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.

Designation: D7751 – 16 (Reapproved 2021)

Standard Test Method for Determination of Additive Elements in Lubricating Oils by EDXRF Analysis[1]

This standard is issued under the fixed designation D7751; 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 covers the quantitative determination of additive elements in unused lubricating oils and additive packages, as shown in Table 1.

1.2 Additive packages require dilution with a contamination free diluent (base oil) prior to analysis. The dilution factor has to be calculated from the expected concentrations to bring the concentrations for all elements into the ranges listed in Table 1.

1.3 Some lubrication oils will contain higher concentrations than the maximum concentrations listed in Table 1. These samples require dilution with a contamination free diluent (base oil) prior to analysis. The dilution factor has to be calculated from the expected concentrations to bring the concentrations for all elements into the ranges listed in Table 1.

1.4 This test method is limited to the use of energy dispersive X-ray fluorescence (EDXRF) spectrometers employing an X-ray tube for excitation in conjunction with the ability to separate the signals of adjacent elements by using a high resolution semiconductor detector.

1.5 This test method uses inter-element correction factors calculated from a fundamental parameters (FP) approach or from another matrix correction method.

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

1.6.1 The preferred concentration units are mg/kg or mass %.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *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, Liquid Fuels, and Lubricants

D6792 Practice for Quality Management Systems in Petroleum Products, Liquid Fuels, and Lubricants Testing Laboratories

D7343 Practice for Optimization, Sample Handling, Calibration, and Validation of X-ray Fluorescence Spectrometry Methods for Elemental Analysis of Petroleum Products and Lubricants

E1621 Guide for Elemental Analysis by Wavelength Dispersive X-Ray Fluorescence Spectrometry

2.2 *ISO Standards:*[3]

ISO 4259 Determination and application of precision data in relation to methods of test

3. Terminology

3.1 *Definitions:*

3.1.1 *energy dispersive X-ray spectrometry, n*—XRF spectrometry applying energy dispersive selection of radiation.

3.2 *Abbreviations:*

[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] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

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TABLE 1 Elements and Range of Applicability

Element	PLOQ in mass %	Max Concentration in mass %
Magnesium	0.018	0.10
Phosphorous	0.024	0.125
Sulfur	0.008	1.94
Chlorine	0.0007	0.05
Calcium	0.002	0.44
Zinc	0.040	0.143
Molybdenum	0.004	0.047

3.2.1 *EDXRF*—Energy Dispersive X-ray Fluorescence Spectrometry.

3.2.2 *FP*—Fundamental Parameters.

4. Summary of Test Method

4.1 A specimen is placed in the X-ray beam, and the appropriate regions of its spectrum are measured to give the fluorescent intensities of magnesium, phosphorus, sulfur, chlorine, calcium, zinc, and molybdenum. Other regions of the spectrum are measured to compensate for matrix variation. To optimize the sensitivity for each element or group of elements, a combination of optimized excitation and detection conditions (for example, different primary beam filters (7.1.3), secondary or polarization targets (7.1.4), and so forth) may be used. The measuring time should be kept as short as possible, typically under 10 min per specimen. Avoid using different measurement conditions that yield only marginally better results for a specific analyte. There may be a correction of measured intensities for spectral overlap.

4.1.1 Concentrations of the elements of interest are determined by comparison of these intensities against a calibration curve using a fundamental parameters (FP) approach, possibly combined with corrections from backscatter. The FP approach uses the physical processes forming the basis of X-ray fluorescence emission in order to provide a theoretical model for the correction of matrix effects. The correction term is calculated from first principle expressions derived from basic physical principles and contain physical constants and parameters that include absorption coefficients, fluorescence yield, primary spectral distribution and spectrometry geometry. The calculation of concentrations in samples is based on making successively better estimates of composition by an iteration procedure.

NOTE 1—The algorithm used for the procedure is usually implemented in the instrument manufacturer's software.

4.2 The EDXRF spectrometer is initially calibrated using a set of standards to collect the necessary intensity data. Each calibration line and any correction coefficient are obtained by a regression of this data, using the program supplied with the spectrometer. (**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 Lubricating oils are formulated with organo-metallic additives, which act, for example, as detergents, antioxidants, antifoaming, or antiwear agents, or a combination thereof. Some of these additives contain one or more of the following elements: magnesium, phosphorus, sulfur, chlorine, calcium, zinc, and molybdenum. This test method provides a means of determining the concentrations of these elements, which in turn provides an indication of the additive content of these oils.

