Designation: D 4951 – 02

An American National Standard

Standard Test Method for Determination of Additive Elements in Lubricating Oils by Inductively Coupled Plasma Atomic Emission Spectrometry[1]

This standard is issued under the fixed designation D 4951; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This test method covers the quantitative determination of barium, boron, calcium, copper, magnesium, phosphorus, sulfur, and zinc in unused lubricating oils and additive packages.

1.2 The precision statements are valid for dilutions in which the mass % sample in solvent is held constant in the range of 1 to 5 mass % oil.

1.3 The precision tables define the concentration ranges covered in the interlaboratory study. However, both lower and higher concentrations can be determined by this test method. The low concentration limits are dependent on the sensitivity of the ICP instrument and the dilution factor. The high concentration limits are determined by the product of the maximum concentration defined by the linear calibration curve and the sample dilution factor.

1.4 Sulfur can be determined if the instrument can operate at a wavelength of 180 nm.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *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 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)[2]

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards[3]

D 4628 Test Method for Analysis of Barium, Calcium, Magnesium, and Zinc in Unused Lubricating Oils by Atomic Absorption Spectrometry[3]

D 4927 Test Methods for Elemental Analysis of Lubricant and Additive Components—Barium, Calcium, Phosphorus, Sulfur, and Zinc by Wavelength-Dispersive X-Ray Fluorescence Spectroscopy[3]

D 5185 Test Method for Determination of Additive Elements, Wear Metals, and Contaminants in Used Lubricating Oils and Determination of Selected Elements in Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)[3]

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance[4]

3. Summary of Test Method

3.1 A sample portion is weighed and diluted by mass with mixed xylenes or other solvent. An internal standard, which is required, is either weighed separately into the test solution or is previously combined with the dilution solvent. Calibration standards are prepared similarly. The solutions are introduced to the ICP instrument by free aspiration or an optional peristaltic pump. By comparing emission intensities of elements in the test specimen with emission intensities measured with the calibration standards and by applying the appropriate internal standard correction, the concentrations of elements in the sample are calculable.

4. Significance and Use

4.1 This test method usually requires several minutes per sample. This test method covers eight elements and thus provides more elemental composition data than Test Method D 4628 or Test Methods D 4927. In addition, this test method provides more accurate results than Test Method D 5185, which is intended for used lubricating oils and base oils.

4.2 Additive packages are blends of individual additives, which can act as detergents, antioxidants, antiwear agents, and

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

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[4] *Annual Book of ASTM Standards*, Vol 05.03.

***A Summary of Changes section appears at the end of this standard.**

so forth. Many additives contain one or more elements covered by this test method. Additive package specifications are based, in part, on elemental composition. Lubricating oils are typically blends of additive packages, and their specifications are also determined, in part, by elemental composition. This test method can be used to determine if additive packages and unused lubricating oils meet specifications with respect to elemental composition.

5. Interferences

5.1 *Spectral*—There are no known spectral interferences between elements covered by this test method when using the spectral lines listed in Table 1. However, if spectral interferences exist because of other interfering elements or selection of other spectral lines, correct for the interference using the technique described in Test Method D 5185.

5.2 *Viscosity Index Improver Effect*—Viscosity index improvers, which can be present in multi-grade lubricating oils, can bias measurements. However, the biases can be reduced to negligible proportion by using the specified solvent-to-sample dilution and an internal standard.

6. Apparatus

6.1 *Inductively-Coupled Plasma Atomic Emission Spectrometer*—Either a sequential or simultaneous spectrometer is suitable, if equipped with a quartz ICP torch and r-f generator to form and sustain the plasma.

6.2 *Analytical Balance*, capable of weighing to 0.001 g or 0.0001 g, capacity of 150 g.

6.3 *Peristaltic Pump, (Recommended)*—A peristaltic pump is strongly recommended to provide a constant flow of solution. The pumping speed must be in the range 0.5 to 3 mL/min. The pump tubing must be able to withstand at least 6 h exposure to the dilution solvent. Fluoroelastomer copolymer[5] tubing is recommended.

6.4 *Solvent Dispenser, (Optional)*—A solvent dispenser calibrated to deliver the required weight of diluent can be advantageous. Ensure that solvent drip does not affect accuracy.

