



## Standard Practice for Writing Quality Control Specifications for Standard Test Methods for Organic Constituents<sup>1</sup>

This standard is issued under the fixed designation D 5789; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice covers specific requirements for incorporating quality control procedures into an ASTM test method.

1.2 The requirements in this practice should be looked upon as the primary requirements for quality control of a specific test method. In many cases, it may be desirable to implement additional quality control criteria to ensure the desired quality of data. The guidelines are intended to be incorporated into a comprehensive approach to quality assurance and quality control that include the more general approaches described in Practices D 3856 and D 4210.

1.3 The specific requirements in this practice may not be appropriate for all test methods. They will vary depending on the type of test method used as well as the analyte being determined and the sample matrix being analyzed.

1.4 This practice is for use with quantitative test methods and may not be applicable to qualitative test methods.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 1129 Terminology Relating to Water<sup>2</sup>

D 1193 Specification for Reagent Water<sup>2</sup>

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water<sup>2</sup>

D 3695 Test Method for Volatile Alcohols in Water by Direct Aqueous—Injection Gas Chromatography<sup>3</sup>

D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water<sup>2</sup>

D 4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data<sup>2</sup>

D 4375 Terminology for Basic Statistics in Committee D-19 on Water<sup>2</sup>

D 5788 Guide for Spiking Organics into Aqueous Samples<sup>3</sup>

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 11.02.

### 3. Terminology

3.1 *Definitions*—For definitions of other terms used in this practice, refer to Terminologies D 1129 and D 4375.

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration standards*—standard solutions of known concentration either purchased from an external source or prepared in-house from materials of known purity or concentration, or both, used to calibrate instrumentation.

3.2.2 *external calibration check*—analysis of an independent standard solution such as a certified reference material of known purity and concentration either obtained from the National Institute of Standards and Technology or other reputable supplier other than the laboratory's usual source. This analysis is carried out periodically to check the accuracy of the laboratory's routine calibration standard solutions.

3.2.3 *matrix spike*—addition of a known concentration of analyte to a sample representing a specific matrix for the purpose of evaluating interference and recovery from matrix components.

3.2.4 *method blank*—reagent water (see Specification D 1193) either known to be free of the constituent of interest or containing only a low, known concentration of the constituent of interest not exceeding five times the estimated minimum detection level. The purpose of the analysis of the method blank is to confirm that the reagents or analytical system, or both, do not contribute a measurable amount of the constituent of interest during analysis of routine samples or, if they do, to determine what that contribution is.

3.2.5 *quality control sample*—a sample of known concentration and composition that is taken through the entire analytical procedure to determine whether the analytical system is in control. The sample can be a certified reference material obtained from an outside source or prepared in-house from materials of known purity and concentration. The quality control (QC) sample shall be prepared from a material that sufficiently challenges the test. Alternatively, the QC sample may be a fully characterized real sample of the matrix that is typically analyzed.

3.2.6 *sample pretreatment*—any handling, manipulation or treatment of a sample prior to subjecting it to the primary mode of analysis. Examples are filtration, digestion, dilution, pH adjustment, or extraction.

3.2.7 *set of samples*—a group of 20 or fewer samples of the

same matrix type that are being analyzed for essentially the same components.

#### 4. Summary of Practices

4.1 This practice prescribes specific features to be included in a mandatory quality control section of each standard test method that specify quality control requirements for that test method. Seven paragraphs are required in all standard test methods that address the following quality control practices: (1) verification of system calibration, (2) verification of control at zero analyte concentration, (3) verification of control at representative analyte concentration, (4) initial demonstration of proficiency, (5) assessment of precision, (6) assessment of bias, and (7) maintenance of interlaboratory traceability. If there are valid reasons why performance of any of the preceding practices are not mandatory for a specific test method, this must be documented in the appropriate paragraph of the quality control section of the test method.