5.2 Several additive elements and their compounds are added to the lubricating oils to give beneficial performance (Table 2).

5.3 Additive packages are the concentrates that are used to blend lubricating oils.

5.4 This test method is primarily intended to be used for the monitoring of additive elements in lubricating oils.

5.5 If this test method is applied to lubricating oils 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.

TABLE 2 Lubricants and Additive Materials

Element	Compounds	Purpose/Application
Calcium	Sulfonates, phenates	Detergent inhibitors, dispersants
Chlorine	Trace contaminants, cleaning agents	
Magnesium	Sulfonates, phenates	Detergent inhibitors
Molybdenum	Dialkylithiophosphate dialkyldithiocarbamate, other molybdenum complexes	Friction modifier additives
Phosphorus	Dithiophosphates, phosphates phosphites	Anti-rusting agents, extreme pressure additives, anti-wear
Sulfur	Base oils, sulfonates, thiophosphates, polysulfides and other sulfurized components	Detergents, extreme pressure additives, anti-wear
Zinc	Dialkyldithiophosphates, dithiocarbamates, phenolates carboxylates	Anti-oxidant, corrosion inhibitors, anti-wear additives, detergents, crankcase oils, hypoid gear lubricants, aircraft piston engine oils, turbine oils, automatic transmission fluids, railroad diesel engine oils, brake lubricants

6. Interferences

6.1 The additive elements found in lubricating oils will affect the measured intensities from the elements of interest to a varying degree. In general the X-radiation emitted by the element of interest can be absorbed by itself (self-absorption) or by the other elements present in the sample matrix. Also the X-radiation emitted from one element can further excite (enhance) another element. These inter-element effects are significant at concentrations varying from 0.03 % by mass, due to the higher atomic number elements (for example, molybdenum), to 1 % by mass, for the lower atomic number elements (for example, sulfur). If an element is present at significant concentrations and an inter-element correction for that element is not employed, the results can be low due to absorption or high due to enhancement.

6.2 Absorption and enhancements effects will be corrected by corrections from the FP approach or by other matrix correction models.

6.3 There can be spectral overlap of one element onto another, and the instrument must include correction procedures for any such overlaps.

7. Apparatus

7.1 *Energy Dispersive X-ray Fluorescent Spectrometer*—Any energy dispersive X-ray fluorescence spectrometer can be used if its design incorporates at least the following features:

7.1.1 *Source of X-ray Excitation*—X-ray tube with palladium, silver, rhodium, or tungsten target. Other targets may be suitable as well. The voltage of the X-ray tube shall be programmable between 4 kV and at least 30 kV for preferential excitation of elements or groups of elements.

7.1.2 *X-ray Detector*—Semiconductor detector with high sensitivity and a spectral resolution value not to exceed 175 eV at 5.9 keV.

7.1.3 *Primary Beam Filters (Optional)*—To make the excitation more selective and to reduce the intensity of background radiation.

7.1.4 *Secondary or Polarization Targets, or Both (Optional)*—To make the excitation more selective and to improve peak-to-background ratio.

7.1.5 *Signal Conditioning and Data Handling Electronics*—That include the functions of X-ray intensity counting, spectra handling by background variation correction, overlap corrections, inter-elements effects corrections, and conversion of X-ray intensity into concentration.

7.1.6 *Helium Purgeable Optical Path (Optional)*—Helium purge improves the sensitivity of low energy X-rays emitted from low atomic number elements (Z< 22).

7.1.7 *Sample Cells*—Providing a depth of at least 6 mm and equipped with replaceable X-ray transparent film.

7.1.8 *Sample Film*—Suitable films include polypropylene, polyester, and polycarbonate with thickness from 3.5 µm to 8 µm. A thick film may limit the performance for low atomic numbers (for example, Magnesium).

7.2 *Instrument Setting-Up Samples (Elemental Reference Samples) (Optional)*—To quantify spectral overlaps. These are not required when the instrument's software does include software to deconvolute spectra.

