



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

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## 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

concentration range of the collaborative study or a reasonably low concentration well above (10 to 15 times) the test method detection limit.

8.4 The acceptance criteria for the control limits (*CL*) are calculated using the equation:

$$CL = X \pm 3 S_T$$

where:

*X* = the expected recovery defined by a function of *C* in the collaborative test, and

*S<sub>T</sub>* = the overall standard deviation projected for that concentration by the collaborative study.

8.5 Specify the corrective action to be taken in event of failure to meet the criteria. Recalibration should be considered. If practical, reanalysis of samples since the last calibration check or QC sample may be necessary.

8.6 For certain test methods, it may be appropriate to waive the requirement to analyze a QC check sample if the analyst can meet the equivalent criteria through the analysis of a spiked sample.

## 9. Initial Demonstration of Proficiency

9.1 *Purpose*—This demonstration is a requirement for the new test method user to confirm that he/she is capable of using the test method to generate meaningful data. It requires a statistically based comparison of data generated by the user to both the precision and bias characteristics established for the test method through collaborative testing (see Practice D 2777) and published in the method.

9.2 *Frequency*—A successful demonstration of capability is required at least once per analyst. The test method writer may indicate other occasions for repeating the test (for example, new instrument).

9.3 The analyte concentration for the proficiency test samples should be selected as described in 8.3.

9.4 The initial demonstration of proficiency with the test method should involve at least seven replicate analyses of the analyte prepared in the reference matrix used in the collaborative test (for example, reagent water). Fewer replicates may be appropriate for test methods that are very time consuming (for example, for a given method it may be desirable to complete the demonstration in one day but seven replicates could not be completed in that time period). Since the power of the demonstration is based upon the number of replicates, additional replication should be considered for tests that require a relatively small effort to perform.

9.5 *Calculation of Maximum Acceptable Limit for Precision:*

9.5.1 For the concentration (*C*) of an analyte, estimate the single operator standard deviation (*S<sub>o</sub>*) from the tabulated summary statistics derived for the reference matrix in the collaborative study or from an equation that defines *S<sub>o</sub>* as a function of *C*. If the collaborative study did not produce an estimate for *S<sub>o</sub>*, calculate an estimate for overall standard deviation (*S<sub>T</sub>*) for the concentration, then divide by 1.5 to estimate *S<sub>o</sub>*. Collaborative study results from similar test methods may be used to estimate a more appropriate relationship between *S<sub>T</sub>* and *S<sub>o</sub>*.

9.5.2 If the proficiency demonstration is performed at the

mean concentration of a Youden pair used in the interlaboratory study the number of degrees of freedom for the *S<sub>o</sub>* estimate at that concentration (*df<sub>sc</sub>*) is equal to the number of pairs of data used to calculate the estimate. If the test concentration was not used in the study, *S<sub>o</sub>* at the test concentration is estimated from interpolation of two or more estimates from the study and *df<sub>sc</sub>* is equal to the average number of pairs of data used for the individual *S<sub>o</sub>* estimates. The degrees of freedom of the analyst's proficiency demonstration (*df<sub>sm</sub>*) is one less than the number of replicates required.

9.5.3 Calculate the maximum acceptable standard deviation (*S<sub>m</sub>*) based on an *F*-test at the 1 % significance level according to the equation:

$$S_m = \sqrt{F_{0.99}} (S_o)$$

where *F* is based on the number of replicates and can be determined using Table 1.

9.6 *Calculation of Acceptance Limits for Bias*—The acceptance limits for the mean of *n* replicate recovery measurements are established using a two-sided Student's *t*-test:

$$X \pm t_{0.99} \sqrt{S_t^2 - \frac{(n-1)S_o^2}{n}}$$

where:

*t<sub>0.99</sub>* = Student's *t* for a two-tailed test at the 99 % confidence level at *N* – 1 degrees of freedom (see Table 2), and

*N* = the number of laboratories that provided usable data in the collaborative study.

9.7 Specify corrective action to be taken in case of failure. Requirements to repeat the test may consider the probability of random failure due to a large number of analytes and the small number of test sample replicates and require the retest only for those analytes for which the analyst failed to meet the criteria.

## 10. Assessment of Precision

10.1 *Purpose*—The replicate analyses of samples is for the laboratory to ensure that the test method is performing the job for which it was intended and to demonstrate that provisions in the test method to address matrix effects, including subsampling procedures in the laboratory, are being properly implemented.

10.2 *Frequency*—The assessment of precision in a sample matrix should be conducted with a frequency equivalent to 5 % of the sample workload and samples should be selected in a manner that will evaluate all representative matrices in a reasonable time period.

