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An American National Standard

Standard Test Methods for Elemental Analysis of Lubricant and Additive Components—Barium, Calcium, Phosphorus, Sulfur, and Zinc by Wavelength-Dispersive X-Ray Fluorescence Spectroscopy[1]

This standard is issued under the fixed designation D 4927; 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 These test methods cover the determination of barium, calcium, phosphorus, sulfur, and zinc in unused lubricating oils at element concentration ranges shown in Table 1. The range can be extended to higher concentrations by dilution of sample specimens. Additives can also be determined after dilution. Two different methods are presented in these test methods.

1.2 *Test Method A (Internal Standard Procedure)*—Internal standards are used to compensate for interelement effects of X-ray excitation and fluorescence (see Sections 1-11, and 17).

1.3 *Test Method B (Mathematical Correction Procedure)*—The measured X-ray fluorescence intensity for a given element is mathematically corrected for potential interference from other elements present in the sample (see Sections 1-6, and 12-17).

1.4 The preferred concentration units are mass percent barium, calcium, phosphorus, sulfur, or zinc.

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 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance[2]

3. Summary of the Test Methods

3.1 A sample specimen is placed in the X-ray beam and the intensity of the appropriate fluorescence lines of barium, calcium, phosphorus, sulfur, and zinc are measured. Instrument response factors related to the concentration of standards enable the determination of the concentration of elements in the tested sample specimens. Enhancement or depression of the X-ray fluorescence of a given element by an interfering element in the sample may occur. Two test methods (*A* and *B*) are described for compensating any interference effect.

TABLE 1 Range of Applicability

Element	Range, Mass %
Barium	0.04 - 8.5
Calcium	0.01 - 1.0
Phosphorus	0.01 - 0.5
Sulfur	0.1 - 4.0
Zinc	0.01 - 0.6

3.2 *Test Method A (Internal Standard Procedure)*—Internal standards are used with the standards and sample specimens to compensate for the potential interelement effects.

3.2.1 *Barium, Calcium, Phosphorus, and Zinc*—A sample specimen that has been blended with a single internal standard solution (containing tin or titanium for barium and calcium, zirconium for phosphorus, and nickel for zinc) is poured into an X-ray cell. Total net counts (peak intensity—background) for each element and its respective internal standard are collected at their appropriate wavelengths. The ratios between elemental and internal standard counts are calculated and converted into barium, calcium, phosphorus, or zinc concentrations, or a combination thereof, from calibration curves.

3.2.2 *Sulfur*—A sample specimen is mixed with a lead internal standard solution and analyzed as described in 3.2.1.

3.3 *Test Method B (Mathematical Correction Procedure)*—The measured intensity for a given element is mathematically corrected for the interference from other elements in the sample specimen. This requires that intensities from all elements in the specimen be obtained.

3.3.1 The sample specimen is placed in the X-ray beam and the intensities of the fluorescence lines of barium, calcium, phosphorus, sulfur, and zinc are measured. A similar measurement is made away from the fluorescence lines in order to obtain a background correction. Concentrations of the elements

[1] These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and are the direct responsibility of Subcommittee D02.03on Elemental Analysis.

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

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

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