

Laboratory Quality Systems
Unit III: Validation of Analytical Procedures
(Weeks 6 - 8)

If you need help, please e-mail prabha@otsc.tamu.edu

Learning Objectives

- Learn both the terminology and procedures for calculating performance indicators in laboratory method validation to assess fitness for purpose with an emphasis on instrumental methods.
- Understand the purpose of analytical procedure validation in a quality control laboratory with an emphasis on rapid methods

Method Validation Overview

Presentation

☐ [Method Validation](#) (33 min.) | [PDF](#)

Excel Demonstrations

Demonstrations	Associated Excel Files and other files
LOD and LOQ (1 min.)	LOD/LOQ Excel File
Horrat Value (2 min.)	Horrat Annotated Excel File Optional Readings: The Horwitz Ratio (HorRAT): A Useful Index of Method Performance with Respect to Precision
Calibration Curve (2 min.)	Calibration Curve Excel File
Cochran Test and Grubbs Test (7 min.)	Cochran and Grubbs Test Annotated Excel File Optional Reading: Sample Criteria for Testing Outlying Observations

Assigned Readings (Applies to all methods)

- ☐ [Harmonized Guidelines for Single Laboratory Validation of Methods of Analysis \(IUPAC Technical Report\)](#)
- ☐ [OTSC SOP – Validation of New Methods of Analysis \(2022\)](#)
- ☐ [Guidelines for the validation and verification of quantitative and qualitative test methods \(National Association of Testing Authorities, Australia, 2012\)](#)
- ☐ [AOAC Guidelines for Single Laboratory Validation of Chemical Methods for Dietary Supplements and Botanicals](#)

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Rapid Tests

Presentation

- ☐ [Method Validation of Rapid Tests](#) (27 min.) | [PDF](#)

Literature used in the Presentation

- ☐ [Design Criteria and Test Performance Specification for Biotechnology Rapid Kits](#)
- ☐ [Aflatoxin risk management in Texas: Test kit approval for maize](#) (Dai et al. 2013)
- ☐ [Design Criteria and Test Performance Specifications for Quantitative Aflatoxin Test Kits](#) (GIPSA, 2018)
- ☐ [BAM: Rapid Methods for Detecting Foodborne Pathogens](#) (FDA, 2001)
- ☐ [Validation Study of Rapid ELISA for Detection of Aflatoxin in Corn](#) (Lupo et al, 2010)
- ☐ [Validation of Qualitative Analytical Methods](#) (Trullols et al, 2004)

Microbiology

Presentation

- ☐ [Validation of Microbiological Processes](#) (31 min.) | [PDF](#)

Required Reading

- ☐ [Guidelines for the Validation of Analytical Methods for the Detection of Microbiological Pathogens in Foods and Feeds](#) (FDA 2015)

Chemical Methods

Presentation

- ☐ [Validation of Analytical Procedures: Chemical](#) | [PDF](#)
 - [Example Excel slide 28: Validation Data](#)
 - [Example Excel slide 31: Calibration Curve](#)

Literature used in the Presentation

- ☐ [AOAC Guidelines for Single Laboratory Validation of Chemical Methods for Dietary Supplements and Botanicals](#)
- ☐ [Rapid Determination of Fumonisin in Corn-Based Products by Liquid Chromatography/Tandem Mass Spectrometry](#) (Li et al, 2010)
- ☐ [Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis](#) (AOAC 2002)

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Spectroscopy

Presentation

- ☐ [Method Validation of NIR Spectroscopy Method](#) (12 min.) | [PDF](#)

Demonstration

- ☐ [NIR Data Evaluation Annotated Excel Sheet](#)

Discussion

Graded Discussion # 4: Due by Midnight on Monday, March 10, 2025 (5 pts)

Method validation is an essential component of the measures that a laboratory should implement to allow it to produce reliable analytical data. All methods selected for use in a laboratory needs to be validated prior to use. Explain the criteria that should be used to select methods in a laboratory. List the steps involved in validating a method including any statistical analysis that should be conducted. Use assigned readings as appropriate.

Submit in course Google Group under Graded Discussion #3 Thread;

Graded Discussion # 5 –Due by Midnight on Monday, March 31, 2025 (5 pts) Reference Materials play a very important rule in method validation. However, in some cases, reference materials are not readily available. In this case, how would you validate the method without a reference material? Use assigned readings as appropriate.

Submit in course Google Group under Graded Discussion #5 Thread;

Assignments

- ☐ [Homework # 5 \(Calculations for Validation\)](#) – Due by Midnight on Monday, March 24, 2025 (20 pts)

Submit via e-mail to outreach@otsc.tamu.edu