6.5 *Specimen Solution Containers*, of appropriate size, glass or polyolefin vials or bottles, with screw caps.

6.6 *Vortexer, (Optional)*—Vortex the sample plus diluent mixture until the sample is completely dissolved.

6.7 *Ultrasonic Homogenizer, Optional*—A bath-type or probe-type ultrasonic homogenizer can be used to homogenizer the test specimen.

7. Reagents and Materials

7.1 *Purity of Reagents*—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.[6]

7.2 *Base Oil*, U.S.P. white oil, or a lubricating base oil that is free of analytes, having a viscosity at room temperature as close as possible to that of the samples to be analyzed. (**Warning**—Lubricating base oils can contain sulfur. For preparation of sulfur standards and blending of additive packages, white oil is recommended.)

7.3 *Internal Standard, (Required)*—An oil-soluble internal standard element is required. The following internal standards were successfully used in the interlaboratory study on precision: Ag, Be, Cd, Co (most common), La, Mn, Pb, Y.

7.4 *Organometallic Standards*—Multi-element standards, containing known concentrations (approximately 0.1 mass %) of each element, can be prepared from the individual metal concentrates. Refer to Practice D 4307 for a procedure for preparation of multicomponent liquid blends. When preparing multi-element standards, be certain that proper mixing is achieved. Commercially available multi-element blends (with known concentrations of each element at approximately 0.1 mass %) are also satisfactory.

7.4.1 More than one multi-element standard can be necessary to cover all elements, and the user of this test method can select the combination of elements and their concentrations in the multi-element standards. It can be advantageous to select concentrations that are typical of unused oils. However, it is imperative that concentrations are selected such that the emission intensities measured with the working standards can be measured precisely (that is, the emission intensities are significantly greater than background) and that these standards represent the linear region of the calibration curve. Frequently, the instrument manufacturer publishes guidelines for determining linear range.

[5] Fluoroelastomer copolymer is manufactured as Viton, a trademark owned by E. I. duPont de Nemours.

[6] *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 *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.

TABLE 1 Elements Determined and Suggested Wavelengths[A]

Element	Wavelength, nm
Barium	233.53, 455.40, 493.41
Boron[B]	182.59, 249.68
Calcium	315.88, 317.93, 364.4, 422.67
Copper	324.75
Magnesium	279.08, 279.55, 285.21
Phosphorus[B]	177.51, 178.29, 213.62, 214.91, 253.40
Sulfur[B]	180.73, 182.04, 182.62
Zinc	202.55, 206.20, 213.86, 334.58, 481.05

[A] These wavelengths are only suggested and do not represent all possible choices.
[B] Wavelengths for boron, phosphorus, and sulfur below 190 nm require that a vacuum or inert gas purged optical path be used.

7.4.2 Some commercially available organometallic standards are prepared from metal sulfonates and therefore contain sulfur. For sulfur determinations, a separate sulfur standard can be required. A sulfur standard can be prepared by blending NIST SRM 1622 with white oil.

7.4.3 Metal sulfonates can be used as a sulfur standard if the sulfur content is known or determined by an appropriate test method such as Test Method D 1552.

7.4.4 Petroleum additives can also be used as organometallic standards if their use does not adversely affect precision nor introduce significant bias.

7.5 *Dilution Solvent*—Mixed xylenes, o-xylene, and kerosine were successfully used in the interlaboratory study on precision.

8. Internal Standardization (Required)

8.1 The internal standard procedure requires that every test solution (sample and standard) have the same concentration (or a known concentration) of an internal standard element that is not present in the original sample. The internal standard is usually combined with the dilution solvent. Internal standard compensation is typically handled in one of two different ways, which can be summarized as follows.

8.1.1 Calibration curves are based on the measured intensity of each analyte divided (that is, scaled) by the measured intensity of the internal standard per unit internal standard element concentration. Concentrations for each analyte in the test specimen solution are read directly from these calibration curves.

8.1.2 For each analyte and the internal standard element, calibration curves are based on measured (unscaled) intensities. Uncorrected concentrations for each analyte in the test specimen solution are read from these calibration curves. Corrected analyte concentrations are calculated by multiplying the uncorrected concentrations by a factor equal to the actual internal standard concentration divided by the uncorrected internal standard concentration determined by analysis.

