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Standard Test Method for Sulfur in Automotive, Heating, and Jet Fuels by Monochromatic Energy Dispersive X-ray Fluorescence Spectrometry[1]

This standard is issued under the fixed designation D7220; 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 test method specifies an energy-dispersive X-ray fluorescence (EDXRF) method for the determination of total sulfur in automotive, No. 2 heating, and jet fuels with a concentration range of 3 mg/kg to 942 mg/kg.

1.1.1 The pooled limit of quantitation of this test method as obtained by statistical analysis of inter laboratory test results is 3 mg/kg sulfur.

1.1.2 This test method is applicable to gasoline, oxygen enriched gasoline (RFG), diesel, diesel/biodiesel blends containing up to twenty volume percent biodiesel, kerosene, jet fuel, jet fuel/biodiesel blends containing up to five volume percent biodiesel and No. 2 home heating oil.

1.2 A fundamental assumption in this test method is that the standard and sample matrix is well matched. Matrix mismatch can be caused by C/H ratio differences between samples and standards or by the presence of other heteroatoms.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 The preferred concentration units are mg/kg sulfur.

1.4 *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.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

3. Terminology

3.1 *Definitions:*

3.1.1 *monochromatic X-radiation, n*—an incident X-ray beam on a sample having a selected photon energy with a narrow energy bandwidth of ±5 % relative to the selected energy.

3.1.1.1 *Discussion*—Monochromatic X-ray radiation in EDXRF instrumentation can be obtained by using Bragg optics (at an angle of è = 45 ± 5°, in the low energy range). Bragg optics (monochromators) create very intense mono-energetic radiation. A combination of a selected X-ray tube (typically a Pd or Ag anode) with a highly ordered pyrolytic graphite (HOPG) Bragg optic can be used to create monochromatic radiation of the characteristic radiation of the anode material of the X-ray tube. The use of such radiation for sample excitation results in increased sensitivity for the determination of sulfur in petroleum products.

3.2 *Abbreviations:*

3.2.1 *DBS*—actual mass of Di-*n*-butyl sulfide, g

3.2.2 *Kcps*—kilo-counts per second.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.3 *EDXRF*—Energy dispersive X-ray spectrometry

3.2.4 *PTFE*—Polytetrafluorethylene

3.2.5 *SDBS*—mass % of sulfur in Di-*n*-butyl sulfide, typically 21.91 %

3.2.6 *SStd*—mg/kg sulfur in the calibration standard

3.2.7 *SStock*—mg/kg of sulfur in the stock standard

3.2.8 *STK*—actual mass of stock standard, g

4. Summary of Test Method

4.1 The sample is placed in the monochromatic X-ray beam, and the peak area of the sulfur Kα line at 2.307 keV is measured. The background spectrum, measured with a sulfur free white oil or other matrix matching blank sample (see 8.4) is adapted to the measured spectrum using adjustment regions following the instrument manufacturer's instructions and then subtracted from the measured spectrum. The resultant net counting rate is then compared to a previously prepared calibration curve or equation to obtain the concentration of sulfur in mg/kg. (**Warning—**Exposure to excessive quantities of X-radiation is injurious to health. The operator needs to take appropriate actions to avoid exposing any part of their body, not only to primary X-rays, but also to secondary or scattered radiation that might be present. The X-ray spectrometer should be operated in accordance with the regulations governing the use of ionizing radiation.)

5. Significance and Use

5.1 This test method provides measurement of total sulfur in automotive, No. 2 heating, and jet fuels with a minimum of sample preparation. A typical analysis time is 180 s to 360 s per sample.

5.2 The quality of automotive, No. 2 heating, and jet fuel can be related to the amount of sulfur present. Knowledge of sulfur concentration is necessary for processing purposes. There are also regulations promulgated in federal, state, and local agencies that restrict the amount of sulfur present in some fuel.

