BRITISH STANDARD

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Ethanol as a blending component for petrol — Determination of total acidity — Colour indicator titration method

The European Standard EN 15491:2007 has the status of a British Standard

ICS 71.080.60

BSI
British Standards

National foreword

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The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum testing and terminology.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Ethanol as a blending component for petrol - Determination of total acidity - Colour indicator titration method

Ethanol comme base de mélange à l'essence - Détermination de l'acidité totale - Méthode de titrage par indicateur coloré

Ethanol zur Verwendung als Blendkomponente in Ottokraftstoff - Bestimmung der Gesamtsäurezahl - Farbindikator-Titration

This European Standard was approved by CEN on 30 June 2007.

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Foreword

This document (EN 15491:2007) 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of an identical text or by endorsement, at the latest by February 2008, and conflicting national standards shall be withdrawn at the latest by February 2008.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 538 [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.

1 Scope

This European Standard specifies a method for determining the total acidity, calculated as acetic acid, of ethanol to be used in petrol blends. It is applicable to ethanol having total acid contents of between 0,003 % (*m/m*) to 0,015 % (*m/m*).

NOTE For the purposes of this European Standard, the term "% (*m/m*)" and "% (*V/V*)" are used to represent the mass fraction and the volume fraction respectively.

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)*

3 Terms and definitions

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

3.1
total acidity
acidity, calculated as acetic acid, determined by titration and colour indicator as given in this standard

4 Principle

A test portion of the ethanol is mixed with an equal volume of neutralized, carbon dioxide free water. The acid content is titrated with potassium hydroxide solution, to the neutral endpoint of phenolphthalein. The total acidity is then calculated as acetic acid.

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 Potassium hydrogen phthalate

5.2 Potassium hydroxide solution 0,01 mol/l, a solution prepared in accordance with 5.2.1 or a commercially available standardized potassium hydroxide solution of equivalent concentration and purity. The reagent shall be protected against carbon dioxide absorption and restandardized frequently enough to detect concentration changes of 0,000 5 mol/l.

5.2.1 Dissolve approximately 0,6 g potassium hydroxide in 1 l of water and standardize using potassium hydrogen phthalate in accordance with 5.2.2.

5.2.2 Dry a quantity of potassium hydrogen phthalate (5.1) in an oven at approximately 120 °C for approximately 2 h. Place in a desiccator and allow to cool. Weigh approximately 0,1 g to the nearest 0,1 mg into a 250 ml flask and record this mass. Add approximately 50 ml of carbon dioxide free water (5.5) and swirl to dissolve. Add 2 drops of phenolphthalein indicator solution (5.3) and using a 50 ml burette (6.5), titrate to neutral endpoint with the potassium hydroxide solution. Carry out a blank determination using the same volume of carbon dioxide free water (5.5). Calculate the concentration *C*, in moles per litre, of the potassium hydroxide solution from the equation:

$$C = \frac{1000m}{204{,}23(V_1 - V_0)} \qquad (1)$$

where

m is the mass, in grams, of potassium hydrogen phthalate;

V_1 is the volume, in millilitres, of potassium hydroxide solution for the titration;

V_0 is the volume, in millilitres, of potassium hydroxide used for the blank.

5.3 Phenolphthalein indicator solution, approximately 10g/l

Weigh approximately 1 g of phenolphthalein into the 100 ml volumetric flask (6.1). Add approximately 20 ml of ethanol (5.4) and swirl until dissolved. Make up to 100 ml with ethanol.

5.4 Ethanol, approximately 95 % (*V/V).*

5.5 Carbon dioxide free water

NOTE A suitable way of preparing carbon dioxide free water is to place approximately 100 ml of water in a 250 ml conical flask (6.3), fitted with a standard ground glass joint, heat to boiling on either a hot plate or gas burner and boil for 2 min to 3 min. Remove the flask and its contents from the heat and insert a soda-lime filled guard tube (6.7) and cool to ambient temperature before use.

5.6 Soda lime, for the guard tube (optional).

5.7 Nitrogen, carbon dioxide free (optional).

6 Apparatus

6.1 Volumetric flask, Class A, 100 ml capacity.

6.2 Measuring cylinder, 100 ml capacity.

6.3 Conical flask, glass, with standard ground glass joint, approximately 250 ml capacity.

6.4 Burette, Class A 50 ml capacity.

6.5 Burette, Class A 10 ml capacity and graduated in 0,05 ml, or less, subdivisions.

6.6 Pipette, Class A 50 ml capacity.

6.7 Glass guard tube, with ground glass joint to fit the conical flask (6.3) (optional).

7 Sampling and sample handling

7.1 Unless otherwise specified, laboratory samples shall be obtained by the procedures described in EN ISO 3170.

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

8 Procedure

8.1 Fill the 10 ml burette (6.5) with the potassium hydroxide solution (5.2).

8.2 Using the measuring cylinder (6.2) measure approximately 50 ml of carbon dioxide free water (5.5) into the conical flask (6.3). Add two drops of phenolphthalein solution (5.3). Titrate with the standardized potassium hydroxide solution (5.2) to a faint pink endpoint.

8.3 Using the pipette (6.6) add 50 ml of the test portion to the neutralised water. Stopper the flask and swirl to mix the test portion and the water.

8.4 Remove the stopper and immediately titrate the mixture using the standardized potassium hydroxide to a faint pink end point. Take care not to prolong the titration as the up-take of atmospheric carbon dioxide by the ethanol water mix will appreciably affect the result.

NOTE Atmospheric carbon dioxide can be prevented from entering the titration flask by bubbling nitrogen (5.7) through the solution during the titration.

8.5 If the density of the ethanol to be tested is not known, determine it, in g/ml, at 15 °C to two decimal places.

9 Calculation

Calculate the total acidity, A_s, as acetic acid, of the sample in % (*m/m*), using the following equation:

$$A_s = \frac{VC \times 0{,}12}{\rho} \qquad (2)$$

where

C is the concentration, in moles per litre, of potassium hydroxide solution, see equation (1);

V is the volume, in millilitres, of potassium hydroxide solution required to neutralize 50 ml of test portion;

ρ is the density, in grams per millilitre, of the test portion at 15 °C.

10 Expression of results

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

11 Precision

11.1 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 biomaterials such as raw wine, molasses, pulp and corn.

NOTE The interlaboratory testing and the statistical evaluation are detailed in Research Report IP 538 [3].

11.2 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 = 0,000 960 4

11.3 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 = 0,001 370

12 Test report

The test report shall contain at least the following information:

a) reference to this European Standard, i.e. EN 15491;

b) type and complete identification of the product tested;

c) result of the test (see 10);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) date of the test.

Bibliography

[1] IP 538/06, *Determination of the total acidity of ethanol – Colour indicator titration method*. Available from the Energy Institute, 61 New Cavendish Street, London, W1G 7AR, UK.

[2] EN ISO 4259, *Petroleum products — Determination and application of precision data in relation to methods of test (ISO 4259:2006).*

[3] Research Report: IP 538/06, *Precision evaluation on IP 538, Determination of the total acidity of ethanol – Colour indicator titration method.* Available from the Energy Institute, 61 New Cavendish Street, London, W1G 7AR, UK.

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