



SLOVENSKI STANDARD
SIST EN 15489:2007
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Ethanol as a blending component for petrol - Determination of water content - Karl Fischer coulometric titration method

Ethanol zur Verwendung als Blendkomponente in Ottokraftstoff - Bestimmung des Wassergehaltes - Coulometrisches Titrationsverfahren nach Karl Fischer

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Ethanol comme base de mélange à l'essence - Détermination de la teneur en eau - Méthode de titrage Karl Fischer par coulométrie

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ICS:

71.080.60	Alkoholi. Etri	Alcohols. Ethers
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ICS 71.080.60

English Version

Ethanol as a blending component for petrol - Determination of water content - Karl Fischer coulometric titration method

Ethanol comme base de mélange à l'essence -
Détermination de la teneur en eau - Méthode de titrage
coulométrique Karl Fischer

Ethanol zur Verwendung als Blendkomponente in
Ottokraftstoff - Bestimmung des Wassergehaltes -
Coulometrisches Titrationsverfahren nach Karl Fischer

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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Foreword

This document (EN 15489:2007) 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of an identical text or by endorsement, at the latest by February 2008, and conflicting national standards shall be withdrawn at the latest by February 2008.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 was prepared by CEN/TC 19’s Ethanol Task Force and is based on the Energy Institute standard IP 539 [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 European Standard specifies a method for the direct determination of water in ethanol to be used in gasoline blends. It is applicable to ethanol having water contents in the range 0,039 % (m/m) to 0,500 % (m/m).

NOTE For the purposes of this document, the term "% (m/m)" is used to represent the mass fraction.

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 determine the applicability of regulatory limitations prior to use.

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.

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 (standards.iteh.ai)

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

3.1 water content

content of water determined by coulometric Karl Fischer procedure as given in this document

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4 Principle

A weighed test portion is injected into the titration vessel of a coulometric Karl Fischer apparatus in which iodine for the Karl Fischer reaction is generated coulometrically at the anode. When all the water has been titrated, excess iodine is detected by an electrometric end-point detector and the titration is terminated. Based on the stoichiometry of the reaction, one mole of iodine reacts with one mole of water, thus the quantity of water is proportional to the total integrated current according to Faraday's Law.

5 Reagents and materials

Use only reagents of recognized analytical grade and water complying with the requirements of grade 3 of EN ISO 3696.

5.1 Xylene, water free

Remove dissolved water from the xylene by adding approximately 100 g of activated molecular sieve (5.3) to approximately 2 l of xylene. Allow to stand overnight.

5.2 Karl Fischer reagent, pyridine free. Use commercially available reagents that meet the performance requirements described in clause 9.

5.2.1 Anode electrolyte solution (anolyte)

5.2.2 Cathode electrolyte solution (catholyte)

5.2.3 Single Karl Fischer Reagents, for use in place of dual electrolyte solutions (5.2.1 and 5.2.2) in cells with or without a diaphragm.

5.3 Molecular sieve pellets

Activate by placing in an oven at 200 °C to 250 °C for 4 h. Remove from the oven and transfer immediately to either a dry glass bottle fitted with a gas-tight closure or a desiccator. Allow to cool before use.

6 Apparatus

6.1 Automatic coulometric Karl Fischer titrator, capable of meeting the requirements given in clause 9.

6.2 Syringes, of glass, with needles of suitable length such that the tip can reach under the surface of the anolyte when inserted through the inlet-port septum. The bores of the needles used shall be kept as small as possible, but large enough to avoid problems arising from back pressure or blocking whilst sampling.

NOTE 1 Needles with bores between 0,5 mm and 0,8 mm have been found suitable.

NOTE 2 Recommended syringe sizes are:

- a) 10 µl with a fixed needle for periodic checking of the titrator performance, and
- b) 1 ml or 2 ml for the ethanol test portion.

6.3 Balance, capable of weighing to 0,1 mg.

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7 Sampling and sample handling

7.1 Samples shall be taken as described in EN ISO 3170.

7.2 Take care to minimise the uptake of atmospheric moisture during sampling and sample handling.

NOTE The use of a glass bottle that can be sealed with a septum has been found suitable for sampling and sample handling. A test portion of the sample can be taken through the septum with a syringe fitted with a needle.

