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Ethanol as a blending component for petrol - Determination of copper content - Graphite furnace atomic absorption spectrometric method

Ethanol zur Verwendung als Blendkomponente in Ottokraftstoff - Bestimmung des Kupfergehaltes - Graphitrohr-Atomabsorptions-Spektrometrie

Éthanol comme base de mélange à l'essence - Détermination de la teneur en cuivre - Méthode par spectrométrie d'absorption avec four en graphite

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English Version

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copper content - Graphite furnace atomic absorption
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Ottokraftstoff - Bestimmung des Kupfergehaltes -
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This European Standard was approved by CEN on 30 June 2007.

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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 Management Centre has the same status as the official versions.

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Contents

Page

Foreword.....	3
1 Scope	4
2 Normative reference	4
3 Terms and definitions	4
4 Principle.....	5
5 Reagents and materials	5
6 Apparatus	5
6.1 Atomic absorption spectrometer	5
7 Samples and sampling.....	6
8 Preparation of blank and calibration solutions	6
8.1 Intermediate calibration solution	6
8.2 Calibration solutions	6
9 Procedure	7
9.1 Instrument preparation.....	7
9.2 Calibration	7
10 Sample analysis.....	8
11 Calculation.....	8
12 Expression of results.....	8
13 Precision.....	8
13.1 Repeatability.....	8
13.2 Reproducibility.....	9
14 Test report	9
Annex A (informative) Example of thermal program of the graphite furnace	10
Bibliography	11

Foreword

This document (EN 15488:2007) 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.

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of an identical text or by endorsement, at the latest by February 2008, and conflicting national standards shall be withdrawn at the latest by February 2008.

This document is based on IP 478 [1].

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, 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 and United Kingdom.

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1 Scope

This standard specifies a procedure for the determination of copper content in ethanol from 0,07 mg/kg to 0,20 mg/kg using graphite furnace atomic absorption spectrometry.

NOTE For the purpose of this document, the terms “% (m/m)” and “% (V/V)” are used to represent the mass fraction, respectively the volume fraction of a material.

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

2 Normative reference

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.

EN ISO 3170, *Petroleum liquids — Manual sampling (ISO 3170:2004)*

EN ISO 3696, *Water for analytical laboratory use – Specification and test methods (ISO 3696:1987)*

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3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 radiant power

P

rate at which energy is transported in a beam of radiant energy

3.2 transmittance

T

ratio of the radiant power transmitted by a material to the radiant power incident upon it

3.3 absorbance

A

logarithm to the base 10 of the reciprocal of the transmittance

NOTE Can be derived by the following equation:

$$A = \log (1/T) = -\log (T) \quad (1)$$

3.4 integrated absorbance

A_i

integrated area under the absorbance peak generated by the atomic absorption spectrometer

4 Principle

The graphite furnace is aligned in the optical path of the atomic absorption spectrometer. A test portion is pipetted onto the platform or at the wall of the graphite furnace. The furnace is heated in consecutive thermal stages, firstly to dry the test portion completely without spattering, then to eliminate excess sample matrix, and finally to volatilise the analyte of interest. During this final stage, the amount of light absorbed by the copper atoms is measured over a specified period. The integrated absorbance A_i , produced by the copper in the test portion, is compared to a calibration curve constructed from copper standards in ethanol solution.

NOTE The second heating stage is used to eliminate as much of the sample matrix as possible before the atomization step, as non-dissociated molecular species are the most common form of interference. Spectrometers are equipped with background correction capabilities to control further possibilities of erroneous results due to molecular absorption.

5 Reagents and materials

5.1 General

All reagents shall be of analytical reagent grade or of higher purity.

5.2 Ethanol, 99,8 % (m/m) minimum purity.

5.3 Copper standard, aqueous solution containing 1 000 mg/l of copper as inorganic salt (e.g. nitrate), or a multi-element aqueous standard containing 1 000 mg/l of copper.

5.4 Water, for analytical laboratory use, conforming to grade 3 of EN ISO 3696.

5.5 Argon, regulated compressed gas of 99,999 % minimum purity for the graphite furnace gas flow system.

5.6 Nitric acid (HNO₃), $c(\text{HNO}_3) \approx 0,5 \text{ mol/l}$
Cautiously add 50 ml \pm 2 ml of nitric acid ($\rho = 1,40 \text{ g/ml}$) to 500 ml \pm 10 ml water (5.4).
Mix and allow to cool to room temperature.
Make up to 1 000 ml with water (5.4).

6 Apparatus

6.1 Atomic absorption spectrometer

6.1.1 Capable of setting the wavelength at 324,8 nm or 327,4 nm, with the appropriate slit, e.g. as specified by the manufacturer, and using peak area integration for the signal and background readings. It shall be equipped with the features described in 6.1.2 to 6.1.4.

NOTE The wavelength of 327,4 nm can be used for the measurements, however, the precision for results measured at this wavelength was not determined.