5.3 If this test method is applied to petroleum materials with matrices significantly different from the calibration materials specified in this test method, the cautions and recommendations in Section 6 should be observed when interpreting the results.

6. Interferences

6.1 When the elemental composition (excluding sulfur) of samples differs significantly from the standards, errors in the sulfur determination can result. For example, differences in the carbon-hydrogen ratio of sample and calibration standards introduce errors in the determination.

6.2 M-85 and M-100 are fuels containing 85 % and 100 % methanol, respectively. They have a high oxygen content leading to significant absorption of sulfur Kα radiation. Such fuels can, however, be analyzed using this test method provided either that correction factors are applied to the results (when calibrating with white oils) or that the calibration standards are prepared to match the matrix of the sample.

6.3 In general, petroleum materials with compositions that vary from the calibration samples as specified in Section 11 can be analyzed with standards made from base materials that are of the same or similar composition. Thus a gasoline may be simulated by mixing *iso*octane and toluene in a ratio that approximates the expected aromatic content of the samples to be analyzed. Standards made from this simulated gasoline can produce results that are more accurate than results obtained using white oil standards.

7. Apparatus

7.1 *Monochromatic X-ray Fluorescence Analyzer*—A Monochromatic Excitation Energy Dispersive XRF spectrometer may be used if its design incorporates as a minimum, the following features:

7.1.1 *Source of X-ray Excitation,* X-ray end window tube with Ag or Pd anode, in combination with HOPG Bragg monochromating X-ray optics. The monochromator must produce monochromatic Ag or Pd L radiation. Other anode materials and monochromators may be utilized, however stated precision and bias may not apply.

7.1.2 *Sample Cell,* providing a sample depth of at least 4 mm and equipped with replaceable X-ray transparent film window.

7.1.3 *X-ray Detector,* with a resolution value not to exceed 175 eV at 5.9 Kcps (10 000 cps). A Si drift chamber has been found suitable for use. Using a detection system with this minimum spectral resolution has been shown to eliminate the potential effect of interference from chlorine on sulfur should either salt contamination, or chlorine from other sources (for example, recycled vegetable oils) occur.

7.1.4 *He-flush,* the system must allow flushing of the optical path with helium (see 8.6). Alternatively, a vacuum of ≤4.0 kPa (≤30.4 Torr) is applied to the optical path.

7.1.5 *Signal Conditioning and Data Handling Electronics,* including the functions of X-ray intensity counting, spectra handling by background subtraction and deconvolution, calculation of overlap corrections and conversion of sulfur X-ray intensity into mg/kg sulfur concentration.

8. Reagents and Materials

8.1 *Purity of Reagents*[3]—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Di-n-butyl Sulfide*—a high-purity standard with a certified analysis for sulfur content. Use the certified sulfur content when calculating the exact concentrations of the calibration

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[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

standards (see 11.1). (**Warning—**Di- *n*-butyl sulfide is both flammable and toxic.)

NOTE 1—It is essential to know the concentration of sulfur in the di-*n*-butyl sulfide, not the purity, since impurities may also be sulfur containing compounds.

8.3 *Drift Correction Monitor(s) (Optional)*—Several different materials have been found to be suitable for use as drift correction monitors. Examples of sulfur containing materials that have been found to be suitable include a renewable liquid petroleum material or a fused glass disk. The monitor's count rate, in combination with count time, shall be sufficient to give a relative counting error of less than 1 %. The count rate for the monitor sample is determined during calibration (see 11.4) and again at the time of analysis (see 12.1). These counting rates are used to calculate a drift correction factor (see 13.1).

8.3.1 Drift correction is usually implemented automatically in software, although the calculation can readily be done manually. For X-ray instruments that are highly stable, the magnitude of the drift correction factor may not differ significantly from unity.

8.4 *White Oil (Light Paraffin Oil) or Matrix-Matching Blank Sample,* with a certified content of less than 0.2 mg⁄kg sulfur. If only one matrix is to be analyzed (for example, diesel) accuracy of results may be improved by using a matrix matched diluent. In these cases, the matrix matched diluent should match approximately the C/H ratio and oxygen content of the material to be analyzed.

