Petroleum products - Determination of low concentration of sulfur in automotive fuels - Energy-dispersive X-ray fluorescence spectrometric method (ISO 13032:2012)

EESTI STANDARDIKESKUS
ESTONIAN CENTRE FOR STANDARDISATION

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN ISO 13032:2012 sisaldab Euroopa standardi EN ISO 13032:2012 ingliskeelset teksti.	This Estonian standard EVS-EN ISO 13032:2012 consists of the English text of the European standard EN ISO 13032:2012.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 01.03.2012.	Date of Availability of the European standard is 01.03.2012.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

ICS 75.160.20

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 13032

March 2012

ICS 75.160.20

English Version

Petroleum products - Determination of low concentration of sulfur in automotive fuels - Energy-dispersive X-ray fluorescence spectrometric method (ISO 13032:2012)

Produits pétroliers - Détermination de la teneur en soufre en faible concentration dans les carburants pour automobiles - Méthode spectrométrique de fluorescence de rayons X dispersive en énergie (ISO 13032:2012)

Mineralölerzeugnisse - Bestimmung niedriger Schwefelgehalte in Kraftstoffen - Energiedispersives Röntgenfluoreszenzspektrometrieverfahren (ISO 13032:2012)

This European Standard was approved by CEN on 29 February 2012.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN ISO 13032:2012: E

Foreword

This document (EN ISO 13032:2012) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin” the secretariat of which is held by NEN, in collaboration with Technical Committee ISO/TC 28 " Petroleum products and lubricants".

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2012, and conflicting national standards shall be withdrawn at the latest by September 2012.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Contents

Page

Foreword ... **iv**
Introduction ... **v**
1 Scope ... **1**
2 Normative references ... **1**
3 Principle ... **1**
4 Reagents and materials ... **1**
4.1 Diluent oil ... **1**
4.2 Sulfur compounds ... **2**
4.3 Reference materials (CRMs) ... **2**
4.4 Quality control samples ... **2**
5 Apparatus ... **2**
5.1 Energy-dispersive X-ray fluorescence instrument ... **2**
6 Sampling and sample handling ... **3**
7 Apparatus preparation ... **4**
7.1 Analyser ... **4**
7.2 Sample cups ... **4**
8 Calibration ... **4**
8.1 General ... **4**
8.2 Preparation of primary standards ... **4**
8.3 Calibration standards ... **5**
8.4 Calibration procedure ... **6**
9 Procedure ... **7**
10 Calculation ... **7**
11 Expression of results ... **7**
12 Precision ... **8**
12.1 General ... **8**
12.2 Repeatability, r ... **8**
12.3 Reproducibility, R ... **8**
13 Test report ... **8**
Annex A (informative) **Matrix effects** ... **9**
Annex B (normative) **Guidelines for measuring low sulfur concentrations** ... **11**
Bibliography ... **12**

Introduction

This International Standard is directed specifically at the lower end of the concentration range covered in ISO 20847[2]. By selecting the instrument type, a better signal-to-background ratio for sulfur K-$L_{2,3}$ emission is assured. A knowledge of the general composition of the sample for analysis is advantageous in obtaining the best test result.

NOTE IUPAC X-ray line notation (S K-$L_{2,3}$) is used in this International Standard; the corresponding Siegbahn X-ray line notation (S-Kα) is being phased out.

Where matrix matching is not used and where the C:H mass ratio of the test sample is known or can be determined, accuracy can be improved by the use of Equation (A.1) (see A.2.3) to correct the result to the C:H mass ratio of the calibration standards, i.e. the reference diluent oil (see 4.1).

Some instruments include the capability for instrument-based matrix correction; notes on the use of this approach to compensate for matrix effects in the test sample are provided in A.3 for information.

This International Standard is based on IP test method PM DU[3] developed by the Energy Institute.

Petroleum products — Determination of low concentration of sulfur in automotive fuels — Energy-dispersive X-ray fluorescence spectrometric method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies an energy dispersive X-ray fluorescence (EDXRF) test method for the determination of sulfur content in automotive gasoline containing up to 3,7 % (*m*/*m*) oxygen [including those blended with ethanol up to 10 % (*V*/*V*)], and in diesel fuels [including those containing up to about 10 % (*V*/*V*) fatty acid methylester (FAME)] having sulfur contents in the range 8 mg/kg to 50 mg/kg.

Other products can be analysed and other sulfur contents can be determined according to this test method; however, no precision data for products other than automotive fuels and for results outside the specified range have been established for this International Standard.

For reasons of spectral overlap, this International Standard is not applicable to leaded automotive gasoline, gasoline having a content of greater than 8 mg/kg lead replacement or to product and feedstock containing lead, silicon, phosphorus, calcium, potassium or halides at concentrations greater than one tenth of the concentration of sulfur measured or more than 10 mg/kg, whichever is the greater.

NOTE For the purposes of this International Standard, the terms "% (*m*/*m*)" and "% (*V*/*V*)" are used to represent the mass fraction, μ, and the volume fraction, ϕ, of a material respectively.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3170:2004, *Petroleum liquids — Manual sampling*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling*

3 Principle

The test portion, in a cup fitted with an X-ray transparent window, is placed in a beam of exciting radiation from an X-ray tube. The intensity of the sulfur K-$L_{2,3}$ characteristic X-radiation is measured and the accumulated count is compared with a calibration curve constructed from sulfur standards covering the range of sulfur contents under examination.

NOTE The exciting radiation can be either direct or indirect via a polarizing or secondary target.

4 Reagents and materials

4.1 Diluent oil

The reference diluent oil is white oil (light paraffin oil) of high purity grade, with a maximum sulfur content of 0,5 mg/kg. However, if only one type of matrix is to be analysed (e.g. motor gasoline), the accuracy of results