10.3 When a high frequency of non-detects are expected, spiked replicates should be used to assess precision.

10.4 Calculate relative standard deviation estimates from duplicate results using the following formula:

$$\text{relative standard deviation, \%} = \left(\frac{R}{\bar{X}}\right) \left(\frac{100}{\sqrt{2}}\right)$$

**TABLE 1 Critical Values of F at 1 % Significance (99 % Confidence) Level**

Degrees of Freedom for Proficiency Demonstration											
Degrees of Freedom for Interlaboratory Study	1	2	3	4	5	6	7	8	9	10	12
1	4052	4999	5403	5625	5764	5859	5928	5981	6022	6056	6106
2	98.50	99.00	99.17	99.25	99.30	99.33	99.36	99.37	99.39	99.40	99.42
3	34.12	30.82	29.46	28.71	28.24	27.91	27.67	27.49	27.34	27.23	27.05
4	21.20	18.00	16.69	15.98	15.52	15.21	14.98	14.80	14.66	14.54	14.37
5	16.26	13.27	12.06	11.39	10.97	10.67	10.46	10.29	10.16	10.05	9.89
6	13.74	10.92	9.78	9.15	8.75	8.47	8.26	8.10	7.98	7.87	7.72
7	12.25	9.55	8.45	7.85	7.46	7.19	6.99	6.84	6.72	6.62	6.47
8	11.26	8.65	7.59	7.01	6.63	6.37	6.18	6.03	5.91	5.81	5.67
9	10.56	8.02	6.99	6.42	6.06	5.80	5.61	5.47	5.35	5.26	5.11
10	10.04	7.56	6.55	5.99	5.64	5.39	5.20	5.06	4.94	4.85	4.71
11	9.65	7.21	6.22	5.67	5.32	5.07	4.89	4.74	4.63	4.54	4.40
12	9.33	6.93	5.95	5.41	5.06	4.82	4.64	4.50	4.39	4.30	4.16
13	9.07	6.70	5.74	5.20	4.86	4.62	4.44	4.30	4.19	4.10	3.96
14	8.86	6.51	5.56	5.04	4.69	4.46	4.28	4.14	4.03	3.94	3.80
15	8.68	6.36	5.42	4.89	4.56	4.32	4.14	4.00	3.89	3.80	3.67
16	8.53	6.23	5.29	4.77	4.44	4.20	4.03	3.89	3.78	3.69	3.55
17	8.40	6.11	5.18	4.67	4.34	4.10	3.93	3.79	3.68	3.59	3.46
18	8.28	6.01	5.09	4.58	4.25	4.01	3.84	3.71	3.60	3.51	3.37
19	8.18	5.93	5.01	4.50	4.17	3.94	3.77	3.63	3.52	3.43	3.30
20	8.10	5.85	4.94	4.43	4.10	3.87	3.70	3.56	3.46	3.37	3.23
21	8.02	5.78	4.87	4.37	4.04	3.81	3.64	3.51	3.40	3.31	3.17
22	7.95	5.72	4.82	4.31	3.99	3.76	3.59	3.45	3.35	3.26	3.12
23	7.88	5.66	4.76	4.26	3.94	3.71	3.54	3.41	3.30	3.21	3.07
24	7.82	5.61	4.72	4.22	3.90	3.67	3.50	3.36	3.26	3.17	3.03
25	7.77	5.57	4.68	4.18	3.86	3.63	3.46	3.32	3.22	3.13	2.99
26	7.72	5.53	4.64	4.14	3.82	3.59	3.42	3.29	3.18	3.09	2.96
27	7.68	5.49	4.60	4.11	3.78	3.56	3.39	3.26	3.15	3.06	2.93
28	7.64	5.45	4.57	4.07	3.75	3.53	3.36	3.23	3.12	3.03	2.90
29	7.60	5.42	4.54	4.04	3.73	3.50	3.33	3.20	3.09	3.00	2.87
30	7.56	5.39	4.51	4.02	3.70	3.47	3.30	3.17	3.07	2.98	2.84
40	7.31	5.18	4.31	3.83	3.51	3.29	3.12	2.99	2.89	2.80	2.66
60	7.08	4.98	4.13	3.65	3.34	3.12	2.95	2.82	2.72	2.63	2.50
120	6.85	4.79	3.95	3.48	3.17	2.96	2.79	2.66	2.56	2.47	2.34
∞	6.63	4.61	3.78	3.32	3.02	2.80	2.64	2.51	2.41	2.32	2.18

**TABLE 2 Student's T for Two-Tailed Test at 99 % Confidence Level <sup>A</sup>**

Degrees of Freedom	t Value	Degrees of Freedom	t Value
1	63.657	21	2.831
2	9.925	22	2.819
3	5.841	23	2.807
4	4.604	24	2.797
5	4.032	25	2.787
6	3.707	26	2.779
7	3.499	27	2.771
8	3.355	28	2.763
9	3.250	29	2.756
10	3.169	30	2.750
11	3.106	40	2.704
12	3.055	50	2.678
13	3.012	60	2.660
14	2.977	120	2.617
15	2.947	∞	2.576
16	2.921	...	...
17	2.898	...	...
18	2.878	...	...
19	2.861	...	...
20	2.845	...	...

