ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

Management and Technical Requirements for Laboratories Performing Environmental Analysis

TNI Standard

P.O. Box 2439
Weatherford, TX 76086
817-598-1624
www.nelac-institute.org

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PREFACE

This Standard is the result of many hours of effort by those volunteers on The NELAC Institute (TNI) Proficiency Testing Committee. The TNI Board of Directors wishes to thank these committee members for their efforts in preparing this Standard as well as those TNI members who offered comments during the voting process.

This Standard may be used by any organization that wishes to implement a program for the accreditation of environmental laboratories.

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Environmental Laboratory Sector

Volume 1

Table of Contents

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1.7.3 Data Acceptance/Rejection Criteria

1.7.3.1 Negative Control – Method Performance: Method Blank

a) While the goal is to have no statistically significant difference from zero, each method blank shall be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. The source of contamination or other bias shall be investigated and measures taken to minimize or eliminate the problem and affected samples reprocessed or data shall be appropriately qualified if:

i. the absolute value of the activity of a targeted analyte in the blank exceeds three times its combined standard uncertainty, AND is greater than 1/10 of the activity measured in any sample; or

ii. the method blank result otherwise affects the sample results as per the method requirements or the project-specific measurement quality objectives.

b) The acceptance criteria for samples associated with a failed method blank shall be calculated in a manner that compensates for sample results based on differing aliquot sizes.

c) When a blank result is determined to be significantly different from zero, the cause shall be investigated and measures taken to minimize or eliminate the problem. Samples associated with a failed blank shall be evaluated as to the best corrective action for the samples (e.g., reprocessing or data qualifying codes).

d) The occurrence of a failed method blank and any associated corrective action shall be noted in the laboratory report to the client.

1.7.3.2 Positive Control – Method Performance: Laboratory Control Sample (LCS)

a) The results of the individual batch LCS are calculated in percent recovery or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation.

b) The individual LCS is compared to the acceptance criteria as published in the mandated method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits or utilize client specified assessment criteria.

c) An LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with an LCS determined to be “out of control” shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.

d) The occurrence of a failed LCS and any associated actions shall be noted in the laboratory report to the client.

1.7.3.3 Sample-Specific Controls

a) Matrix Spike; Matrix Spike Duplicates

i. The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R), relative percent difference (RPD), or other appropriate

statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation for %R, RPD or other statistical treatment used.

ii. The results are compared to the acceptance criteria as published in the mandated method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix spike results outside established criteria, corrective action shall be documented or the data reported with appropriate data qualifying codes.

iii. The occurrence of a failed matrix spike and any associated actions shall be noted in the laboratory report to the client.

b) Replicates

i. The results from replicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD) or another statistical treatment (e.g., normalized differences).

ii. The laboratory shall document the calculation for relative percent difference or other statistical treatments.

iii. Results are compared to the acceptance criteria as published in the mandated method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For replicate results outside established criteria, corrective action shall be documented or the data reported with appropriate data qualifying codes.

iv. The occurrence of a failed replicate and any associated actions shall be noted in the laboratory report to the client.

1.7.4 Sample Handling

a) All samples that require thermal preservation shall be considered acceptable if the arrival temperature of a representative sample container is either within 2°C of the required temperature or the method specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable.

i. Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 1.7.4.a. In these cases, the samples shall be considered acceptable if the samples were received on ice.

ii. If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required. Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

b) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or chlorine, prior to or during sample preparation or analysis.

ENVIRONMENTAL LABORATORY SECTOR

VOLUME 1

MANAGEMENT AND TECHNICAL REQUIREMENTS FOR LABORATORIES PERFORMING ENVIRONMENTAL ANALYSIS

Module 7: Quality Systems for Toxicity Testing

TNI Standard

P.O. Box 2439
Weatherford, TX 76086
817-598-1624
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PREFACE

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This Standard supplements Module 2, Quality Systems General Requirements, and may be used by any organization that wishes to implement a program for the accreditation of environmental laboratories.

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VOLUME 1, MODULE 7

Quality Systems for Toxicity Testing

Table of Contents

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VOLUME 1, MODULE 7

Quality Systems for Toxicity Testing

1.0 TOXICITY TESTING

1.1 Introduction

This Standard applies to laboratories measuring the toxicity and/or bioaccumulation of contaminants in effluents (whole effluent toxicity or WET), receiving waters, sediments, elutriates, leachates and soils. In addition to the essential quality control standards described below, some methods may have additional or other requirements based on factors such as the type of organism evaluated and contain detailed quality control requirements for toxicity testing activities. The evaluation of laboratories for this discipline is in conjunction with a quality system as specified in the general requirements module. Adherence to quality systems requirements will ensure that all quality control procedures specified in this module are being followed.

1.2 Scope

The essential quality control procedures applicable to toxicity measurements are included in this Standard. Additional quality control requirements that are specified by method, regulation or project shall be met by laboratories.

1.3 Terms and Definitions

The relevant definitions from TNI, Volume 1, Module 2, Section 3.0 apply. Definitions related to this document, which are used differently or do not exist in the above references are defined below.

1.3.1 Additional Terms and Definitions

When referred to in this module, “sensitivity” relates to the meaning referenced in the accredited method.

1.3.2 Exclusions and Exceptions

Reserved

1.4 Method Selection

When it is necessary to use testing methods not covered by an approved method, these shall be subject to agreement with the data user and shall include a clear specification of the data user’s requirements and the purpose of the environmental test. The method developed shall have been validated appropriately before use.

The characteristics of validated methods (e.g., the uncertainty of the results, limit of repeatability and/or reproducibility, robustness against external influences and/or cross-sensitivity against interference from the matrix of the sample/test object), as assessed for the intended use, shall be relevant to the users’ needs.

