



Standard Test Method for Measurement of Hindered Phenolic Antioxidant Content in HL Turbine Oils by Linear Sweep Voltammetry¹

This standard is issued under the fixed designation D 6810; 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 voltammetric determination of hindered phenol antioxidants in new or used Type HL turbine oils in concentrations from 0.0075 weight % up to concentrations found in new oils by measuring the amount of current flow at a specified voltage in the produced voltammogram.

1.2 *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 1193 Specification for Reagent Water²
- D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products³
- D 4378 Practice for In-Service Monitoring of Mineral Turbine Oils for Steam and Gas Turbines³
- D 6224 Practice for In-Service Monitoring of Lubricating Oil for Auxiliary Power Plant Equipment⁴
- D 6447 Test Method for Hydroperoxide Number of Aviation Turbine Fuels by Voltammetric Analysis⁵

2.2 ISO Standard:

- ISO 6743 Part 4—Lubricants, Industrial Oils, and Related Products⁶

3. Summary of Test Method

3.1 A measured quantity of sample is dispensed into a vial containing a measured quantity of alcohol-based electrolyte solution and containing a layer of sand. When the vial is

shaken, the hindered phenol antioxidants and other solution soluble oil components present in the sample are extracted into the solution and the remaining droplets suspended in the solution are agglomerated by the sand. The sand/droplet suspension is allowed to settle out and the hindered phenol antioxidants dissolved in the solution are quantified by voltammetric analysis. The results are calculated and reported as weight percent of antioxidant or as millimoles (mmol) of antioxidant per litre of sample for prepared and fresh oils and as a percent remaining antioxidant for used oils.

3.2 Voltammetric analysis is a technique that applies electro-analytic methods when a sample to be analyzed is mixed with an electrolyte and a solvent and placed within an electrolytic cell. Data is obtained by measuring the current passing through the cell as a function of the potential applied, and test results are based upon current, voltage and time relationships at the cell electrodes. The cell consists of a fluid container into which is mounted a small, easily polarized working electrode, and a large nonpolarizable reference electrode. The reference electrode should be massive relative to the working electrode so that its behavior remains essentially constant with the passage of small current; that is, it remains unpolarized during the analysis period. Additional electrodes, auxiliary electrodes, can be added to the electrode system to eliminate the effects of resistive drop for high resistance solutions. In performing a voltammetric analysis, the potential across the electrodes is varied linearly with time, and the resulting current is recorded as a function of the potential. As the increasing voltage is applied to the prepared sample within the cell, the various additive species under investigation within the oil are caused to electrochemically oxidize. The data recorded during this oxidation reaction can then be used to determine the remaining useful life of the oil type. A typical current-potential curve produced during the practice of the voltammetric test can be seen by reference to Fig. 1. Initially, the applied potential produces an electrochemical reaction having a rate so slow that virtually no current flows through the cell. As the voltage is increased, as shown in Fig. 1, the electro-active species (for example, substituted phenols) begin to oxidize at the working electrode surface, producing an anodic rise in the current. As the potential is further increased,

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.09 on Oxidation.

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² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 05.02.

⁴ Annual Book of ASTM Standards, Vol 05.03.

⁵ Annual Book of ASTM Standards, Vol 05.04.

⁶ Available from International Organization for Standardization (ISO), 1 rue de Varembe, Case postale 56, CH-1211, Geneva 20, Switzerland.