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An American National Standard

Standard Methods of CHEMICAL ANALYSIS FOR METALS IN NEW AND USED LUBRICATING OILS¹

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These methods have been adopted for use by government agencies to replace Method 5601 of Federal Test Method Standard No. 791b.

1. Scope

1.1 These methods of chemical analysis cover the determination of barium, tin, silica, zinc, aluminum, calcium, magnesium, and sodium and potassium in new and used lubricating oils. Other metallic elements, sulfur, phosphorus, and chlorine in amounts commonly found in lubricating oils do not interfere in this method.

NOTE 1—These methods do not cover the determination of lead, copper, and iron.

2. Summary of Methods

2.1 The analytical procedures follow the well-known scheme of separating the metals into groups for more convenient determination, as shown in Fig. 1. This scheme provides a rapid and accurate method for the determination of all, several, or any one of the metals as may be seen necessary from an initial qualitative inspection of the oil sample.

2.2 To avoid its interference throughout the procedure, barium is separated along with the metals of the hydrogen sulfide group. The 8-hydroxyquinoline serves as an excellent means for removing iron, aluminum, and zinc from phosphate interference. Phosphate and sulfate ions are removed after the calcium determination to provide for a more accurate determination of sodium and potassium.

3. Preparation of Sample

3.1 Heat the sample of oil to $60 \pm 10^\circ\text{C}$ and agitate it in the original container until all sediment is homogeneously suspended in the oil. If the original container is of opaque ma-

terial, or if it is more than three quarters full, transfer the entire sample to a clear glass bottle having a capacity at least one third greater than the volume of the sample. Transfer all traces of sediment from the original container to the bottle by violent agitation of portions of the sample in the original container. After complete suspension of all sediment, strain the sample or a convenient aliquot through a 100-mesh screen for the removal of large contaminating particles.

3.2 Heat and thoroughly mix the strained oil or aliquot before taking *each* portion of the sample for test. Homogeneity will be indicated by the precision of the analyses.

4. Purity of Reagents and Materials

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.² 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.

¹ These methods are under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants.

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² "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N.Y., and the "United States Pharmacopeia."