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Standard Test Method for ZINC IN LUBRICATING OILS AND ADDITIVES (POLAROGRAPHIC METHOD)¹

This standard is issued under the fixed designation D 1549; 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.

The committee responsible for this standard has voted its withdrawal. In the absence of substantial reasons that it should be continued, the Society will approve withdrawal from publication in February 1986.

¹NOTE—Section 2 was added editorially and sections renumbered in July 1984.

1. Scope

1.1 This test method covers the determination (by the polarographic method) of zinc in new lubricating oils and lubricating oil additives containing no more than 2 % zinc. Since cadmium is used as the pilot ion, this metal interferes and must be absent from the sample.

NOTE 1—Methods D 811 also covers the determination of zinc.

NOTE 2—Although the relative precision of this test method remains about the same up to 8 % zinc content, this was considered unsuitable for commercial purposes above the 2 % zinc level.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Applicable Document

2.1 ASTM Standard:

D 811 Methods of Chemical Analysis for Metals in New and Used Lubricating Oils²

3. Summary of Method

3.1 The sample is ignited and burned until only carbon and ash remain. The residue is treated with sulfuric acid, heated to drive off most of the acid, and ignited in a furnace at 550 to 600°C. The ash is treated with hydrochloric acid, and the acid extract is transferred to a volumetric

flask. Standard volumes of cadmium—electrolyte solution and gelatin solution are added; and the solution is diluted to volume with water. The zinc content of the solution is determined polarographically.

4. Apparatus

4.1 *Electric Muffle Furnace*, capable of operating between 150 and 600°C and of maintaining a temperature of $575 \pm 25^\circ\text{C}$.

4.2 *Electrolysis Assembly*—A suitable electrolysis assembly is shown in Fig. 1. It consists of the following component parts:

4.2.1 *Polarographic Cell*—The cell shown in Fig. 1 has been found satisfactory (Note 3). An index mark should be made to indicate the volume of solution to be introduced for analysis (about 10 mL).

NOTE 3—Any other cell that will perform similarly may be used.

4.2.2 *Cathode*—A piece of marine barometer tubing approximately 10 to 20 cm in length, with a bore of about 0.06 mm, is connected by means of a small piece of plastic tubing³ to a stand tube 75 cm in length. The stand tube is tapered at the bottom to approximately the outer diameter of

¹ This method is under the jurisdiction of ASTM Committee D-2 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 Vinyl tubing has been found satisfactory for this purpose.