8.2 Dissolve the organometallic compound representing the internal standard in dilution solvent and transfer to a dispensing vessel. The stability of this solution must be monitored and prepared fresh (typically weekly) when the concentration of the internal standard element changes significantly. The concentration of internal standard element shall be at least 100 times its detection limit. A concentration in the range of 10 to 20 mg/kg is typical.

NOTE 1—This test method specifies that the internal standard is combined with the dilution solvent because this technique is common and efficient when preparing many samples. However, the internal standard can be added separately from the dilution solvent as long as the internal standard concentration is constant or accurately known.

9. Sampling

9.1 The objective of sampling is to obtain a test specimen that is representative of the entire quantity. Thus, take lab samples in accordance with the instructions in Practice D 4057. The specific sampling technique can affect the accuracy of this test method.

10. Preparation of Apparatus

10.1 *Instrument*—Design differences between instruments, ICP excitation sources, and different selected analytical wavelengths for individual spectrometers make it impractical to detail the operating conditions. Consult the manufacturer's instructions for operating the instrument with organic solvents. Set up the instrument for use with the particular dilution solvent chosen.

10.2 *Peristaltic Pump*— If a peristaltic pump is used, inspect the pump tubing and replace it, if necessary, before starting each day. Verify the solution uptake rate and adjust it to the desired rate.

10.3 *ICP Excitation Source*—Initiate the plasma source at least 30 min before performing an analysis. During this warm up period, nebulize dilution solvent. Inspect the torch for carbon build-up during the warm up period. If carbon build-up occurs, replace the torch immediately and consult the manufacturer's operating guide to take proper steps to remedy the situation.

NOTE 2—Carbon that accumulates on the tip of the torch injector tube can be removed by using nebulizer gas that consists of approximately 1 % oxygen in argon.

10.3.1 Generally, carbon build-up can be minimized by increasing the intermediate argon flow rate or lowering the torch, or both, relative to the load coil.

NOTE 3—Some manufacturers recommend even longer warm up periods to minimize changes in the slopes of the calibration curves.

10.4 *Wavelength Profiling*—Perform any wavelength profiling that is specified in the normal operation of the instrument.

10.5 *Operating Parameters*—Assign the appropriate operating parameters to the instrument task file so that the desired elements can be determined. Parameters to be included are element, wavelength, background correction points (optional), interelement correction factors (refer to 5.1), integration time, and internal standard compensation (required). Multiple integrations (typically three) are required for each measurement. A typical integration time is 10 s.

11. Preparation of Test Specimens

11.1 *Diluent*—Diluent refers to the dilution solvent containing the internal standard (refer to 8.2).

11.2 Test specimen solutions are prepared in the same way that calibration standards are prepared (refer to 12.2). The mass % oil in diluent must be the same for calibration standards and test specimen solutions.

11.2.1 *Lubricating Oil Specimens*—Weigh appropriate amount of the test specimen to the nearest 0.001 g. The weight of the test specimen taken will vary depending upon the metal concentration of the specimen. Dilute by mass with the diluent. Mix well.

11.2.2 *Additive Packages*—The concentrations of additive elements in additive packages are typically ten times the concentrations in lubricating oils. Therefore, additive packages are first blended with base oil before adding diluent.

11.2.2.1 Weigh appropriate amount of the additive package to the nearest 0.001 g. The weight of the test specimen taken will vary depending upon the metal concentration in the

TABLE 2 Repeatability

NOTE 1—X = mean concentration, mass %.

Element	Range, mass %	Sample	Repeatability, mass %
Ba	0.13	Oil	0.011
Ba	3.4	Additive	0.20
B	0.01–0.02	Oil	0.0017
B	0.11–0.13	Additive	0.0093
Ca	0.012–0.18	Oil	$0.0145\ (X + 0.152)^{0.67}$
Ca	0.8–4.1	Additive	$0.0363\ X$
Cu	0.01–0.02	Oil	0.0008
Cu	0.11	Additive	0.0054
Mg	0.05–0.14	Oil	$0.0159\ X^{0.7}$
Mg	0.35–0.82	Additive	$0.0473\ X$
P	0.05–0.12	Oil	$0.0264\ X$
P	0.7–1.3	Additive	$0.0313\ (X + 0.294)$
S	0.3–0.8	Oil	0.016
S	3.0–3.2	Additive	0.14
Zn	0.05–0.13	Oil	$0.0212\ (X + 0.0041)$
Zn	0.7–1.4	Additive	0.035

TABLE 3 Reproducibility

NOTE 1—X = mean concentration, mass %.