#### 5. Significance and Use

5.1 To ensure that analytical results obtained from using any ASTM Committee D-19 test method are valid and accurate within the confidence limits required by the end user, quality control measures must be taken to confirm that the test method is meeting these requirements at the time of analysis. The quality control measures that are specified in this practice reflect the results of the interlaboratory study (see Practice D 2777) and are to be addressed in each ASTM test method to provide assurance that the test method is being performed up to its demonstrated capability.

#### 6. Verification of System Calibration

6.1 *Purpose*—The calibration of an instrument establishes its response characteristics over a concentration range. Reagent standardization usually establishes strength and assumes response is stoichiometric throughout the projected concentration range. The inherent precision of the calibration procedure must be considered in the preparation of these requirements.

6.2 *Frequency*, The test method must address the preparation of a calibration curve or a calibration/standardization check each day the test method is used.

6.3 *Calibration Curve*—The calibration section of a test method must include the preparation of a calibration curve and indicate the number of calibration standard concentrations required and address any additional criteria on the form of the curve. Frequencies for preparation of the full calibration curve are left to the test method writer's judgment of practicality.

6.4 *Calibration Check*—A single calibration standard at the start of the day can be used to verify that the most recently prepared full calibration curve is still useable. The requirement for and evaluation of a calibration check standard should appear in the quality control section. The test method writer should specify a concentration to be used and must specify acceptance criteria for each analyte being measured. The criteria should be relatively simple (for example, observed response within  $\pm 10\%$  of the anticipated response) and based upon historical data for calibrations (if available) or judgement. Corrective action must address the need for a new calibration curve or check.

6.5 Alternative calibration procedure permitted in the test method, such as internal standard, external standard, or single-point calibration procedures must be specified in the test method.

6.6 For certain complex test methods it may be appropriate to analyze a QC check sample (Section 8) at the start of the day and analyze a calibration check standard only when all QC check sample criteria are not met.

#### 7. Verification of Control at Zero Analyte Concentration

7.1 *Purpose*—Reagents must be demonstrated to be of sufficient high purity to permit their use without lowering the accuracy of the determination. Reagent water must be demonstrated to be free of interfering substances. Where practical, these two checks are combined into the analysis of a reagent water blank processed through the entire test method (method blank); corrective action for a contaminated method blank may include isolation of contamination through analysis of individual blanks for each reagent.

7.2 *Frequency*—The frequency of specified analysis of a method blank shall be once per day for test methods where the analyst normally prepares samples and analyzes them in one continuous time frame. For methods that involve laborious sample preparation (for example, pesticides, herbicides, etc.) and typically employ batch processing for sample preparation at one time and analysis at a different time or batch framework, or both, one method blank per sample preparation batch is required.

7.3 The test method writer shall designate how to handle measurable responses for analytes in method blanks. If the blank is much smaller than the statistical noise of the system it may be discountable. Responses caused by reagents are normally not tolerated and not used to correct results. Unavoidable responses in some closed measurement systems can be subtracted from all results by incorporating the method blank into a calibration curve prepared using procedural standards, or by subtracting the response for the blank from the response for samples. Unavoidable responses contributed by contaminants in reagent water may be used to correct calibration standards prepared from the same water, but are not normally used to correct organic measurements in samples.

#### 8. Verification of Control at Representative Analyte Concentration

8.1 *Purpose*—The analysis of a QC check sample is used to demonstrate, on an ongoing basis, that the laboratory is operating in control and the analytical test method is functioning correctly.

8.2 *Frequency*—The QC check sample should be analyzed on a frequency equivalent to at least 5 % of the sample workload.

8.3 The test method writer shall specify a concentration (C) for the test, but also reference this practice if the data requirements of the method user are focussed on a different concentration. The concentration selected shall fall within the range of concentrations used in the collaborative study, and could be chosen on the basis of its significance (for example, a common regulatory standard). It is generally desirable, however, to select either a concentration near the center of the