SLOVENSKI STANDARD

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Etanol kot komponenta za dodajanje motornemu bencinu ï Doloļ evanje pH vrednosti

Ethanol as a blending component for petrol - Determination of pHe

Ethanol zur Verwendung als Blendkomponente in Ottokraftstoff - Bestimmung des pHe-Wertes

Ethanol comme base de mélange a l'essence - Détermination du pHe

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Ethanol as a blending component for petrol - Determination of pHe

Ethanol comme base de mélange à l'essence - Détermination du pHe

Ethanol zur Verwendung als Blendkomponente in Ottokraftstoff - Bestimmung des pHe-Wertes

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

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 15490:2007) 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of an identical text or by endorsement, at the latest by February 2008, and conflicting national standards shall be withdrawn at the latest by February 2008.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 an extract and translation from a French Wine Regulation [1]. This method has been developed for European fuel ethanol specifications. Interlaboratory studies found that the method is not robust enough. Therefore it is better not to use it to determine the presence of any strong acids or alkalis that may be present in fuel ethanol. This Standard has insufficient precision to fulfill actual fuel specification needs (see Annex A). A test for strong acid and for the presence of alkaline substance will be developed by CEN/TC 19 to replace the pHe test method.

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.

1 Scope

This European Standard describes a procedure for the determination of acid strength, as a pHe value, of ethanol to be used as a blend component for gasoline.

For further background on the applicability of this document, the user is referred to the information in Annex A.

NOTE For the purposes of this European Standard, the term "% (*V/V*)" is used to represent the volume fraction.

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)*

3 Terms and definitions

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

3.1
pHe
measure of the acid strength

4 Principle

The pHe of a test portion of ethanol held at 22 °C ± 2 °C is measured using an electrode containing lithium chloride (LiCl) in an ethanol medium connected to a pH meter system.

NOTE In non-aqueous media, it is not possible to carry out absolute pH measurements and a pHe value is not directly comparable to pH values of water solutions.

5 Reagents and materials

Use only reagents of recognized analytical grade.

5.1 Buffer solutions, of pH (4,00 ± 0,02), pH (7,00 ± 0,02) and pH (10,00 ± 0,02), within their valid period for use.

CAUTION — Buffer solutions can deteriorate with time and it is important that they are used within their valid period of use.

NOTE Buffer solutions may be prepared from either commercially available tablets dissolved in water or commercially available aqueous solutions.

5.2 Ethanol, 99 % (*V/V*) containing 1 % (*V/V*) water.

5.3 Water, conforming to Grade 3 of EN ISO 3696.

5.4 Lithium chloride solution, 1mol/l in 99 % (*V/V*) ethanol (5.2).

5.5 Sodium hydroxide aqueous solution, approximately 1 mol/l

5.6 Hydrochloric acid aqueous solution, approximately 1 mol/l

6 Apparatus

6.1 pH Meter, capable of reading to 0,01 pH with an accuracy of ± 0,01 pH (or ± 1 mV), preferably fitted with a temperature compensator.

6.2 Combined pH electrode, filled with a 1 mol/l solution of lithium chloride (LiCl) in 99 % (*V/V*) ethanol.

6.3 Magnetic stirrer

6.4 PTFE covered magnetic follower.

6.5 Glass beakers, approximately 100 ml capacity fitted with a cover to minimise the ingress of carbon dioxide from the air.

NOTE The cover should either be a rigid plastic cover with a hole to allow the electrode to pass through or a film of disposable plastic placed over the top of the beaker after the electrode has been inserted.

6.6 Timer, capable of measuring seconds.

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 water 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 Clean/rehydrate new electrodes before use in alcoholic solutions and after measuring the pHe of 10 ethanol samples by alternatively soaking them in 1 mol/l sodium hydroxide solution (5.5) for approximately 30 s then 1 mol/l hydrochloric acid solution (5.6) for approximately 30 s. Repeat this procedure 3 times.