Element	Range, mass %	Sample	Reproducibility, mass %
Ba	0.13	Oil	0.019
Ba	3.4	Additive	0.66
B	0.01–0.02	Oil	0.0035
B	0.11–0.13	Additive	0.016
Ca	0.012–0.18	Oil	$0.0208\ (X + 0.152)^{0.67}$
Ca	0.8–4.1	Additive	$0.114\ X$
Cu	0.01–0.02	Oil	0.0017
Cu	0.11	Additive	0.016
Mg	0.05–0.14	Oil	$0.0624\ X^{0.7}$
Mg	0.35–0.82	Additive	$0.198\ X$
P	0.05–0.12	Oil	$0.101\ X$
P	0.7–1.3	Additive	$0.115\ (X + 0.294)$
S	0.3–0.8	Oil	0.061
S	3.0–3.2	Additive	0.372
Zn	0.05–0.13	Oil	$0.0694\ (X + 0.0041)$
Zn	0.7–1.4	Additive	0.115

TABLE 4 Calculated Precision, mass %, at Selected Concentrations, mass %

Element		Concentration				
		0.01	0.05	0.1	0.5	1.0
Ba	repeatability			0.011		
	reproducibility			0.019		
B	repeatability	0.0017		0.0093		
	reproducibility	0.0035		0.016		
Ca	repeatability	0.0043	0.0050	0.0058		0.036
	reproducibility	0.0061	0.0071	0.0083		0.114
Cu	repeatability	0.0008		0.0054		
	reproducibility	0.0017		0.016		
Mg	repeatability		0.0020	0.0032	0.024	
	reproducibility		0.0076	0.0124	0.099	
P	repeatability		0.0013	0.0026		0 .041
	reproducibility		0.0051	0.0101		0.149
S	repeatability				0.016	
	reproducibility				0.061	
Zn	repeatability		0.0011	0.0022		0.035
	reproducibility		0.0037	0.0072		0.115

specimen. Add approximately ten times this amount of base oil, weighed to the nearest 0.001 g. Dilute this mixture by mass with diluent. Mix well.

11.3 Record all weights and calculate dilution factors by dividing the sum of the weights of the diluent, sample, and base oil (if any) by the weight of the sample.

12. Preparation of Calibration Standards and Check Standards

12.1 *Diluent*—Diluent refers to the dilution solvent containing the internal standard (refer to 8.2).

12.2 The user of this test method has the option of selecting the dilution factor, that is, the relative amounts of sample and diluent. However, the mass % sample in diluent (for calibration standards and test specimens) must be constant throughout this test method, and the mass % sample in diluent must be in the range of 1 to 5 mass %.

12.2.1 All references to *dilute* and *diluting* in this test method refer to the user-selected dilution.

12.3 *Blank*—Prepare a blank by diluting the base oil or white oil with the diluent.

12.4 *Working Standards*—Weigh to the nearest 0.001 g, approximately 1 to 3 g of each multi-element standard (refer to 7.4) into separate bottles. Dilute by mass with the diluent.

12.5 *Check Standard*—Prepare instrument check standards in the same manner as the working standards such that the concentrations of elements in the check standards are similar to the concentrations of elements in the test specimen solutions. It is advisable to prepare the check standard from alternative sources of certified organometallic standards.

13. Calibration

13.1 The linear range of all calibration curves must be determined for the instrument being used. This is accomplished by running intermediate standards between the blank and the working standards and by running standards containing higher concentrations than the working standards. Analyses of test specimen solutions must be performed within the linear range of the calibration curve.

13.2 At the beginning of the analysis of each set of test specimen solutions, perform a two-point calibration using the blank and working standard.

13.3 Use the check standard to determine if each element is in calibration. When the results obtained with the check standard are within 5 % (relative) of the expected concentrations for all elements, proceed with the analysis. Otherwise, make any adjustments to the instrument that are necessary and repeat the calibration.

