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**Committee D02 on Petroleum Products and Lubricants
Subcommittee D02.09 on Oxidation**

Research Report D02-1548

**Interlaboratory Study to Establish Precision Statements for ASTM
D6810, Standard Test Method for Measurement of Hindered Phenolic
Antioxidant Content in HL Turbine Oils by Linear Sweep Voltammetry**

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COMMITTEE D02 ON PETROLEUM PRODUCTS AND LUBRICANTS

Research Report: D02: _____

Measurement of Remaining Hindered Phenolic Antioxidant Content in Turbine Oils by Linear Sweep Voltammetry

1. Introduction:

There is a need for a rapid test that uses small sample volumes for measuring remaining hindered phenolic antioxidant content in Turbine oils from oxidation testing or field use (condition monitoring programs). A new method was proposed for this purpose using linear sweep voltammetry, referred to as a voltammogram. This study was performed using 2 different types of Voltammograms (manufactured by Chevron and Fluitec International) to measure the Remaining Hindered Phenolic Antioxidant Content in Turbine Oils by Linear Sweep Voltammetry.

The objectives of the round robin were to:

- 1) To establish voltammetric test method for measurement of hindered phenolic antioxidant content in Turbine oils.
- 2) Determine whether the voltammetric method can be used to measure hindered phenol antioxidant depletion to monitor oxidation condition in turbine lubricants.
- 3) Establish the test precision. Determine the repeatability standard deviation s_r , and test reproducibility standard deviation s_R for this voltammetric test method. From these indices, calculate the repeatability and reproducibility limits for the test method.
- 4) Establish if test precision is affected by the presence of amine antioxidants in the same oil formulations.

2. Test Method:

The test method, version 2 of the draft test method, titled the “ Standard Test Method for Measurement of Hindered Phenolic Antioxidant Content in Turbine Oil by Linear Sweep Voltammetry”, was distributed to the round robin participants prior to this study. A copy of this method is attached to this research report.

3. Participating Laboratories, Equipment and Test materials

Eight laboratories agreed to participate in this round robin program, from which seven of the eight laboratories completed their analyses submitted their test results. We have coded the participating labs with letter codes (A-G), such that they are not identified in this research report. A table of the participating laboratories is attached to this report.

The test equipment and supplies consisted of the following.

Test equipment:

- Voltammogram (type Fluitec RULER, model 500S and type Chevron)
- Voltammograph Operation Manual
- Data acquisition software (RULER software R-DAS version 2.61), strip chart recorder or X,Y plotter

Test supplies consisted of the following:

7-ml vials containing prepared electrolytic solutions supplied by Fluitec International. These will be referred to as Green or Yellow Voltammetric Antioxidant test solvents.

- Green test solutions – neutral test solution as described in STM draft version 02
- Yellow test solution – basic test solution as described in STM draft version 02

The test materials (oil samples) consisted of 1liter samples of 3 different participants, which supplied the test oils. The suppliers of these test oils were Pennzoil Quaker State, Fluitec International, and Chevron Products.

25-ml of each test oil were supplied to the eight participants of the round robin. The sample identifications we used for this study as well the general characteristics of the test oils are shown below:

<u>Sample ID</u>	<u>Description</u>
LUB01001	Fresh turbine oil A – containing phenols and amines
LUB01002	Used turbine oil A – in-service oil for approx. 10,000 hrs.
LUB01003	Used turbine oil A – in-service oil for approx. 20,000 hrs.
LUB01004	Used turbine oil A – in-service oil for approx. 30,000 hrs.
LUB01005	Fresh turbine oil B – with low level of hindered phenol & no amine antioxidant
LUB01006	Fresh turbine oil C – with medium level of hindered phenol (same hindered phenol as in Fresh oil B) & no amine antioxidant
LUB01007	Fresh turbine oil D – with High Level of hindered phenol (same hindered phenol as in Fresh Oil B &C) and no amine antioxidant
LUB01008	Fresh turbine oil E - with medium level of hindered phenol (same hindered phenol as in Fresh oil B, C, & D) and amine antioxidant
LUB01009	Fresh turbine oil F – industrial turbine oil with amines and hindered phenols
LUB01010	Used turbine oil F – industrial turbine oil from in-service application

Oils in the study were turbine oils - Industrial turbine oils containing an R&O additive Package - from the same ISO VG 32 grade

The oil samples were shipped from Fluitec International, Dayton OH by September 20, 2000.

4. Interlaboratory Test Program Instructions: