SLOVENSKI STANDARD
oSIST prEN 15491:2021

01-april-2021

Etanol kot komponenta za dodajanje motornemu bencinu - Določevanje celotne kislosti - Titracijska metoda z barvnim indikatorjem

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

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

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

Ta slovenski standard je istoveten z: **prEN 15491**

ICS:

71.080.60	Alkoholi. Etri	Alcohols. Ethers
75.160.20	Tekoča goriva	Liquid fuels

oSIST prEN 15491:2021 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 15491

April 2021

ICS 75.160.20; 71.080.60

Will supersede EN 15491:2007

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 draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 19.

If this draft becomes a European Standard, 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.

This draft European Standard was established by CEN 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, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

Warning : This document is not a European Standard. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a European Standard.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Contents

Page

European foreword 3
1 Scope 4
2 Normative references 4
3 Terms and definitions 4
4 Principle 4
5 Reagents and materials 5
6 Apparatus 6
7 Sampling and sample handling 6
8 Procedure 6
9 Calculation 7
10 Expression of results 7
11 Precision 7
11.1 General 7
11.2 Repeatability, *r* 7
11.3 Reproducibility, *R* 7
12 Test report 8
Bibliography 9

European foreword

This document (prEN 15491:2021) 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 document is currently submitted to the CEN Enquiry.

This document will supersede EN 15491:2007. It was originally prepared by CEN/TC 19’s Ethanol Task Force and is based on the Energy Institute standard IP 538 [1].

In comparison with the previous edition, the following technical modification has been made:

— the purging step (8.4) has been made mandatory for clarification to the user and for better comparison of the results. This has no effect on the method precision.

1 Scope

This document 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*) and 0,015 % (*m/m*).

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

WARNING — Use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the document, and to fulfil statutory and regulatory restrictions for this purpose.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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)*

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

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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

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 end point 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 end point 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 formula:

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

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, heated to boiling on either a hot plate or gas burner and boiled for 2 min to 3 min. The flask and its contents are removed from the heat, a soda-lime (5.6) filled guard tube (6.7) is inserted, and cooled to ambient temperature before use.

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

5.7 Nitrogen, carbon dioxide free.

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 minimize 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 end point.

8.3 Using the pipette (6.6) add 50 ml of the test portion to the neutralized 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.

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

NOTE The up-take of atmospheric carbon dioxide by the ethanol water mix affects the result.

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 formula:

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

where

C is the concentration, in moles per litre, of potassium hydroxide solution, see Formula (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 4259:2006 [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 1 The interlaboratory testing and the statistical evaluation are detailed in Research Report IP 538 [3].

NOTE 2 The precision was later checked and confirmed by a further analysis following EN ISO 4259-1:2017 [4].

11.2 Repeatability, *r*

The difference between two independent results obtained using this method for test material considered to be the same in the same laboratory, by the same operator using the same equipment within short intervals of time, in the normal and correct operation of the method that is expected to be exceeded with a probability of 5 % due to random variation, conforms to the following:.

r = 0,000 960 4

11.3 Reproducibility, *R*

The difference between two independent results obtained using this method for test material considered to be the same in different laboratories, where different laboratory means a different operator, different equipment, different geographic location, and under different supervisory control, in the normal and correct operation of the method that is expected to be exceeded with a probability of 5 % due to random variation, conforms to the following:

R = 0,001 370

12 Test report

The test report shall contain at least the following information:

a) reference to this document, i.e. prEN 15491:2021;

b) type and complete identification of the product tested;

c) result of the test (see Clause 10);

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

e) date of the test.