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Standard Guide for QC of Screening Methods in Water[1]

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1. Scope

1.1 This guide covers general considerations for the Quality Control practices for use with screening methods for organic and inorganic constituents in water. Methods are provided by various standard setting bodies, governmental agencies, as well as many domestic and international manufacturers.

1.2 This guide provides general QC procedures that are applicable to a broad range of screening methodologies. These procedures help to ensure the quality of data that is generated. Additional, method-specific or project specific requirements may be necessary. This guide also includes general considerations regarding proper utilization of screening methods.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]

D 4840 Guide for Sampling Chain-of-Custody Procedures[2]

D 5172 Guide for Documenting the Standard Operating Procedures Used for the Analysis of Water[2]

D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis[3]

D 5905 Practice for the Preparation of Substitute Wastewater[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminology D 1129 and Practice D 5847.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *action level*, *n*—a concentration of the analyte of concern at which some further action is required or suggested.

3.2.2 *batch*, *n*—a set (group) of samples analyzed such that results of analysis of the QC samples analyzed with the batch are indicative of the quality of the results of analysis of samples in the batch. The number of samples in the batch is defined by the task group responsible for the method.

3.2.2.1 *Discussion*—See Practice D 5847 for definition and discussion of batch and batch size.

3.2.3 *false negative*, *n*—a negative response for a sample that contains the target analyte(s) at or above the stated action level.

3.2.4 *false positive*, *n*—a positive response for a sample that contains the target analyte(s) below the stated action level.

3.2.5 *qualitative method*, *n*—a validated method that detects presence or absence of an analyte at a specified screening limit.

3.2.6 *screening limit*, *n*—the concentration of analyte that can be determined with a given certainty. The task group responsible for the method establishes the determination of the screening limit.

3.2.7 *screening method*, *n*—a method that is used to separate or categorize samples.

3.2.7.1 *Discussion*—An example would be a method that provides results that would be used to separate samples into those that contain an analyte above or below a specified action level.

3.2.8 *semi-quantitative method Type 1*, *n*—a method whose results are given in specified, discreet concentration ranges.

3.2.8.1 *Discussion*—Two types of examples of this would include semi-quantitative immunoassays or test strips. The cutoff concentration of the ranges has been predefined.

3.2.9 *semi-quantitative method Type 2*, *n*—a method whose results are reported as a single number along with the stated uncertainty.

3.2.9.1 *Discussion*—The uncertainty will be reported as (standard deviation of x at a concentration of y). The values of x and y can be established from the Initial Demonstration of Performance study.

4. Significance and Use

4.1 Screening methods are often used to determine the presence or absence of a specific analyte, groups of analytes, classes of compounds or other indicators of chemical compounds in order to determine if further analysis or action is necessary. The determination whether to proceed with further action is useful in reducing the number of negative results for which the screening method serves as a surrogate.

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[1] This guide is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.02.

4.2 The use of screening methods, whether to generate qualitative or semi-quantitative results, is increasingly becoming a useful tool for regulatory monitoring, process control, and site characterization. The appropriate use of a screening method, or any other method for that matter, is dependent upon the Data Quality Objectives (DQOs) that are defined by the user of the data.

4.3 Persons responsible for assessing the quality of the data generated by the use of screening methods should have detailed Quality Control guidelines by which to assess data quality.

5. Consideration for Selection of an Appropriate Screening Method

5.1 The screening method chosen must be appropriate for the Action Level of the project.

5.2 The chosen screening method must allow for the necessary number of samples to be run in a timely manner, not to exceed the storage limits of the sample as defined by the method.

5.3 Many screening methods will give a positive result for several compounds or class of compounds. It must be determined if a more specific method is necessary to eliminate these positive results.

5.4 It is essential that an appropriate documentation system be established. A Standard Operating Procedure (SOP) for the screening method containing the appropriate QC elements from this guide must be available. See Guides D 4840 and D 5172 for information on establishing a SOP for a method.

5.5 Issues relating to timeliness of sample collection and analysis should be considered when selecting an appropriate screening method. For example, for analyses that must be run immediately following sample collection, a method capable of being run in the field may be necessary.

5.6 When selecting a field screening method, considerations must be given to the expected field conditions. Factors such as humidity, power requirements, temperature, effects of ambient lighting, etc must be addressed.

6. Structure of a Quality Control System for Screening Methods

6.1 *General Considerations*:

6.1.1 Due to possible difficulties in performing several of these requirements in the field it is acceptable to perform those QC requirements in the laboratory.