6.1.2 Copper hollow cathode lamp

6.1.3 Background correction system, capable to cover the 324,8 nm or 327,4 nm wavelength.

6.1.4 Graphite furnace accessory, which uses pyrolytically coated graphite tubes with or without integrated platforms.

6.2 Autosampler, capable of reproducibly delivering 10 $\mu\text{l} \pm 0,1 \mu\text{l}$ aliquots to the graphite furnace.

6.3 Micropipette, capable of reproducibly delivering volumes in the range 20 μl to 100 μl with an accuracy of $\pm 0,4 \mu\text{l}$ at 50 μl .

6.4 Polyethylene containers, 25 ml volume.
Rinse the containers with water (5.4).

6.5 Analytical balance, capacity of 100 g and capable of weighing to the nearest 0,01 g.

6.6 Ultrasonic bath

7 Samples and sampling

Unless otherwise specified, laboratory samples shall be obtained by the procedures described in EN ISO 3170. High density polyethylene containers shall be used. The containers should be carefully cleaned and rinsed with pure water to avoid contamination.

Samples should be analysed as soon as possible after removal from bulk supplies, to prevent loss of copper.

NOTE High density polyethylene containers are used to prevent losses of copper by wall adsorption.

8 Preparation of blank and calibration solutions

8.1 Intermediate calibration solution

Weigh a polyethylene container (6.4) and transfer with a micropipette (6.3) 100 µl of copper standard (5.3). Add water (5.4) to the container to bring the mass up to approximately 10,0 g and re-weigh to the nearest 0,01 g.

Seal the container, mix well and mark with the copper content calculated as following:

$$S = \frac{V_c}{m_w} \quad (2)$$

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where

S is the copper content of the intermediate calibration solution in mg/kg;

V_c is the volume of copper standard (5.3) in µl;

m_w is the mass of water (5.4) in g.

NOTE This procedure leads to intermediate calibration solution with nominal copper content corresponding to 10 mg/kg. The intermediate solution is stable for up to one week.

8.2 Calibration solutions

The calibration solutions shall be prepared daily.

Weigh five polyethylene containers (6.4), add about 5 g ethanol (5.2) to each container and then respectively 20 µl, 40 µl, 60 µl, 80 µl and 100 µl of intermediate calibration solution (8.1). Reserve a sixth container for the preparation of the blank.

Add ethanol (5.2) to each of the six containers to bring the mass up to approximately 10,0 g and re-weigh to the nearest 0,01 g.

Seal each container, mix well and mark with the copper content (including the blank) calculated as following:

$$C = \frac{V_i \cdot S}{m_e} \quad (3)$$

where

C is the copper content of the calibration solution in $\mu\text{g/kg}$;

S is the copper content of the intermediate calibration solution (8.1) in mg/kg ;

V_i is the volume of intermediate calibration solution (8.1) in μl ;

m_e is the mass of ethanol (5.2) in g.

NOTE This procedure leads to calibration solutions with nominal copper contents corresponding to 0 $\mu\text{g/kg}$, 20 $\mu\text{g/kg}$, 40 $\mu\text{g/kg}$, 60 $\mu\text{g/kg}$, 80 $\mu\text{g/kg}$ and 100 $\mu\text{g/kg}$ respectively.

9 Procedure

9.1 Instrument preparation

9.1.1 Set the spectrometer (6.1) at a wavelength of 324,8 nm or 327,4 nm, and the appropriate slit width.

9.1.2 Condition new (or re-installed) graphite tube assemblies with the temperature program provided by the spectrometer manufacturer.

9.1.3 Adjust the autosampler (6.2) tip as near as possible to the graphite platform surface but avoid contact between tip and surface. Use the blank solution (8.2) to evaluate the correct delivery of ethanol solutions. The delivered aliquot should distribute evenly onto the platform. Autosampler cups made of polyethylene, polypropylene or polytetra-fluorethylene (PTFE) can be used. Use nitric acid (5.6) as the rinse solution of the autosampler.

Due to the high volatility and surface tension of ethanol, the positioning of the tip and the correct delivery of sample aliquots should be checked periodically, as uneven delivery leads to erroneous measurements. Check the correct delivery of aliquots when the integrated absorbance of two repeated injections differ by more than 15 %.

9.2 Calibration

9.2.1 Apply the thermal program for copper analysis as given in the instrument manufacturer's instructions. Due to the different design of graphite furnace atomizers, the thermal program shall be developed according to the atomizer characteristics. Refer to the instrument manual to set the best temperatures for copper pyrolysis and atomization steps. A thermal program is given as an example in Annex A.

9.2.2 Temperatures, ramp and hold times of drying steps shall be optimised so that the sample dries completely without spattering.

9.2.3 The ramp time for the pyrolysis step may be lengthened if it appears that an excess amount of smoke from the sample matrix is generated very quickly as the furnace heats from the second drying step (Drying 2) to the pyrolysis. Also, all smoke shall be evolved at least 5 s before the end of the pyrolysis step. If smoke still evolves at the end of the pyrolysis step, the hold time shall be lengthened.

9.2.4 For the spectrometer trace of absorbance versus atomization hold time, the absorbance at the end of the atomization hold time should return to the initial baseline absorbance. If this is not observed, increase the atomization hold time until this is attained.