13.4 Calibration curves can be constructed differently, depending on the implementation of internal standard compensation.

13.4.1 When analyte intensities are ratioed to internal standard intensities, the calibration curve is, in effect, a plot of I(Re) versus analyte concentration and:

$$I(Re) = (I(e) - I(Be))/I(is) \quad (1)$$

where:

$I(Re)$ = intensity ratio for analyte e,
$I(e)$ = intensity for analyte e,
$I(Be)$ = intensity of the blank for analyte e, and
$I(is)$ = intensity of internal standard element.

13.4.2 When internal standard compensation is handled by multiplying all results for a certain test specimen by the ratio of the actual internal standard concentration to the determined internal standard concentration, the calibration curve is, in effect, a plot of ($I(e) - I(Be)$) versus analyte concentration.

14. Analysis

14.1 Analyze the test specimen solutions in the same manner as the calibration standards (that is, same integration time, background correction points (optional), plasma conditions, and so forth). Between test specimens nebulize dilution solvent for a minimum of 60 s.

14.2 When the concentration of any analyte exceeds the linear range of the calibration, prepare another test specimen by mixing the sample with base oil before adding diluent (refer to 11.2.2.1, for example). Then, reanalyze.

14.3 Analyze the check standard after every fifth test specimen solution. If any result is not within 5 % of the expected concentration, recalibrate the instrument and reanalyze the test specimen solutions back to the previous acceptable check standard analysis.

15. Quality Assurance/Quality Control (required)

15.1 Confirm the performance of the instrument and the test procedure by analyzing a control (QC) sample.

15.1.1 When QA/QC protocols are already established in the testing facility, these may be used to confirm the reliability of the test result.

15.1.2 When there is no QA/QC protocol established in the testing facility, Appendix X1 can be used as the QA/QC protocol.

15.2 Users of this test method are advised that in contractual agreements, one or more of the contracting parties can and may make Appendix X1 a mandatory practice.

16. Calculation and Report

16.1 Calculate concentrations, based on sample, using (Eq 1). Generally, the ICP software performs this calculation automatically.

$$C = S \times \frac{(W_1 + W_2 + W_3)}{W_1} \quad (2)$$

where:

C = analyte concentration in the sample, mass %,
S = analyte concentration in the test specimen, mass % (refer to Section 14),
W_1 = sample mass, g,
W_2 = diluent mass, g, and
W_3 = base oil mass (if any), g.

16.2 For each analyte, report mass % to three significant figures.

17. Precision and Bias [7]

17.1 The precision of this test method was determined by statistical analysis of interlaboratory results. Fourteen participating laboratories analyzed twelve samples in duplicate. Most laboratories performed the analyses at three different levels of dilution, namely, 1 mass % sample in solvent, 2 mass % sample and 5 mass % sample. In this study, dilution solvents were limited to mixed xylenes, o-xylene, and kerosine. The most

[7] Interlaboratory study data are available from ASTM International Headquarters. Request RR:D02-1349.

common source of organometallic standards was metal sulfonates. Most laboratories used a peristaltic pump, and approximately half of the laboratories used background correction. The sample set comprised eight oils, five of which were multi-grade oils, and four additive packages.

17.1.1 *Repeatability*—The difference between two 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 values in Table 2 only in one case in twenty.

17.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different operators working in different laboratories on identical test materials, would in the long run, in the normal and correct operation of the test method, exceed the values in Table 3 only in one case in twenty. (Also, see Table 4.)

18. Keywords

18.1 additive-elements; barium; boron; calcium; copper; emission-spectrometry; ICP; inductively-coupled plasma atomic emission spectrometry; internal standard; lubricating oils; magnesium; phosphorus; sulfur; zinc

APPENDIXES

(Nonmandatory Information)

X1. GENERIC QUALITY CONTROL STATEMENT FOR D02 TEST METHODS

X1.1 Confirm the performance of the instrument or the test procedure by analyzing a quality control (QC) sample that is, if possible, representative of the samples typically analyzed.

X1.2 Prior to monitoring the measurement process, the user of the method needs to determine the average value and control limits of the QC sample (see Practice D 6299 and MNL7[8]).