8.2 Wash the electrode free of acid and alkali with water (5.3) and allow draining. If the electrode is not to be used immediately, place in and store in a solution of lithium chloride in ethanol (5.4).

9 pH meter and electrode system calibration

9.1 Set up the pH meter in accordance with the manufacture's instructions and, if fitted, adjust the temperature compensator to 22 °C ± 2°C.

9.2 Remove the combined electrode from the lithium chloride solution (5.4), wash with water (5.3) and allow draining.

9.3 Rinse the combined electrode (6.2) with pH 4,00 buffer solution (5.1).

9.4 In a 100 ml beaker (6.5), place sufficient volume of pH 4,00 buffer solution (5.1) at 22 °C ± 2°C to cover the electrode. Insert the magnetic follower (6.6).

9.5 Place the beaker and its contents on the magnetic stirrer (6.3).

NOTE Automatic pH meters may require the user to input the pH value of the buffer solution.

9.6 Place the combined electrode (6.2) into the pH 4,00 buffer solution (5.1). Cover the beaker (see NOTE under 6.5).

9.7 Switch on the magnetic stirrer (6.3) and allow the pH meter reading to stabilize.

9.8 If necessary adjust the reading to 4,00.

9.9 Remove the combined electrode and rinse it with water (5.3).

9.10 Repeat 9.3 to 9.9 using the pH 10 buffer solution (5.1) (see NOTE under 9.5). If necessary, adjust the pH meter reading to 10,00 using the slope adjustment.

10 Apparatus verification

10.1 Before testing each day and after cleaning/rehydrating the electrode, see clause 8, verify the correct functioning of the electrode/pH meter system using a pH 7,00 buffer solution (5.1) in accordance with 10.2 to 10.7.

10.2 Rinse the combined electrode with water (5.3) then with pH 7,00 buffer solution (5.1).

10.3 In a 100 ml beaker (6.5), place sufficient volume of pH 7,00 buffer solution (5.1) at 22 °C ± 2°C to cover the electrode. Insert the magnetic follower (6.4).

10.4 Place the beaker and its contents on the magnetic stirrer (6.3).

10.5 Place the combined electrode (6.2) into the pH 7,00 buffer solution (5.1), cover the beaker (see NOTE under 6.5).

10.6 Switch on the magnetic stirrer (6.3) and allow the pH meter reading to stabilize (see NOTE under 9.5).

10.7 The pH meter shall read pH 7,00 ± 0,04.

10.8 If the reading is not within the tolerance in 10.7, remove the combined electrode, clean in accordance with 8.1 and repeat 10.1 to 10.6.

10.9 If the reading is still not within the tolerance in 10.7, recalibrate the electrode and pH meter in accordance with clause 9.

11 Procedure

11.1 If a temperature compensator is fitted to the pH meter, check and, if necessary, adjust it to 22 °C ±2 °C.

11.2 Bring the ethanol (5.2) to be tested to 22 ºC ± 2 ºC and place sufficient volume of it to cover the electrode in a 100 ml beaker (6.5).

11.3 Place the beaker and the test portion on a magnetic stirrer (6.3). Insert the magnetic follower (6.4) and the electrode and cover the beaker to minimise the ingress of atmospheric carbon dioxide.

11.4 Stir the test portion, allow the pHe reading to stabilize, see note. Record the pHe to the nearest 0,01.

NOTE For some samples this may be in excess of 4 min. In such circumstances extra care should be taken to minimise the ingress of atmospheric carbon dioxide during the measurement.

12 Expression of results

Report the pHe of the sample to the nearest 0,01.

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. Typical values are given in Table 1.

NOTE The interlaboratory testing and the statistical evaluation are detailed in an UNGDA Report, September 2006 (UNGDA, 174 blvd Camélinat, 92247 MALAKOFF Cedex, France, www.ungda.org).

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.

$$r = 0{,}033\,X \qquad (1)$$

where

X is the average of results being compared.

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.

$$R = 0{,}096\,X \qquad (2)$$

where

X is the average of results being compared.