles of Environmental Analysis*, Anal Chem, 1983, 55, 2210-2218.
- ❑ Taylor, J.K. *Validation of Analytical Methods*, Anal Chem, 1983, 55, 600A-608A.
- ❑ Environmental Laboratory Advisory Board, *Recommendations for the Implementation of Performance Based Measurement Systems*, 1999
- ❑ USEPA, *Availability, Adequacy, and Comparability of Testing Procedures for the Analysis of Pollutants Established Under Section 304 (h) of the Federal Water Pollution Control Act*, January 1988.

Test Methods & Method Validation

5.4.1 General

5.4.2 Selection of Methods

5.4.3 Laboratory Developed Methods

5.4.4 Non-standard Methods

5.4.5 Validation of Methods



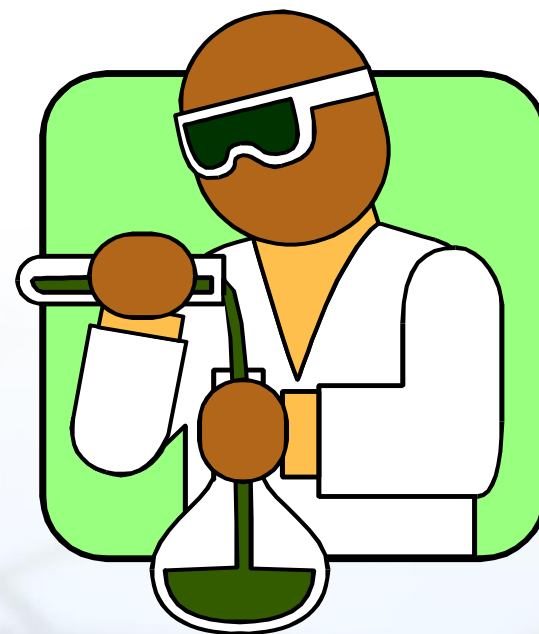
5.4.1 General

Use appropriate method
SOPs required*

Deviations allowed if:

- documented
- technically justified
- authorized
- accepted by client

*Published methods do not need to be rewritten if they can be followed without additional instructions.



5.4.2 Method Selection

- Use methods which meet needs of client and are **appropriate for the intended use**
- Published, reference methods are preferred
- Must use latest edition of method unless it is not appropriate or possible
- Non-standard methods may be used if
 - ❑ Appropriate for intended use
 - ❑ Validated
 - ❑ Client informed
- Laboratory must verify competence in using method (validate)



5.4.3 Laboratory Developed Methods

- Introduction of methods must be as a planned activity
- Development activities must be assigned to qualified personnel with adequate resources
- Requires effective communication with all parties



5.4.4 Non-standard Methods

- Subject to agreement with client
- Requires clear client specification
- Requires validation before use



Method Validation: A Demonstration of Competence

Verification of key performance measures – bias, precision, sensitivity, range

Comparison to published method performance, and / or

Comparison to data quality needs

aka:

- ❑ Method Validation (TNI)
- ❑ Demonstration of Capability (TNI)
- ❑ Initial Precision and Recovery Test (OW)
- ❑ Demonstration of Proficiency (OSW)

Method Validation Goals

- Document method's ability to be used to generate appropriate data.
- Document laboratory's ability to generate valid data.
- Document analyst's ability to perform method correctly.
- *One study can be used for all three goals.*

Validation

“ . . . suitability of methodology for providing useful analytical data . . . ”

Taylor 1983

“ . . . document that the required data quality can be met and that the methodology is suitable for its intended purpose . . . ”

ELAB, 1999

“ . . . confirmation by effective evidence that the particular requirements for a specific intended use are fulfilled . . . ”

ISO 17025

Establishing Performance Needs

1. Ideal – Measurement Quality Objectives (MQOs) established
2. Second best alternative– Compare to arbitrary limit
3. Third best alternative – Compare to reference method performance
4. Last – Document performance obtained

Levels of Verification Effort

Limited matrix, single facility;

- Industrial discharger

Limited matrices; multiple facilities

- Commercial laboratory analyzing wastewater

Unlimited matrices and facilities

- Instrument company with new technology

General Rules for Validation

- Establish performance requirements first
- At least three concentrations
- Matrix of “equal challenge”
- 65 –135 % good target for trace analysis
- Check for outliers / mistakes
- Compare to performance needs
- Record and Archive Results

Outcome from Validation Process

Statements of precision, bias, sensitivity, range

“Such statements are often misinterpreted; they merely describe the results of the exercise and are, at best, estimates of typical performance expectations for the method. However, such information should be obtained to the extent possible since it provides a quantitative basis for judging performance capability.”

Taylor 1983

Why Validate?

Assists in method selection

Provides indication of potential utility

Useful guide for best performance that can be expected

Provides basis for comparison of alternative methods

Helps establish legal standing

Meets TNI accreditation requirements

Misuse of Validation Data

Quality of all future measurements



5.4.5 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.



5.4.5 Validation of Methods

Laboratory shall validate:

- ❑ non-standard methods
- ❑ laboratory developed methods
- ❑ standard methods used outside scope
- ❑ modifications of standard methods

Validation **as extensive as necessary**

New validation required if changes made

Results assessed against intended use



Validation Techniques

Note: should be one of, or a combination of:

- calibration using reference standards or reference materials;
- comparison of results achieved 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 and practical experience.



Validation Outcome

NOTE Validation is always a balance between costs, risks and technical possibilities. There are many cases in which the range and uncertainty of the values (e.g. accuracy, detection limit, selectivity, linearity, repeatability, reproducibility, robustness and cross-sensitivity) can only be given in a simplified way due to lack of information.



CONCLUSIONS

- Methods must be validated to ensure they are fit for the intended purpose.
- Rigorous method validation is a key aspect of any measurement system.
- Method validation may represent ideal performance.
- There are many acceptable approaches.