

ISSC Method Application and Single Lab Validation Summary of Required Elements for Acceptance of a Method for Use in the NSSP

The purpose of single laboratory validation in the National Shellfish Sanitation Program (NSSP) is to ensure that the analytical method under consideration for adoption by the NSSP is fit for its intended use in the Program. A summary of required elements has been developed which explores and articulates the need for the method in the NSSP; provides an itemized list of method documentation requirements; and, sets forth the performance characteristics to be tested as part of the overall process of single laboratory validation. For ease in application, the performance characteristics listed under validation criteria in this document have been defined and accompany the summary of required elements as part of the process of single laboratory validation. Further a generic protocol has been developed that provides the basic framework for integrating the requirements for the single laboratory validation of all analytical methods intended for adoption by the NSSP. Methods submitted to the Interstate Shellfish Sanitation Conference (ISSC) Laboratory Committee for acceptance will require, at a minimum, six (6) months for review from the date of submission.

Name of the New Method	
Name of the Method Developer/Submitter	
Developer Contact Information	
<u>Required Elements</u>	<u>Brief Description</u>
A. Need for the New Method	
1. Clearly define the need for which the method has been developed.	
2. What is the intended purpose of the method?	
3. Is there an acknowledged need for this method in the NSSP?	
4. What type of method? i.e. chemical, molecular, culture, etc.	
B. Method Documentation	
1. Method documentation includes the following information:	
Method Title	
Method Scope	
References	
Principle	
Any Proprietary Aspects	
Equipment Required	
Reagents Required	
Sample Collection, Preservation and Storage Requirements	
Safety Requirements	
Clear and Easy to Follow Step-by-Step Procedure	
Quality Control Steps Specific for this Method	
Laboratory Evaluation Checklist for Use During Evaluations of Proper Method Implementation	
C. Validation Criteria	
1. Accuracy / Trueness	
2. Measurement Uncertainty	
3. Precision Characteristics (repeatability and reproducibility)	
4. Recovery	
5. Specificity	

6. Working and Linear Ranges	
7. Limit of Detection	
8. Limit of Quantitation / Sensitivity	
9. Ruggedness	
10. Matrix Effects	
11. Comparability (if intended as a substitute for an established method accepted by the NSSP)	
D. Other Information	
1. Cost of the Method	
2. Special Technical Skills Required to Perform the Method	
3. Special Equipment Required and Associated Cost	
4. Abbreviations and Acronyms Defined	
5. Details of Turn Around Times (time involved to complete the method)	
6. Provide Brief Overview of the Quality Systems Used in the Lab	
Submitters Signature	Date:
Submission of Validation Data and Draft Method to Committee	Date:
Reviewing Members	Date:
Accepted	Date:
Recommendations for Further Work	Date:
Comments:	

DEFINITIONS

1. **Accuracy/Trueness** - Closeness of agreement between a test result and the accepted reference value.
2. **Analyte/measurand** - The specific organism or chemical substance sought or determined in a sample.
3. **Blank** - Sample material containing no detectable level of the analyte or measurand of interest that is subjected to the analytical process and monitors contamination during analysis.
4. **Comparability** - The acceptability of a new or modified method as a substitute for an established method in the NSSP. Comparability must be demonstrated for each substrate or tissue type by season and geographic area if applicable.
5. **Fit for purpose** - The analytical method is appropriate to the purpose for which the results are likely to be used.
6. **HORRAT value** - HORRAT values give a measure of the acceptability of the precision characteristics of a method.⁴
7. **Limit of Detection** - The minimum concentration at which the analyte or measurand can be identified. Limit of detection is matrix and analyte/measurand dependent.⁴
8. **Limit of Quantitation/Sensitivity** - The minimum concentration of the analyte or measurand that can be quantified with an acceptable level of precision and accuracy under the conditions of the test.
9. **Linear Range** - The range within the working range where the results are proportional to the concentration of the analyte or measurand present in the sample.
10. **Measurement Uncertainty** - A single parameter (usually a standard deviation or confidence interval) expressing the possible range of values around the measured result within which the true value is expected to be with a stated degree of probability. It takes into account all recognized effects operating on the result including: overall precision of the complete method, the method and laboratory bias and matrix effects.
11. **Matrix** - The component or substrate of a test sample.
12. **Method Validation** - The process of verifying that a method is fit for purpose.¹
13. **Precision** - The closeness of agreement between independent test results obtained under stipulated conditions.^{1, 2} There are two components of precision:
 - a. **Repeatability** - The measure of agreement of replicate tests carried out on the same sample in the same laboratory by the same analyst within short intervals of time.
 - b. **Reproducibility** - The measure of agreement between tests carried out in different laboratories. In single laboratory validation studies reproducibility is the closeness of agreement between results obtained with the same method on replicate analytical portions with different analysts or with the same analyst on different days.
14. **Quality System** - The laboratory's quality system is the process by which the laboratory conducts its activities so as to provide data of known and documented quality with which to demonstrate regulatory compliance and for other decision-making purposes. This system includes a process by which appropriate analytical methods are selected, their capability is evaluated, and their performance is documented. The quality system shall be documented in the laboratory's quality manual.
15. **Recovery** - The fraction or percentage of an analyte or measurand recovered following sample analysis.
16. **Ruggedness** - The ability of a particular method to withstand relatively minor changes in analytical technique, reagents, or environmental factors likely to arise in different test environments.⁴
17. **Specificity** - The ability of a method to measure only what it is intended to measure.¹
18. **Working Range** - The range of analyte or measurand concentration over which the method is applied.

REFERENCES:

1. Eurachem Guide, 1998. The Fitness for Purpose of Analytical Methods. A Laboratory Guide to Method Validation and Related Topics. LGC Ltd. Teddington, Middlesex, United Kingdom.
2. IUPAC Technical Report, 2002. Harmonized Guidelines for Single-Laboratory Validation of Methods of Analysis, Pure Appl. Chem., Vol. 74, (5): 835-855.
3. Joint FAO/IAEA Expert Consultation, 1999. Guidelines for Single-Laboratory Validation of Analytical Methods for Trace-Level Concentrations of Organic Chemicals.
4. MAF Food Assurance Authority, 2002. A Guide for the Validation and Approval of New Marine Biotxin Test Methods. Wellington, New Zealand.
5. National Environmental Laboratory Accreditation. , 2003. Standards. June 5.
6. EPA. 2004. EPA Microbiological Alternate Procedure Test Procedure (ATP) Protocol for Drinking Water, Ambient Water, and Wastewater Monitoring Methods: Guidance. U.S. Environmental Protection Agency (EPA), Office of Water Engineering and Analysis Division, 1200 Pennsylvania Avenue, NW, (4303T), Washington, DC 20460. April.