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**1 July 2009**

**Committee D02 on Petroleum Products and Lubricants  
Subcommittee D02.96 on In-Service Lubricant Testing and Condition  
Monitoring Services**

**Research Report D02-1667**

**Interlaboratory Study to Establish Precision Statements for ASTM  
D7412-09 - New Test Method for Condition Monitoring of Phosphate  
Antiwear Additives in Used Petroleum and Hydrocarbon Based  
Lubricants by Trend Analysis using Fourier Transform Infrared (FT-IR)  
Spectrometry**

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**1. Introduction:**

A pilot study was conducted to establish an interim Repeatability Statement for D7412-09 - New Test Method for Condition Monitoring of Phosphate Antiwear Additives in Used Petroleum and Hydrocarbon Based Lubricants by Trend Analysis using Fourier Transform Infrared (FT-IR) Spectrometry

**2. Test Method:**

The test method used for this pilot study is ASTM D7412-09. To obtain a copy of D7412, go to ASTM's website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service by phone at 610-832-9585 (8:30 a.m. - 4:30 p.m. Eastern U.S. Standard Time, Monday through Friday) or by email at [service@astm.org](mailto:service@astm.org).

**3. Participating Laboratories:**

The following laboratories participated in this interlaboratory study

1. Thermal-Lube Inc.  
255 Avenue Labrosse  
Pointe-Claire, Québec  
Canada  
H9R 1A3

2. McGill IR Group  
McGill University  
21111 Lakeshore Road  
Ste-Anne-de-Bellevue, Québec  
Canada  
H9X 3V9

**4. Description of Samples:**

The samples consisted of six (6) samples of in-service diesel engine oils for direct trend analysis and a series of six (6) diesel engine oils taken from an engine at various time intervals plus a sample of the corresponding fresh oil (reference oil) for differential trend analysis.

Samples supplied by:

1. Thermal-Lube Inc.  
255 Avenue Labrosse  
Pointe-Claire, Québec  
Canada  
H9R 1A3

**5. Interlaboratory Study Instructions**

Laboratory participants were emailed the test program instructions. For a copy of the instructions, please see Annex A.

**6. Description of Equipment/Apparatus<sup>1</sup>:**

For information on the equipment/apparatus used by each laboratory, please see Annex B.

**7. Data Report Forms:**

Each laboratory was provided with a data report form for the collection of data. A copy of the data is provided in Annex C.

Please note: The laboratories have been randomly coded and cannot be identified herein.

**8. Statistical Data Summary:**

This was a single laboratory data collection in order to develop an interim Repeatability Statement for ASTM D7412. The repeatability was calculated as  $1.960v2 s$ , where  $s$  is the standard deviation of the paired replicates of six (6) samples.

**9. Precision and Bias Statement:**

Interim Repeatability—The difference between repetitive results, from non-diluted samples, obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

Procedure A (*Direct Trend Analysis*)—Repeatability ( $r$ ) = 0.53 absorbance units/0.100 mm.

Procedure B (*Differential Trend Analysis*) —Repeatability ( $r$ ) = 0.57 absorbance units/cm.

Bias— The procedures in this test method have no bias because the phosphate antiwear additive values can be defined only in the terms of the test method and no accepted reference method or value is available.

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<sup>1</sup> The equipment listed was used to develop a precision statement for D7412-09. This listing is not an endorsement or certification by ASTM International.

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