<sup>A</sup> Youden, W. J., *Statistical Methods for Chemists*, John Wiley & Sons, New York, NY.

where:

$R$  = range of duplicates, and

$X$  = average of duplicates.

10.5 The statistical derivation of acceptance criteria for the range of duplicates using  $S_o$  estimates from the collaborative

study are generally not warranted because of the difficulty in developing appropriate summary statistics and the lack of sufficient degrees of freedom for a critical assessment. The test method should suggest guidelines for the agreement of duplicates and suggest corrective actions for when results are outside these guidelines. The analyst should be encouraged to use ranges developed internally to construct control charts as described in Guide D 3856 and Practice D 4210.

### 11. Assessment of Bias

11.1 *Purpose*—The quality control section shall require that a portion of all samples be spiked with the analytes of interest to ensure that the test method is performing the job for which it was intended and to demonstrate that provisions in the test method to address matrix effects are being properly implemented. Generally, the expectation is that the test method will perform as well, or nearly as well, on the sample as it does on a QC sample. For additional details on spiking procedures, see Guide D 5788.

11.2 *Frequency*—The spiking of a sample matrix should be conducted with a frequency equivalent to 5 % of the sample workload and samples should be selected in a manner that will evaluate all representative matrices in a reasonable time period. The laboratory should be encouraged to maintain a data base of bias estimates for each analyte and matrix type.

11.3 When background analyte concentrations are expected

to be low, the concentration for the spike should be the same used in 8.3 and 9.3. Depending upon the method and sample holding times, it may be reasonable to require analysis of the unspiked sample prior to selecting the spike. This should be done to prevent the addition of spikes lower than the background concentrations in the sample. If spike concentrations are close to analyte concentrations in the unspiked sample, the resulting bias estimates will be more variable than is representative of the actual performance of the method.

11.4 Calculate bias, as percent recovery, using the following formula:

$$\text{recovery, \%} = \left( \frac{A - B}{C} \right) 100$$

where:

- A = concentration found in spiked sample,
- B = concentration found in unspiked sample, and
- C = concentration of analyte added in spiked sample.

11.4.1 Allowance must be made for any significant dilution of the sample. If the unspiked sample was essentially free of analyte or the spike-to-background ratio of concentrations was ten or more, the percent recovery obtained should fall within the control limits described in 8.4.

11.5 Specify the action required when spike recoveries are outside of established limits. It may be necessary to analyze a QC sample or to recalibrate.

11.6 If water samples are not complex and analyte concentrations are usually very low, the spiked sample and QC sample become redundant. In this case, it may be appropriate to reduce the nominal frequency for each test.

**12. Maintenance of Interlaboratory Traceability**

12.1 *Purpose*—The periodic analysis of a certified reference material (CRM) and participation in interlaboratory proficiency studies are used to provide an independent verification of calibration and quantification practices. The laboratory results become indexed or traceable to a national or international standard of performance.

12.2 *Frequency*—The test method should specify analysis of a CRM or participation in an interlaboratory proficiency study on a quarterly basis, or both.

12.3 Certified reference materials from the National Institute of Standards and Technology (NIST), or similar reference material from other agencies or reputable commercial sources may be used. Results from analysis of the independent reference material must be within the control limits specified by the outside source if available; otherwise either the criteria in 6.4 or 8.4 as appropriate. Refer to Guide D 3856 for further information on external calibration checks.

**13. Keywords**

13.1 blanks; calibration; organics in water; quality control; reference material; spiked samples

**APPENDIX**

**(Nonmandatory Information)**

**X1. REFERENCE STATISTICS FROM THE INTERLABORATORY METHOD STUDY**

X1.1 The example in this appendix uses the interlaboratory study data for 2-pentanol, determined with other volatile alcohols in water by direct aqueous injection gas chromatography using Test Method D 3695. Six laboratories ( $N = 6$ ) participated in the interlaboratory study following the guidance of Practice D 2777. The degrees of freedom for reagent water in the historical interlaboratory study was 12: (laboratories  $\times$  concentration levels) – (laboratories) = (6  $\times$  3) – 6 = 12. For the analyte 2-pentanol, the range of the three concentrations studied was 39 to 197 mg/L. The study showed that test method bias was negligible, as the mean concentration found,  $X$ , was not significantly different than the true concentration, or fortification level,  $C$ , and the overall precision ( $S_T$ ) and the single operator precision ( $S_o$ ) for reagent water over this range could be related to  $X$  by regression equations:

$$X = C \tag{X1.1}$$

$$S_T = 0.04X - 0.007 \text{ mg/L} \tag{X1.2}$$

$$S_o = 0.009 X + 0.25 \text{ mg/L} \tag{X1.3}$$

*X1.2 Design for the Quality Control Sample Analyses:*

X1.2.1 The quality control requirements for a method are

usually established before the interlaboratory study to ensure the study will produce method statistics that can be used to establish performance-based criteria for the QC requirements. In this example, the task group has decided that the most representative concentration ( $C$ ) to monitor laboratory control of 2-propanol is 100 mg/L. It is also determined that seven replicates would be used for the initial demonstration of proficiency. The degrees of freedom for the proficiency demonstration is  $n - 1 = 6$ .

X1.2.2 The precision and bias estimates for 2-propanol at  $C = 100$  mg/L are calculated from equations X1.1 to X1.3:

$$X = 100 \text{ mg/L} \tag{X1.4}$$

$$S_T = 0.04(100) - 0.007 = 3.99 \text{ mg/L} \tag{X1.5}$$

$$S_o = 0.009 X + 0.25 \text{ mg/L} = 1.15 \text{ mg/L} \tag{X1.6}$$

X1.2.3 The acceptance criteria for the verification of control at the representative concentration are calculated as  $X \pm 3 S_T$  or  $100 \pm 3(3.99) = 88.0 - 112.0$  mg/L.

*X1.3 Calculation of Precision and Bias Criteria for the Initial Demonstration of Proficiency:*

X1.3.1 *Precision*—From Table 1, the value of  $F$  for  $6 \times 12$

df is 4.82. The maximum acceptable standard deviation,  $S_m$ , is calculated as  $(4.82)^{1/2} \times 1.15$  mg/L, that is, 2.52 mg/L.

X1.3.2 *Bias (As Recovery)*—From Table 2, the Student's  $t$  for 6 df is 3.71. The acceptance limits for a 100 mg/L test concentration is:

$$100 \pm 3.71 \sqrt{(3.99)^2 - \frac{(1.15)^2}{7}} \quad (\text{X1.7})$$

or 85.3 to 114.7 mg/L.

X1.3.3 The final design for the proficiency demonstration requires the analyst to analyze seven replicates of a solution of 2-pentanol at 100 mg/L. The mean recovery and standard deviation of the seven test results are calculated in milligrams per litre and compared to the acceptance limit. If the mean recovery of the seven results is 85.3 – 114.7 mg/L and the standard deviation is less than 2.06 mg/L, the analyst has successfully demonstrated that the instrument and operator are functioning at the level expected for the test procedure. Example language for inclusion in the standard is as follows:

X1.3.3.1 *Initial Demonstration of Proficiency*—If the analyst has not performed the test procedure before, a precision and accuracy study must be performed to demonstrate analyst proficiency. Analyze seven replicates of a standard solution prepared from a CRM containing 100 mg/L of each analyte of

interest in Table X1.1. Each replicate must be taken through the complete analytical test method. Calculate the mean ( $X$ ) and standard deviation ( $S_o$ ) of these values and compare to the maximum acceptable standard deviation and acceptance range for mean recovery in Table X1.1. If the criteria for  $X$  and  $S_o$  are not met for all analytes of interest, this demonstration must be repeated until the mean and standard deviation are within acceptable limits.

**TABLE X1.1 Criteria for Quality Control Requirements**

NOTE 1—These criteria are derived from the interlaboratory precision and bias data presented in 12.1. Refer to this practice for directions for developing acceptance criteria from these data if a different analyte concentration is more suitable for this demonstration.

Analyte	Test Concentration	QC Check	Proficiency Demonstration	
		Acceptance Range for QC Check Sample	Maximum Acceptable Standard Deviation	Acceptance Range for Mean Recovery
Isopropanol	100 mg/L	...	...	...
2-Pentanol	100 mg/L	88.0 to 112.0 mg/L	2.06 mg/L	85.3 to 114.7 mg/L
1-Pentanol	100 mg/L	...	...	...
1-Hexanol	100 mg/L	...	...	...
2-Ethyl hexanol	100 mg/L	...	...	...

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