6.1.2 The following are suggested at a minimum for ongoing QC.

6.1.2.1 Run a method blank containing no analyte, using reagent water as described in Specification D 1193, with every batch to verify the test will produce a negative result.

6.1.2.2 Run a standard or set of standards with every batch. This may also serve as the calibration verification.

6.1.2.3 Run a sample duplicate with every batch. Ensure that the replicate results meet the methods performance criteria.

6.1.3 It is required that the analyst using the screening method proves their proficiency with the test. The task group responsible for the method will establish proficiency requirements.

6.1.4 When performing matrix evaluations it is recommended to use the actual matrix if possible. If not, a similar matrix should be used. Example: Use substitute wastewater, as described in Practice D 5905, for a wastewater matrix. This will determine the suitability of the method in the matrix of interest. It is also suggested a laboratory control sample (LCS) be run in a representative matrix.

6.1.5 It is recommended that all screening methods be compared to a reference method to provide further detail of the screening methods capabilities and limitations. This is useful when establishing or verifying false positives/ negatives and recoveries in actual samples.

6.1.6 Specific requirements of a QC system for screening methods will be dependent upon the type of analysis being performed.

6.1.7 Semi-quantitative Type 2 methods require either preparing a user-generated calibration curve prior to running analyses, or verifying the manufacturers pre-programmed calibration curve with standards before or during sample analysis.

6.2 *Qualitative Methods*:

6.2.1 The following tests are recommended.

6.2.1.1 Run a method blank containing no analyte to verify the test will produce a negative result.

6.2.1.2 Run a standard of the analyte of interest to verify the test will produce a positive result.

6.2.1.3 Establish the screening limit of the method. Ensure the screening limit is below the action level of interest.

6.2.1.4 If possible run a representative matrix without the target analyte and verify a negative response. Spike the sample with the target analyte and verify a positive response.

6.3 *Semi-Quantitative Type 1 Methods*:

6.3.1 It is suggested all of the tests for qualitative methods are performed plus these additional analyses.

6.3.1.1 Run standards that have concentrations at the predefined concentration cutoffs. For some methods this will serve as the calibration and for others it will be a calibration verification.

6.3.1.2 Perform a matrix spike; ensure the results are in the appropriate concentration range.

6.3.1.3 Perform a false positive/ false negative study at all of the concentrations of interest.

6.3.1.4 Perform a precision study. When performing precision for semi-quantitative Type 1 methods the precision will be reported as the number of times the result was in the same range. Example: 8 of 9 replicates fell in the concentration range of 5 to 10 ppm.

6.3.1.5 Perform a bias study. When performing bias for Semi-Quantitative Type 1 methods the bias will be based on whether the result was in the correct concentration range. Example: When running a 7 ppm standard the result was in the range of 5 to 10 ppm.

6.4 *Semi-Quantitative Type 2 Methods*:

6.4.1 It is suggested all of the tests for qualitative methods are performed plus these additional analyses.

6.4.1.1 Perform a calibration or calibration verification of the method.

6.4.1.2 Run a matrix spike; determine the recovery of the spike.

6.4.1.3 Run a set of standards to determine the bias of the method.

6.4.1.4 Perform a precision study. This will establish the uncertainty that is reported in the final result.

7. Inter-laboratory Comparison

7.1 When performing an Inter-laboratory study the following should be considered.

7.1.1 When determining precision for qualitative and Semi-Quantitative Type 1 methods define the precision as in 6.3.1.4.

7.1.2 When performing bias for qualitative and Semi-Quantitative Type 1 methods define the bias as in 6.3.1.5.

7.1.3 Semi-Quantitative Type 2 methods can typically be evaluated as quantitative methods.

8. Screening Method Validation

8.1 A validated screening method will have had the following analyses performed.

8.1.1 All of the appropriate quality control requirements from Section 6 will have been run within a single laboratory.

8.1.2 The method will be run using an independent reference material (IRM).

8.1.3 The method will be compared to a reference method.

8.1.4 An inter-laboratory study will be performed.

9. Reporting Results

9.1 All data that is generated following this guide must reference this guide number and report the type of method performed as described in 3.2 (that is, qualitative, semi-quantitative Type 1, semi-quantitative Type 2).

10. Keywords

10.1 qualitative; quality control; screening; semi-quantitative

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL LITERATURE RESOURCE

X1.1 The following literature was not reference in this document but is valuable for additional information on this subject.

X1.1.1 *Guide to Method Flexibility and Approval of EPA Water Methods*, Draft Guide, December, 1996.