8.5 *X-ray Transparent Film*—Any film that resists attack by the sample, is free of sulfur, and is sufficiently X-ray transparent can be used, with film thickness of between 2 µm to 6 µm. Films can include polyester, polypropylene, polycarbonate, and polyimide. Typically polycarbonate with a thickness of 5 µm to 6 µm is used. However, samples of high aromatic content can dissolve polyester and polycarbonate films. It is important that samples, standards, and blanks be measured using the same batch of film to avoid bias.

8.6 *Helium Gas,* minimum purity 99.9 %.

8.7 *Sample Cells,* compatible with the sample and the geometry requirements of the spectrometer. Disposable cells are recommended.

8.8 *Calibration Check Samples,* portions of one or more liquid petroleum or product standards of known sulfur content and not used in the generation of the calibration curve. The check samples shall be used to determine the accuracy of the initial calibration (see 11.5).

8.9 *Quality Control Samples,* stable petroleum or product samples representative of the samples of interest that are run on a regular basis to verify that the system is in statistical control.

NOTE 2—Verification of system control through the use of QC samples and control charting is highly recommended. It is recognized that QC procedures are the province of the individual laboratory. Suitable QC samples can often be prepared by combining retains of typical samples.

9. Sampling, Test Specimens, and Test Units

9.1 Samples shall be taken in accordance with the instructions in Practices D4057 or D4177 when applicable.

9.2 When reusable sample cells are used, clean and dry cells before each use. Disposable sample cells shall not be reused. For each sample, an unused piece of X-ray film is required for the sample cell. Avoid touching the inside of the sample cell, the portion of the window film in the cell, or the instrument window that is exposed to X-rays. Oil from fingerprints can affect the reading when determining low levels of sulfur. Wrinkles in the film will affect the intensity of the sulfur X-rays transmitted. Therefore, it is essential that the film be taut and clean to ensure reliable results. When handling the window film, avoid touching the central part (the part that actually forms the optical window) as this can lead to contamination from sweat, grease, or other petrochemical products. Similarly, discard any film that has been exposed to the atmosphere (for example, hanging outside of the film roll dispensing box). Also, when opening a new roll of film, discard the first metre, since some films are packaged in plastic bags that contain sulfur. The analyzer may need recalibration if the type or thickness of the window film is changed. After the sample cell is filled, make a small vent hole in the lid of the sample cell. Place the sample in the cell using techniques consistent with good practice for the particular instrument being used. Although sulfur radiation will emerge from only a small distance into the sample, scatter from the sample cell and the sample can vary. Laboratory personnel shall ensure that the sample cell is filled above a minimum depth, beyond which additional sample does not significantly affect the count rate. Generally, fill the sample cell to a minimum of three-fourths of the cell's capacity.

9.3 Consider sample reactivity, volatility and level of sulfur to be determined when preparing multiple samples for analysis or when an auto-sampler is utilized. Examples, analyze volatile samples, such as gasoline or samples with low (< 50 mg/kg) sulfur content directly after the sample cell is sealed (see 9.2). Follow recommendations to utilize techniques consistent with good practice for the sulfur concentration range, sample matrix type, sample cell, window film and the apparatus being used (see 8.5, 8.7, 9.2, 9.4, and 9.5).

9.4 If the instrument is equipped with a replaceable secondary/safety window, it must be replaced with every sample to ensure there is no cross-contamination. When changing the window, follow the precautions given in 9.2.

9.5 Impurities or thickness variations, which may affect the determination of low levels of sulfur, have been found in polyester films and may vary from lot to lot. Therefore, the test method shall be verified after starting each new roll or batch of film.

9.6 When connecting a new helium gas cylinder, always run a blank measurement to ensure the helium gas line is purged of air.