8 Apparatus preparation

8.1 Follow the manufacturer's directions for preparation and operation of the titration apparatus.

8.2 Seal all joints and connections to the titration cell to prevent atmospheric moisture from entering.

8.3 If a single Karl Fischer solution (5.2.3) is used, add this solution to the cell.

8.4 If separate electrolyte solutions are used, add the anolyte (5.2.1) to the outer compartment of the titration cell to the level recommended by the manufacturer. Add the catholyte (5.2.2) to the inner compartment of the titration cell to a level 2 mm to 3 mm below the level of the anolyte.

8.5 Turn on the titrator and stirrer. Allow the residual moisture in the titration cell to be titrated until the end-point is reached. Do not proceed beyond this stage until the background current (or background titration rate) is constant and less than the maximum recommended by the manufacturer of the instrument.

NOTE High background current for a prolonged period can be attributable to moisture on the inside walls of the titration cell. Gentle swirling of the cell will wash the inside with electrolyte. Also check all fittings to ensure atmospheric moisture does not enter the cell. It is recommended that the titrator be permanently switched on to stabilize to a low background current.

8.6 If the vessel becomes contaminated with a previously tested organic sample, thoroughly clean the anode and cathode compartments with xylene (5.1). If the frit becomes blocked, thoroughly clean with xylene. Do not use acetone or other ketones for cleaning or drying the apparatus.

9 Apparatus verification

The water titrated is a direct function of the coulombs of electricity consumed. However, reagent performance deteriorates with use and shall be regularly monitored by accurately injecting 10 µl of water. Suggested monitoring intervals are done initially when fresh reagents are used and then after every 10 injections.

Replace the anolyte (5.2.1) and catholyte (5.2.2) solutions, or the single Karl Fischer solution (5.2.3), whenever one of the following occurs:

- a) result from a 10 µl injection of water is outside 10 000 µg ± 200 µg, or persistently high;
- b) unstable background current, or phase separation in the outer compartment, or organic products coating the electrodes;
- c) total sample content of the titration cell exceeds one third of the volume of the anolyte;
- d) titrator displays error messages which suggests replacing the electrolytes (see the manufacturer's instructions).

10 Procedure

10.1 Using a clean, dry syringe (6.2) to withdraw at least three portions of the sample and discard as waste. Immediately withdraw a 1 ml to 2 ml test portion of the sample, clean the needle with a paper tissue and using the balance (6.3) weigh the syringe and contents to the nearest 0,1 mg.

10.2 Insert the needle through the inlet-port septum, start the titration and, with the tip of the needle just below the liquid surface, inject the entire test portion. Withdraw the syringe, wipe the needle with a clean tissue, and reweigh the syringe to the nearest 0,1 mg.

10.3 The apparatus will automatically titrate the water present. After the end-point is reached, record the water titrated from the display on the titrator (6.1).

11 Calculation

Calculate the water content of the sample, C_s , expressed in % (m/m), using the following equation:

$$C_s = \frac{m_2}{m_1 \times 10^4} \quad (1)$$

where

m_1 is the mass of the test portion, expressed in grams;

m_2 is the mass of water displayed by the titrator, expressed in micrograms.

12 Expression of results

Report the water content of the sample to the nearest 0,001 % (m/m).

13 Precision

13.1 General

The precision given was derived from statistical analysis by EN ISO 4259 [2] of the results of interlaboratory testing of a matrix of ethanol samples produced in Europe from bio materials such as raw wine, molasses, pulp and corn.

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NOTE The interlaboratory testing and the statistical evaluation are detailed in Research Report: IP 539 [2].

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13.2 Repeatability, r

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty.

$$r = 0,011\ 16 (X + 1) \quad (2)$$

where

X is the average of results being compared.

Typical values are given in Table 1.

13.3 Reproducibility, R

The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty.

$$R = 0,018\ 80 (X + 1) \quad (3)$$

where

X is the average of results being compared.