



Designation: D7751 – 16

Standard Test Method for Determination of Additive Elements in Lubricating Oils by EDXRF Analysis¹

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

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. 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

D6792 Practice for Quality System in Petroleum Products 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:

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

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>.

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