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Committee D02 on Petroleum Products
Subcommittee D.02.03on Elemental Analysis

(Interim) Research Report D02-1732

(This Research is Preliminary and is issued for WK 18448 Task Group review and comment purposes)

Interlaboratory Study (ILS) to Establish Precision Statements for ASTM D7751, Standard Test Method for Determination of Additive Elements in Lubrication Oils by EDXRF Analysis

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1. Summary :

D.02.03 (Elemental Analysis) - WK 18448 Task Group activity led to an interlaboratory study (ILS-0308/501) that was conducted in summer 2009 in compliance with ASTM E691 guidelines. It involved a new EDXRF- method draft. The ILS included the analysis of a variety of various lubrication oils.

Overall findings of the statistical analysis of the data according to D6300/D2PP and D6259 demonstrated support for a preliminary precision statement.

2. Introduction:

This Research Report contains documentation and Interlaboratory Study (ILS-0308/0501) data compiled to support the new ASTM standard test method; Standard Test Method for Determination of Additive Elements in Lubrication Oils by EDXRF Analysis, ASTM work item WK18448 with a preliminary precision statement.

The new work item was started to create a new test method which should offer an alternative to ASTM D6481 – Standard Test Method for Determination of Phosphorus, Sulfur, Calcium, and Zinc in Lubrication Oils by Energy Dispersive X-ray Fluorescence Spectroscopy. In comparison to this test method also new types of detection systems can be used, the scope of elements and concentrations is expanded based on the new possibilities when using high resolution EDXRF detection systems.

3. Collaboration and Test Method:

Using TG input gathered from various sources, WK 18448 was written to accommodate the proposed apparatus and used for ILS -0308/0501 described herein.

4. Participating Laboratories:

The following 14 laboratories participated in this interlaboratory study:

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

5. Description of Samples:

There were 16 samples of varying targeted matrices and additive concentrations used for this study. For a listing of sample types and target concentration, please see annex B. Below is a list of ILS samples:

Sample A	Industrial oil spiked with 238 ppm Conostan Ba
Sample B	Industrial oil
Sample C	Industrial oil
Sample D	Transmission oil
Sample E	Transmission oil
Sample F	Motor engine oil
Sample G	Motor engine oil
Sample H	Motor engine oil
Sample I	Motor engine oil
Sample J	Motor engine oil, mix 1/3 sample F, G, H
Sample K	Marine oil Medium Ca
Sample L	Marine oil High Ca spiked with 238 ppm Conostan Si
Sample M	Marine oil Medium Ca + spiked with 500 ppm Chlorine
Sample N	Multiblend A
Sample O	Multiblend B
Sample P	Multiblend C