7.3 *Drift Correction Monitors (Optional)*—To correct for instrumental drift. At least two samples are necessary to correct both sensitivity and possible changes in the background. For each element and scatter region, there shall be one providing a count rate similar to samples from the upper end of the calibration and another providing a count rate as if from a blank. This last can be a blank oil. For the high concentration of each element, a glass disk, XRF fusion bead, or pressed pellet have all been found to be satisfactory. Elemental reference samples (7.2) may also be used.

7.3.1 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.

7.4 *Quality Control (QC) Samples*—Samples for use in establishing and monitoring the stability and precision of an analytical measurement system. Use homogeneous materials, similar to samples of interest and available in sufficient quantity to be analyzed regularly for a long period of time.

7.5 For additional information, also refer to Practice D7343.

8. Reagents and Materials

8.1 *Purity of Reagents*[4]—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 *Diluent Solvent*—A suitable solvent containing less than 10 mg/kg of sulfur and containing less than 1 mg/kg of metals as well as of all other elements of interest (for example, base oil). If diluted samples are analyzed at low levels of sulfur, a lower sulfur content of the diluent solvent should be used and must be corrected for when recalculating the concentrations for the original, not-diluted sample. The precision stated in this test method does not apply to diluted samples.

8.3 *Helium Gas*—Minimum purity 99.9 %.

8.4 *Calibration Standard Materials:*

8.4.1 Commercially available calibration solutions.

8.4.2 Certified concentration solutions, of liquid organometallic salts, the following standard materials can be used:

8.4.2.1 *Calcium 2-Ethylhexanoate,* approximately 12.3 % by mass calcium.

8.4.2.2 *Zinc Cyclohexanebutyrate,* approximately 16.2 % by mass zinc.

[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar 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.

8.4.2.3 *Bis(2-Ethylhexyl)Hydrogen Phosphate,* 97 % purity (approximately 9.62 % by mass phosphorus).

8.4.2.4 *Di-n-butyl Sulfide,* 97 % purity (approximately 21.9 % by mass sulfur).

8.4.2.5 *Magnesium-2-ethylhexoate,* (2.99 % magnesium).

8.4.2.6 *1-Chlorooctane,* 98 % purity, (23.9 % by mass chlorine).

8.4.2.7 Commercially available single element standard for molybdenum based on molybdenumsulfonate.

8.4.2.8 *Stabilizers,* 2-ethylhexanoic acid, 2-ethylamine, also proprietary stabilizer/chelating solutions are available commercially. Stabilizers shall be free of the additive element.

NOTE 2—In addition to the calibration standard materials identified in 8.4, single or multielement calibration standards can also be prepared from materials similar to the samples being analyzed, provided the calibration standards to be used have previously been characterized by independent primary (for example, gravimetric or volumetric) analytical techniques to establish the elemental concentration mass % levels.

9. Hazards

9.1 Occupational health and safety standards for X-rays and ionizing radiation shall be observed. It is also recommended that proper practices be followed as presented by most manufactures documentation or described in Guide E1621.

10. Sampling and Test Specs and Units

10.1 Samples shall be taken in accordance with the instructions in Practices D4057 or D4177. For sample handling, also refer to Practice D7343.

10.2 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 sample 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 analytes. Wrinkles in the film will affect the intensity of the 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. Discard film that has been exposed to the atmosphere (for example, hanging outside of the film roll dispensing box). The analyzer may need recalibration if the type or thickness of the window film is changed. Fill the sample cell to a consistent depth (sample cells typically have a fill mark), no lower than 4 mm. Refer to the manufacturer's instructions for the use of the sample cell if necessary. If the instrument is equipped with a replaceable secondary/safety window it must be replaced when damaged or contaminated. When determining low concentrations, it is recommended to replace the window prior to each measurement. When changing, it follows the precautions given in 10.2.

10.3 Impurities or thickness variations, which may affect the determination of low levels of analytes, have been found in polyester films and may vary from lot to lot. Therefore, the method shall be verified after starting each new roll or batch of film. (When opening a new roll of film, it may be recommended to discard the first meter as some films are packaged in plastic bags that contain sulfur.)

10.4 When connecting a new helium gas cylinder, always run a blank measurement to ensure the helium gas line is purged of air. When using QC samples, check the performance by running QC sample(s).

11. Preparation of Apparatus

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

12. Calibration and Standardization

12.1 *Preparation of Calibration Standards:*

12.1.1 Precisely weigh the organometallic solutions and phosphorus and sulfur solutions with the diluent solvent along with the appropriate stabilizer.