X1.3 Record the QC results and analyze by control charts or other statistically equivalent techniques to ascertain the statistical control status of the total test process (see Practice D 6299 and MNL7[8]). Any out-of-control data should trigger investigation for root cause(s). The results of this investigation may, but not necessarily, result in instrument recalibration.

X1.4 In the absence of explicit requirements given in the test method, the frequency of QC 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 is routinely analyzed. However, when it is demonstrated that the testing is under statistical control, the QC testing frequency may be reduced. The QC sample precision should be periodically checked against the ASTM method precision to ensure data quality.

X1.5 It is recommended that, if possible, the type of QC sample that is regularly tested be representative of the sample 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 the anticipated storage conditions.

X1.6 Refer to relevant documents (see Practice D 6299 and MNL7) for further guidance on QC and control charting techniques.

[8] *ASTM MNL7*, "Manual on Presentation of Data Control Chart Analysis, 6th Ed, Section 3: Control Chart for Individuals", available from ASTM International Headquarters.

X2. AIDS TO THE ANALYST

X2.1 Check the temperature control of the ICP and ensure stable environmental conditions. This can include temperature control of the spray chamber.

X2.2 Employ adequate mixing and sampling procedures. Ultrasonic homogenizers and vortex mixers are recommended.

X2.3 Use the analytical wavelengths and background correction option specified in the test method. When there is a choice of analytical wavelengths, choose sensitive lines. Ensure that the selected lines are not subject to spectral interferences.

X2.4 When spectral interferences cannot be avoided, determine and implement accurate interference correction factors.

X2.5 When sulfur is to be determined, be apprised that many commercially-available standards contain non-certified levels of sulfur. Separate sulfur standards can be advantageous.

X2.6 When preparing multi-element standards, ensure that the various reagents are mutually soluble.

X2.7 Before use, check the accuracy of element concentrations of commercially-obtained standards. Either compare with alternative sources or analyze by independent methods.

X2.8 Ensure that all glassware, and so forth, that contacts samples and standards does not contaminate.

X2.9 Select solvents and other reagents that do not contain significant levels of the analytes. Wavelength scanning can indicate contaminated reagents.

X2.10 By experiment, determine the frequency of standards preparation. Then, prepare fresh, as needed.

X2.11 Periodically, as needed, determine the linearity of the calibration curves. Perform quantitative analyses with linear curves only.

X2.12 Inspect the torch for cracks. Discard defective torches.

X2.13 Use clean torches that do not have carbon accumulation.

X2.14 After initially igniting the plasma, allow the instrument to warm up a minimum of 30 min.

X2.15 Inspect the peristaltic pump tubing daily, and replace deteriorating tubing. Daily replacement is recommended.

X2.16 Prepare and analyze reagent blanks. When blank values are significant, correct for the blank or select alternative reagents that give insignificant blank values.

X2.17 To minimize memory effects, allow sufficient solvent rinse time (minimally, 60 s) between determinations.

X2.18 Report results using the number of significant figures specified in the test method.

X2.19 Dilute the standard oils and sample oils by the same factor. This factor shall be in the range specified by the test method.

X2.20 Implement internal standardization as specified by the test method.

X2.21 When carbon build-up in the torch is problematic, adjust experimental conditions to eliminate the problem. Such adjustments can include (*1*) reducing the sample uptake rate, (*2*) increasing the intermediate argon gas flow rate, (*3*) use a jacketed, chilled spray chamber, (*4*) lowering the torch, relative to the RF load coil.

SUMMARY OF CHANGES

Subcommittee D02.03 has identified the location of selected changes to this standard since the last issue (D 4951 - 00) that may impact the use of this standard.

(1) Updated the requirements for the specimen solution containers in 6.5 to account for container sizes that fall out of the range previously specified, due to instrument or auto-sampler requirements.
(2) Updated the requirements for the base oil specified in 7.2.
(3) Updated the requirements in 11.2.1 and 11.2.2.1 to provide flexibility in the amount of oil or additive package that needs to be weighed for the analysis.
(4) Corrected errors in Table 4 concerning the repeatability value cited for P at 0.1 mass %, as well as the reproducibility value cited for P at 0.05 mass %.