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CONTROLLED DOCUMENT

Purpose

This procedure describes how OTSC will select and validate new laboratory analytical procedures. The laboratory will use methods which meet the need of FFCS and other OTSC customers. Standard methods are preferable; however, non-standard and laboratory developed methods may be used when deemed more appropriate.

Scope / Field of Application

This procedure applies to all analytical procedures used in the OTSC laboratory.

Responsibilities

Chemists, Research Scientists – investigate available methods, perform testing, generate and analyze data.

Laboratory Management – evaluate methods, review data.

Quality Manager - specify validation requirements, determine if method meets the needs of the organization, approve method.

Procedure

Selection of method:

1. The need for a new method will be determined. This may be initiated by the State Chemist, FFCS, Lab Manager, Quality Manager, or Chemist.

2. Available methods will be investigated and evaluated by the Chemist, Research Scientist, and/or Lab Management. (Cost analysis determination/benefits will be part of the evaluation process).

3. The selected method will be approved by the Quality Manager.

Validation/Verification of method:

1. Determine method development procedures (Quality Manager).

2. Laboratory Management will assign the project to a Chemist or Research Scientist.

3. Chemist will procure appropriate chemicals, supplies, and equipment.

4. Chemist will prepare written document summarizing procedures to be used. (Note: modifications to the procedures used are often required).

5. Quality Manager will approve the procedures.

6. Chemist will collect preliminary data and submit the results to the Quality Manager for review.

7. Chemist will complete collection of data.

8. Laboratory Management or designee will review data, prepare summary, and submit report to Quality Manager.

9. SOP will be prepared.

10. Quality Manager will approve the method.

11. If the method will be used to record results in the LIMS, Chemist will provide calculations, lowest reporting level and other data to LIMS manager. LIMS manager will set up method in LIMS and coordinate with IT department for setup in FFCS database.

Minimum Validation Acceptance Criteria:

1. Accuracy/Recovery – A minimum of 7 independent analyses per concentration level covering the analytical range. It is preferable to use certified reference materials (CRM). If CRMs are not available proficiency test program samples (AAFCO, Magruder, AOCS, etc.) may be used. Purified standards/chemicals can be used if no other reference samples are available. Spiked samples may also be required. The least preferred method to determine accuracy is to compare the results to those results obtained using another already validated procedure.

2. Precision – Typically for single lab validation one performs **r** replicate analyses of **m** test portions over a period of **d** days for each sample type (matrix) **n**, where **r** is the number of replicates (2,3,...), **m** is the number of test portions in each group, **d** is the number of days, and **n** is the number of different sample types.

r x **m** should never be less than 10
n should be at least 2 (preferably more)
d should be at least 2

3. Calibration – The calibration line should include 4-5 reference points. Single point calibrations must be used cautiously. Correlation coefficients of >0.99 should be the goal but with modern line fitting software, nonlinear calibration lines are acceptable.

4. Limits of detection/limits of quantification - for some customers, limits of detection must be demonstrated. Typically this is defined as the analyte concentration at 3 times the signal produced by analyzing a blank. For OTSC purposes the limit of detection is not as significant as the limit of quantification. Typically this is defined as the analyte concentration at 10 times the signal produced by analyzing a blank. However, the useful limit of quantification is the lowest analyte level the laboratory can measure with an accuracy and precision acceptable to its customer. Unless otherwise specified OTSC will document the typical LOD/LOQ (3x and 10x blank). Note: generally OTSC does not report any analytical results that are not bracketed by the calibration curve. Therefore, the lowest reported result would be based on the lowest standard of the curve, provided the response has good precision.

5. Criteria - Accuracy and precision target limits will be taken from AOAC (Horwitz). Precision or repeatability is calculated as the relative standard deviation (coefficient of variability) and accuracy is calculated as % recovery. See table below:

Concentration	Repeatability (%)	Recovery (%)
100 %	1.3	98-102
10 %	1.9	98-102
1 %	2.7	97-103
0.1 %	3.7	95-105
0.01 %	5.3	90-107
0.001 %	7.3	80-110
1 ppm	11	80-110
100 ppb	15	80-110
10 ppb	21	60-115
1 ppb	30	40-120

Revision History

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