10. Preparation of Apparatus

10.1 Set up the apparatus in accordance with the manufacturer's instructions. Whenever possible, the instrument should remain energized to maintain optimum stability.

11. Calibration and Standardization

11.1 *Preparation of Calibration Standards:*

TABLE 1 Nominal Composition of Stock Standard

Sulfur Content, mg/kg	Mass of White Oil or Matrix-Matching Blank, g	Mass of Di-*n*-butyl Sulfide, g
2498	197.72	2.28

11.1.1 *Preparation of Stock Calibration Standard*—Accurately weigh white oil or matrix-matching blank sample to the nearest 0.1 mg as shown in Table 1 into a suitable narrow-necked container, and then accurately weigh in the nominal quantity of Di-*n*-butyl sulfide. Mix thoroughly (a PTFE coated stirrer is advisable) at room temperature. Calculate the concentration in the stock standard to 1 mg/kg using the following equation:

$$SStock = \left[DBS \times SDBS \div (DBS + Blank)\right] \times 1000 \quad (1)$$

where:

SStock = mg/kg of sulfur in the stock standard,
DBS = actual mass of Di-*n*-butyl sulfide, g,
SDBS = the mass % of sulfur in Di-*n*-butyl sulfide, typically 21.91 %, and
Blank = actual mass of white oil or matrix-matching blank sample.

11.1.2 *Preparation of Calibration Standard*—Accurately weigh the nominal quantity of white oil or matrix-matching blank to the nearest 0.1 mg as shown in Table 2 into a suitable narrow-necked container, and then accurately weigh in the nominal quantity of stock standard. Mix thoroughly (a PTFE coated stirrer is advisable) at room temperature. Calculate the concentration of sulfur in each standard to 0.1 mg/kg using the following equation:

$$SStd = STK \times SStock \div (STK + Blank) \quad (2)$$

where:

SStd = mg/kg sulfur in the calibration standard, and
STK = actual mass of stock standard, g.

NOTE 3—Commercially available standards can be used provided their sulfur concentrations are accurately known and they approximate the nominal concentrations listed in Table 2.

NOTE 4—If desired, additional standards can be analyzed with concentrations between those listed in Table 2.

11.1.3 *Storage of Standards and QC Samples*—Store all standards and QC samples in glass bottles in a cool, dark place until required. The glass bottles shall either be dark or wrapped in opaque material, and closed with glass stoppers, inert plastic lined screw cups, or impermeable enclosures, As soon as any sediment or change of concentration is observed, discard the sample.

11.2 Establish calibration curve data by carefully measuring the net intensity of the emitted sulfur radiation from each of the standards by the procedure described in Sections 11 and 12.

11.3 *Calibration Model*—Construct a calibration model by either:

11.3.1 Using the software and algorithms supplied by instrument manufacturer, or

11.3.2 Fitting the data to the following equation:

$$S = a \times R + b \quad (3)$$

where:

S = sulfur concentration in mg/kg,
R = net intensity for the sulfur radiation,
a = slope, and
b = intercept.

11.4 When using drift correction monitors, determine the intensity of the drift correction monitor sample(s) during the calibration procedure. The value determined corresponds to the factor *A* in 13.1.

11.5 Immediately after completing the calibration, determine the sulfur concentration of one or more of the calibration check samples (see 8.8). The measured value shall be in the range defined by the certified concentration ± the repeatability of this test method. When this is not the case, the calibration or calibration standards are suspect, and corrective measures should be taken and the calibration rerun. The degree of matrix mismatch between samples and standards should also be considered when evaluating a calibration. If the calibration intercept *b* in 11.3.2 shows a negative value, this indicates a significant *S* concentration in the blank sample.

12. Procedure

12.1 When using drift correction monitors, before analyzing samples on a given day, analyze the drift correction monitor(s) and determine the counting rate, using the same material as used at the time of calibration. The value determined corresponds to the factor *B* in 13.1.

