This Research Report is issued under the fixed designation RR: D02-1732. You agree not to reproduce or circulate or quote, in whole or part, this document outside of ASTM International Committee/Society activities, or submit it to any other organization or standards body (whether national, international or other) except with the approval of the Chairman of the Committee having jurisdiction and the written authorization of the President of the Society. If you do not agree to these conditions, please immediately destroy all copies of this document. *Copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. All rights reserved.*

1 October 2011

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

Technical contact: Dirk Wissmann SPECTRO Analytical Instruments GmbH +49 2821 892 4147 dirk.wissmann@ametek.com

ASTM International 100 Barr Harbor Drive West Conshohocken, PA 19428-2959

This research report is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this research report may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or serviceastm.org (e-mail); or through the ASTM website (www.astm.org).

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this research report. Users of this research report are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

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:

Oxford Instruments	PANalytical	Rigaku Americas Corporation
Analytical	Bruno Vrebos	Jeff Weller
Christelle Petiot	Almelo	4543 Clearwater Lane
Halifax Road	The Netherlands	Naperville, IL 60564
High Wycombe, Bucks	+31 546 534 377	630 961 4968
HP123SE, UK	bruno.vrebos@panalytical.com	jeff.weller@rigaku.com
+44 1494 479 274		
christelle.petiot@oxinst.com		
PDVSA INTEVEP	Spectro Analytical Instruments	Chevron Products Company
Georgia Sanabria	GmbH	Thomas Bell
Urb. Santa Rosa, sector El	Dirk Wissmann	100 Chevron Way
Tambor, Los Teques	Boschstr. 10	Richmond, CA 94802
Edo. Miranda, PO Box 76343	47533 Kleve, Germany	
Caracas 1070-A, Venezuela	+49 2821 892 4147	tcbell@chevron.com
sanabriag@pdvsa.com	dirk.wissmann@ametek.com	
Verkol S.A.	CTL Ostrava	VYSOKA ŠKOLA BANSKA-
Francisco Urquiola	Mrs. Dipl. Ing. T. PECINKOVA	TECHNICKA UNIVERZITA
B° Zalain, 42	Nam. Svatopluka Cecha 4	Mr. Doc. Ing. V. TOMASEK,
31789 Bera de Bidasoa	702 09 OSTRAVA 1	17.listopadu 15
Navarra- Spain	Czech Republic	708 33 Ostrava-Poruba

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

RR: D02-1732

furquiola@verkol.es	t.pecinkova@cs.mfcr.cz	Czech Republic
		vladimir.tomasek@vsb.cz
Comma Oil and Chemicals	Lucas Oil Products	Oxford Instruments America
Ltd.	Mark Negast	Terry Suscavage
Edward Wright	302 North Sheridan Street	300 Baker Avenue
Lower Range Road	Corona, CA	Concord, MA
Gravesend	951 493 1149	978 369 9933
Kent DA12 2QX, UK		terry.suscavage@oxinst.com
+44 1474 546 268	markn@lucasoil.com	
T.Wright@commaoil.com		
Kuwait Petroleum Belgium	BP Belgium Lubes Plant Gent	Chevron Global Lubricants
NV	Bram Hofman	Texaco Petrolifera
Bruno Palmaers	Langerbruggekaai 18	Rafa Garijo
Petroleumkaai 7	9000 Gent, Belgium	Calle Villa de Madrid 34
2020 Antwerpen, Belgium	+32 9 257 32 70	Poligono Industrial Fuente del
+32 3 247 38 52	Bram.Hofman@ec1.bp.com	Jarro
Bruno.palmaers@Q8.be		46988 Paterna Valencia, Spain
		garijr@chevron.com
Inspectorate Antwerp NV		
Felix Anyakudo Noorderlaan 183 B1		
2030 Antwerpen, Belgium +32 3 546 08 81		
fan@Inspectorate.be		

<u>Please note:</u> 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

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.