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

12.2 Establish calibration curve data by carefully measuring the intensity of the emitted radiation from each of the standards by the procedure described in Sections 12 and 13. The recommended X-ray lines for the elements' determination are listed in Table 3. Intensity for other regions of interest in the spectrum may also be measured in order to apply background corrections.

12.3 Construct a calibration model using the software and algorithms supplied by instrument manufacturer.

12.4 When using drift correction monitors, determine the intensity of the drift correction monitor sample(s) after the calibration procedure.

12.5 After completing the calibration, determine the concentrations of one or more of the QC samples (see 7.4). The measured value shall be within the control limits of the QC samples. See Practice D6299 for guidance to set up control limits. When this is not the case, the calibration or calibration standards are suspect and corrective measures should be taken and the calibration repeated.

13. Procedure

13.1 When using drift correction monitors, prior to analyzing samples on a given day, analyze the drift correction

TABLE 3 Recommended X-ray Lines for Individual Analysis

Element	Preferred Line
Magnesium	K-$L_{2,3}$ (Kα)
Phosphorous	K-$L_{2,3}$ (Kα)
Sulfur	K-$L_{2,3}$ (Kα)
Chlorine	K-$L_{2,3}$ (Kα)
Calcium	K-$L_{2,3}$ (Kα)
Zinc	K-$L_{2,3}$ (Kα)
Molybdenum	K-$L_{2,3}$ (Kα)

monitor(s) and determine the counting rate, using the same material as used at the time of calibration.

13.2 Analyze QC samples prior to analyzing a batch of samples, as described in 12.5.

13.3 When analyzing additive packages or lubricating oil with concentration outside the ranges listed in Table 1, dilute the sample with a contamination free diluent (base oil) prior to analysis. The dilution factor has to be calculated from the expected concentrations to bring the concentrations for all elements into the ranges listed in Table 1.

13.4 Place the sample in an appropriate cell using techniques consistent with good practice for the particular instrument being used. When filling the sample cup, follow procedure described in 10.2.

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

13.6 Determine the intensities for all elements of interest.

NOTE 3—It is recommended that each sample is prepared and analyzed immediately. Also, it is recommended that care be taken not to leave the test sample in the instrument after the measurement process has finished.

14. Calculation or Interpretation of Results

14.1 When using the drift correction monitor, calculate correction factors for changes in instrument sensitivity. Use these correction factors to calculate corrected intensities for the elements of interest.

14.2 Calculate the concentrations of the elements of interest by inserting the intensities in the calibration created in Section 12. In many cases the instrument vendor will provide software with the required calculations.

14.3 When analyzing a sample which has been diluted prior to analysis, correct the results with the dilution factor used for the dilution of the sample.

15. Report

15.1 For all samples analyzed, calculate and report the result in accordance with Section 14. Report the result as the total concentration in mass % to three significant figures (x.xx, 0.xxx) at levels greater than 0.1 %, to two significant figures (0.0xx) at levels greater than 0.01 %, and to one significant figure (0.00x) at levels equal or lower than 0.01 %. State that the results were obtained according to Test Method D7751.

16. Precision and Bias

16.1 *Precision*—The precision of this test method was determined by statistical analysis of the interlaboratory study (ILS) results. The ILS was conducted in 2016[5], and included 21 laboratories analyzing 16 samples (blended lubricating oils) in duplicates. The participating laboratories used a combination of FP and other matrix and interelements effects corrections. A variety of calibration standards was used.

16.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in Table 4 and Table 5 in only one case in twenty.

16.1.2 *Reproducibility*—The difference between successive test results obtained by different operators with different apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in Table 6 and Table 7 in only one case in twenty.

16.1.3 *Bias*—No information can be presented on the bias of the procedure in Test Method D7751 for measuring magnesium, phosphorus, sulfur, chlorine, calcium, zinc, and molybdenum, because no material having an accepted reference value was available in the interlaboratory study.

17. Keywords

17.1 additive elements; additives; calcium; chlorine; EDXRF; energy dispersive; lubricating oil; magnesium; molybdenum; phosphorus; spectrometry; sulfur; X-ray; XRF; zinc

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1846. Contact ASTM Customer Service at service@astm.org.

TABLE 4 Repeatability Equations

Element	Concentration Range, mass %	Repeatability, mass %
Magnesium	0.018 to 0.10	$0.02443 * (X+0.0009)^{0.4031}$
Phosphorous	0.024 to 0.125	$0.002403 * X^{0.1476}$
Sulfur	0.008 to 1.94	$0.01440 * X^{0.8482}$
Chlorine	0.0007 to 0.05	$0.003124 * X^{0.4269}$
Calcium	0.002 to 0.44	$0.008940 * X^{0.5148}$
Zinc	0.040 to 0.143	$0.01896 * X^{0.7789}$
Molybdenum	0.004 to 0.047	$0.08504 * X^{1.075}$

TABLE 5 Calculated Repeatability Values (all in mass %)

Concentration	Magnesium	Phosphorous	Sulfur	Chlorine	Calcium	Zinc	Molybdenum
0.002	...	...	...	0.0002	0.0004	...	...
0.004	...	...	...	0.0003	0.0005	...	0.0002
0.008			0.0002	0.0004	0.0007	...	0.0005
0.01	...	...	0.0003	0.0004	0.0008	...	0.0006
0.018	0.005	...	0.0005	0.0006	0.0011	...	0.0011
0.025	0.006	0.0014	0.0006	0.0006	0.0013	...	0.0016
0.04	0.007	0.0015	0.0009	0.0008	0.0017	0.0015	0.0027
0.05	0.007	0.0015	0.0011	0.0009	0.0019	0.0018	0.0034
0.10	0.010	0.0017	0.002	...	0.0027	0.0032	...
0.125	...	0.0018	0.002	...	0.0031	0.0038	...
0.140	...	0.0018	0.003	...	0.0032	0.0041	...
0.44	...	...	0.007	...	0.0059	...	...
0.75	...	...	0.011	...	...	...	...
1.000	...	...	0.014	...	...	...	...
1.94	...	...	0.025	...	...	...	...

TABLE 6 Reproducibility Equations

Element	Concentration Range, mass %	Repeatability, mass %
Magnesium	0.018 to 0.10	$0.05625 * (X+0.0009)^{0.4031}$
Phosphorous	0.024 to 0.125	$0.01009 * X^{0.1476}$
Sulfur	0.008 to 1.94	$0.06605 * X^{0.8482}$
Chlorine	0.0007 to 0.05	$0.02541 * X^{0.4269}$
Calcium	0.002 to 0.44	$0.02708 * X^{0.5148}$
Zinc	0.040 to 0.143	$0.08179 * X^{0.7789}$
Molybdenum	0.004 to 0.047	$0.1911 * X^{1.075}$

TABLE 7 Calculated Reproducibility Values (all in mass %)

Concentration	Magnesium	Phosphorous	Sulfur	Chlorine	Calcium	Zinc	Molybdenum
0.002	...	...	...	0.0018	0.0011	...	...
0.004	...	...	...	0.0024	0.0016	...	0.0005
0.008			0.0011	0.0032	0.0023	...	0.0011
0.01	...	...	0.0013	0.0036	0.0025	...	0.0014
0.018	0.011	...	0.0022	0.0046	0.0034	...	0.0025
0.025	0.013	0.0058	0.0029	0.0053	0.0041	...	0.0036
0.04	0.016	0.0062	0.0043	0.0064	0.0052	0.0067	0.0060
0.05	0.017	0.0064	0.0052	0.0071	0.0058	0.0079	0.0076
0.10	0.022	0.0071	0.0094	...	0.0083	0.0136	...
0.125	...	0.0074	0.0113	...	0.0093	0.0162	...
0.140	...	0.0075	0.0125	...	0.0098	0.0177	...
0.44	...	...	0.0329	...	0.0177	...	...
0.75	...	...	0.0517	...	...	...	...
1.000	...	...	0.0661	...	...	...	...
1.94	...	...	0.1159	...	...	...	...

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. See Practice D6792.

X1.1.1 As part of the QC procedure, also a portion of the base 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.[6]

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.[6]

[6] MNL7, *Manual on Presentation of Data and Control Chart Analysis*, ASTM International.

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 at least once each testing day with routine samples. The QC sample testing precision should be periodically checked against the ASTM method precision to ensure data quality. See Practice D6299 and MNL 7.[6]

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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