12.2 Place the sample in an appropriate cell using techniques consistent with good practice for the particular instrument being used. Although sulfur radiation will penetrate only a small distance into the sample, scatter from the sample cell and the sample can vary. Laboratory personnel have to ensure that the sample cell is filled above a minimum depth; beyond which additional sample does not significantly affect the count rate. Generally, fill the sample cell to a minimum of three-fourths of the cell's capacity. Make a small vent hole in the lid of the sample cell.

12.3 Place the sample in the instrument, and perform measurement according to instrument manufacturer's instructions.

12.4 Determine the peak area of the sulfur Kα radiation at 2.307 keV. The net count-rate will be calculated by subtracting the background. The background spectrum, measured with a sulfur-free white oil or other matrix matching blank sample (see 8.4) is adapted to the measured spectrum using adjustment regions following the instrument manufacturer's instructions and then subtracted from the measured spectrum.

NOTE 5—When using a Pd target X-ray tube, background adjustment regions at 2.86 keV to 2.92 keV and 2.95 keV to 3.04 keV have been found suitable.

12.5 Determine the corrected counting rate and calculate the concentration of the sample as described in Section 13.

NOTE 6—It is recommended to prepare and analyze each sample directly. Also, it is recommended to take care not to leave the test sample in the instrument after the measurement process has finished. If the spectrometer is equipped with an autosampler, it is recommended to use only one position of the autosampler. This minimizes the risks of sample

TABLE 2 Nominal Calibration Standards

Low Range Sulfur Content, mg/kg	Mass of White Oil or Matrix-Matching Blank, g	Mass of Stock Standard, g	High Range Sulfur Content, mg/kg	Mass of White Oil or Matrix- Matching Blank, g	Mass of Stock Standard, g
0	100.0	0.0	50	98.0	2.0
5	99.8	0.2	100	96.0	4.0
10	99.6	0.4	250	90.0	10.0
25	98.8	1.2	500	80.0	20.0
50	98.0	2.0	750	70.0	30.0
			1000	60.0	40.0

cell leakage, or deformation, or both.

13. Calculation or Interpretation of Results

13.1 When using the drift correction monitor described in 8.3, calculate a correction factor for changes in daily instrument sensitivity as follows:

$$F = A \div B \tag{4}$$

where:

A = counting rate of the drift correction monitor as determined at the time of calibration (see 11.4), and

B = counting rate of the drift correction monitor as determined at the time of analysis (see 12.1).

13.2 The net counting rate is given by the spectral deconvolution of the sulfur Kα line after subtracting the background spectrum.

13.2.1 The use of the factor F to correct the counting rate of sulfur is optional.

13.3 Calculate the sulfur content of the sample by inserting the corrected net counting rate in the chosen calibration model from Section 11. In many cases the instrument vendor will provide software or the required calculations.

14. Report

14.1 For all samples analyzed, report the result calculated in 13.3. Report the result as the total sulfur content in mg/kg rounding to two significant figures up to 10 mg⁄kg and to three significant figures for results greater or equal 10 mg/kg using Practice E29. State that the results were obtained in accordance with Test Method D7220.

15. Precision and Bias

15.1 The precision and bias of the test method, as determined by statistical examination of interlaboratory test results, [4,5] was determined according to Practice D6300. One of each certified diesel sample,[6] (NIST 2770), gasoline sample (NIST 2299), and kerosene sample (NIST 1616b) were provided as test samples (results not supplied) to determine the bias of the test method.

NOTE 7—The precision estimate was calculated by treating the entire ILS dataset as a statistical sampling from one population since analysis showed no statistically significant differences (95 % confidence level) between precision and mean estimates sub grouped by gasoline, diesel and kerosene materials.

NOTE 8—Volatile or reactive materials may not meet the stated precision of the method because selective loss of light materials is possible before and during analysis by this method. Another possible mechanism is the sulfur enrichment of the sample cup window resulting in higher sulfur values.