1.5 Method Validation

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

1.6 Demonstration of Capability (DOC)

1.6.1 General

Prior to acceptance and institution of any method for data reporting, satisfactory initial DOC is required (see Section 1.6.2).

Thereafter, ongoing DOC (Section 1.6.3), as per the quality control requirements in Section 1.7.1.2 is required.

In cases where a laboratory analyzes samples using a method that has been in use by the laboratory for at least one year prior to applying for accreditation, and there have been no significant changes in personnel or method, the ongoing DOC shall be acceptable as an initial DOC. The laboratory shall have records on file to demonstrate that an initial DOC is not required.

For the initial DOC, appropriate records as discussed in Section 1.6.2.1 shall be completed.

An initial DOC shall be completed each time there is a change in personnel, or method.

In general, this demonstration does not test the performance of the method in real world samples. However, before any results are reported, the initial DOC shall be performed. An intial DOC may be completed by a group of analysts and is for situations in which several individuals perform part of a set of activities that would produce a testing result.

All demonstrations shall be documented. All data applicable to the demonstration shall be retained and readily available at the laboratory.

1.6.2 Initial DOC

An initial DOC shall be made prior to using any method, and at any time there is a significant change in personnel or method or any time that a method has not been performed by the laboratory or analyst in a twelve (12) month period.

1.6.2.1 The laboratory shall document each initial DOC in a manner such that the following information is available for each affected employee:

a) analyst(s) involved in preparation and/or analysis;

b) matrix;

c) species and endpoint(s);

d) identification of method(s) performed;

e) identification of laboratory-specific SOP used for analysis, including revision number;

f) date(s) of analysis;

g) summary of analyses, including information outlined in Section 1.6.2.2.

1.6.2.2 If the method or regulation does not specify an initial DOC, the following procedure is acceptable. It is the responsibility of the laboratory to document that other approaches to initial DOC are adequate.

Each analyst shall meet the quality control requirements as specified in Section 1.7.1.2.

1.6.3 Ongoing DOC

The laboratory shall have a documented procedure describing ongoing DOC. The analyst(s) shall demonstrate on-going capability by meeting the quality control requirements of the method, laboratory SOP, client specifications, and/or this Standard. It is the responsibility of the laboratory to document that other approaches to on-going demonstration of capability are adequate. This on-going demonstration may include performing another initial demonstration of capability as per 1.6.2 or a documented process of analyst review using QC samples can serve as the annual on-going demonstration of capability. QC samples shall be reviewed to identify patterns for individuals or groups of analysts and determine if corrective action or retraining is necessary.

1.7 Technical Requirements

1.7.1 Quality Control

The laboratory shall have quality control procedures for monitoring the validity of environmental tests undertaken. The resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results. This monitoring shall be planned and reviewed and may include, but not be limited to, the following:

a) regular use of certified reference materials and/or internal quality control using secondary reference materials;

b) participation in inter-laboratory comparison or proficiency-testing program;

c) replicate tests using the same or different methods;

d) retesting of retained samples; and

e) correlation of results for different characteristics of a sample (for example, total phosphate should be greater than or equal to orthophosphate).

1.7.1.1 Essential Quality Control Procedures

These general quality control principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory and are further described in this module. The standards for any given test type shall assure that the applicable principles are addressed:

a) All laboratories shall have detailed written protocols in place to monitor the following quality controls:

   i. positive and negative controls to monitor tests such as blanks, spikes, reference toxicants;

   ii. tests to define the variability and/or repeatability of the laboratory results such as replicates;

   iii. measures to evaluate method capability, such as percent minimum significant difference (PMSD);

   iv. selection of appropriate formulae to reduce raw data to final results such as regression and statistical analyses;

   v. selection and use of reagents and standards of appropriate quality;

Control chart limits are expected to be exceeded occasionally regardless of how well a laboratory performs. Acceptance limits for point estimates (ICp, ECp) that are based on 95% confidence limits should theoretically be exceeded for one in twenty tests. Depending on the dilution factor and test sensitivity, control charts based on hypothesis test values (NOEC, NOAEC) may be expected to be exceeded on a similar frequency. Test results that fall outside of control chart limits at a frequency of 5% or less, or which fall just outside control chart limits (especially in the case of highly proficient laboratories which may develop relatively narrow acceptance limits over time), are not rejected de facto. Such data are evaluated in comparison with control chart characteristics including the width of the acceptance limits and the degree of departure of the value from acceptance limits.

Laboratories shall develop acceptance/rejection policies, consistent with the methods, for SRT data which considers source of test organisms, the direction of the deviation, test dilution factor, test sensitivity (for hypothesis test values), testing frequency, out-of-control test frequency, relative width of acceptance limits, inter-test CV, and degree of difference between test results and acceptance limits.

In the case of reference toxicant data which fails to meet control chart acceptance criteria, the test data are examined for defects, corrective action taken and the test repeated if necessary, using a different batch of organisms or the data is qualified.

Intra-laboratory precision is determined on an ongoing basis through the use of control charts. The control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC50 for acute tests, or as appropriate hypothesis test values, such as the NOEC or NOAEC, over time within a laboratory.

1.7.2.2 Negative Controls

The test acceptability criteria specified in the method shall be achieved for both the reference toxicant and the effluent or environmental sample toxicity test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity tests.

1.7.2.3 Selection of Appropriate Statistical Analysis Methods

a) Methods of data analysis and reporting as specified by language in the regulation, permit, or the method shall be followed as required.

b) Toxicity data shall be plotted on semi-logarithmic graph paper, relating time, mortality, and effluent concentration to verify computational results.

1.7.3 Sample Handling

All samples shall be chilled to 0-6°C during or immediately after collection except as prescribed by the method and approved by the regulatory agency having authority for program oversight.