15.2 The precision of the test method is as follows:

15.2.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operation conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the value:

$$0.6430 \cdot X^{0.504} \text{ mg/kg} \tag{5}$$

where:

X = the mean sulfur content of two results between 3 and 942 mg/kg, only in one case in twenty. (See Table 3.)

15.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the value:

$$1.1121 \cdot X^{0.504} \text{ mg/kg} \tag{6}$$

where:

X = the mean sulfur content of two results between 3 and 942 mg/kg, only in one case in twenty. (See Table 4.)

15.3 *Bias*—One of each certified diesel sample (NIST 2770), gasoline sample (NIST 2299), and kerosene sample (NIST 1616b) were provided as blind test samples. The observed differences between the ILS determined averages and the ARV (Accepted Reference Values) of the NIST standards were not statistically significant at the 95% confidence level. See Table 5.

16. Keywords

16.1 automotive fuel; diesel; gasoline; petroleum; polarization; spectrometry; sulfur; X-ray

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[4] Supporting data (original 2006 ILS) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1592. Contact ASTM Customer Service at service@astm.org.

[5] Supporting data (monochromatic 2012 ILS) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1736. Contact ASTM Customer Service at service@astm.org.

[6] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

TABLE 3 Example Repeatability Values, All Sample Types

Sulfur Content, mg/kg	Repeatability, mg/kg
3	1.1
5	1.4
10	2.1
25	3.3
50	4.6
100	6.5
250	10.4
500	14.7
942	20.3

TABLE 4 Example Reproducibility Values, All Sample Types

Sulfur Content, mg/kg	Reproducibility, mg/kg
3	1.9
5	2.5
10	3.5
25	5.6
50	8.0
100	11.3
250	18.0
500	25.5
942	35.1

TABLE 5 Comparison of NIST and ASTM Interlaboratory Study (RR) Results

NIST SRM Number	Sulfur, mg/kg NIST	Matrix	Average Measured, mg/kg Sulfur ASTM ILS	Observed Difference, mg/kg Sulfur	Statistically Significant (95% Confidence Level)?
NIST 2770	41.57 (±0.39)	Diesel	41.8	0.2	No
NIST 2299	13.6 (±1.5)	Gasoline	12.5	-1.1	No
NIST 1616b	8.4 (±0.12)	Kerosene	8.9	0.5	No

APPENDIX

(Nonmandatory Information)

X1. QUALITY CONTROL INFORMATION

X1.1 The performance of the instrument or test procedure should be confirmed by analyzing quality control (QC) samples.

X1.1.1 As part of the QC procedure, a portion of the white mineral oil used to make up the calibration standards should be analyzed in order to confirm the instrument blank value has not changed since the initial calibration.

X1.2 Prior to routine use of this test method, the user should determine the average value and control limits of the QC samples. See Practice D6299 and MNL 7.[7]

X1.3 The QC results should be recorded and monitored by control charts or other statistically equivalent techniques to determine the statistical control status of the total testing process. See Practice D6299 and MNL 7.[7]

X1.4 The frequency of control testing is dependent on the criticality of the quality being measured, the demonstrated stability of the testing process, and customer requirements. Generally, a QC sample should be analyzed each testing day with routine samples. The QC frequency should be increased if a large number of samples are routinely analyzed. However, when it is demonstrated that the testing is under statistical control, the QC testing frequency may be reduced. The QC sample testing precision should be periodically checked against the ASTM method precision to ensure data quality. See Practice D6299 and MNL 7.[7]

X1.5 It is recommended that, if possible, the type of QC sample that is regularly tested be representative of the material routinely analyzed. An ample supply of QC sample material should be available for the intended period of use, and must be homogeneous and stable under anticipated storage conditions.

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[7] MNL 7, *Manual on Presentation of Data Control Chart Analysis*, 6th Edition, ASTM International, W. Conshohocken, PA.

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