



SLOVENSKI STANDARD

SIST EN 15691:2009

01-junij-2009

Etanol kot komponenta za dodajanje motornemu bencinu - Določevanje celotnega suhega ostanka (nehlapna snov) - Gravimetrična metoda

Ethanol as a blending component for gasoline - Determination of total dry residue (involatile material) - Gravimetric method

Ethanol zur Verwendung als Blendkomponente in Ottokraftstoff - Bestimmung des gesamten Trockenrückstandes (nichtflüchtige Bestandteile) - Gravimetrisches Verfahren

Ethanol comme constituant d'essence - Détermination de la residue totale (matériau volatil) - Méthode gravimétrique

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Ta slovenski standard je istoveten z: EN 15691:2009

ICS:

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

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en,fr,de

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EUROPEAN STANDARD

EN 15691

NORME EUROPÉENNE

EUROPÄISCHE NORM

March 2009

ICS 75.160.20

English Version

Ethanol as a blending component for petrol - Determination of dry residue (involatile material) - Gravimetric method

Ethanol comme base de mélange à l'essence -
Détermination du résidu sec (produits non volatils) -
Méthode gravimétrique

Ethanol zur Verwendung als Blendkomponente in
Ottokraftstoff - Bestimmung des Trockenrückstandes
(nichtflüchtige Bestandteile) - Gravimetrisches Verfahren

This European Standard was approved by CEN on 24 January 2009.

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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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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Page

| | |
|--|---|
| Foreword..... | 3 |
| 1 Scope | 4 |
| 2 Normative references | 4 |
| 3 Principle | 4 |
| 4 Apparatus | 4 |
| 5 Sampling | 4 |
| 5.1 Preparation of samples | 4 |
| 5.2 Verification and quality control | 5 |
| 6 Procedure | 5 |
| 7 Calculation..... | 5 |
| 8 Expression of results | 5 |
| 9 Precision | 5 |
| 9.1 General..... | 5 |
| 9.2 Repeatability, r | 6 |
| 9.3 Reproducibility, R | 6 |
| 10 Test report | 6 |
| Bibliography | 7 |

[SIST EN 15691:2009](https://standards.iteh.ai/catalog/standards/sist/20d798d2-aa25-4f53-b99f-055d96f37049/sist-en-15691-2009)

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Foreword

This document (EN 15691:2009) 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 September 2009, and conflicting national standards shall be withdrawn at the latest by September 2009.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document was prepared by CEN/TC 19's Ethanol Task Force under responsibility of its Working Group 21 and is based on a test method mentioned in a European Wine Regulation [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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EN 15691:2009 (E)**1 Scope**

This European Standard specifies a procedure for the determination of dry residue in ethanol by gravimetric (desiccation) method in the range (10 to 25) mg/100 ml.

NOTE In an interlaboratory study [2] the method described has been tested at levels down to 3,5 mg/100 ml, but the precision appeared to be insufficient at such low levels.

WARNING — Use of this document may involve hazardous equipment, materials and operations. This method does not purport to address to all of the safety problems associated with its use, but it is the responsibility of the user to search and 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)*

3 Principle

Dry residue is determined by the weighing of the residue left by evaporation of alcohol on a boiling water bath and drying in a drying oven.

Dry residue includes all matter that is non-volatile under specified physical conditions.

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4 Apparatus

4.1 Evaporating dish (100 to 250 ml).

4.2 Boiling water bath.

4.3 Pipette, 100 ml, class A.

4.4 Oven, capable of being held at a temperature of (103 ± 2) °C.

4.5 Desiccator, containing freshly activated silica gel (or equivalent desiccant) with moisture content indicator.

4.6 Analytical balance, capable of weighing with an accuracy of 0,1 mg.

5 Sampling**5.1 Preparation of samples**

Unless otherwise specified in the commodity specification, samples shall be taken as described in EN ISO 3170.

Collect the samples in glass bottles. Samples shall be stored at room temperature prior to analysis.

5.2 Verification and quality control

It is recommended to prepare a sodium chloride solution with a content of 100 mg/l in ethanol from neutral ethanol free of dry residue and from an aqueous sodium chloride solution the content of which is 10 g/l. For example, 10 ml of the aqueous solution can be introduced in 1 l of this neutral alcohol without residue. This is a solution with a dry residue content of 10 mg/100 ml.

6 Procedure

Place clean dry evaporating dishes (4.1) into the oven for 30 minutes, then place them into the desiccator (4.5) for 30 minutes.

Use a tool to manipulate dishes, do not touch directly with fingers.

Accurately weigh, to the nearest 0,1 mg, the clean dry evaporating dishes (4.1) (M_0).

Pipette (4.3) 100 ml of sample or quality control and introduce respectively into the dishes. Place the dishes with sample on the boiling water bath (4.2) and allow to dry.

NOTE In general, it takes about two hours to evaporate the alcohol on the water bath.

Place the dishes in the oven (4.4) at $(103 \pm 2) ^\circ\text{C}$ for 30 minutes and then transfer dishes with residue into a desiccator (4.5). Allow the dishes to cool for 30 minutes and then weigh, to the nearest 0,1 mg, the dishes with residue (M_1).

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7 Calculation

The content of dry residue, D , expressed in mg/100 ml is given by:

$$D = M_1 - M_0 \quad (1)$$

where

D is the expression for dry residue,

M_1 is the mass, in mg, of the dish and residue after drying,

M_0 is the mass, in mg, of the clean dry dish.

8 Expression of results

Report the content of dry residue rounded to the nearest 1 mg/100 ml.

9 Precision

9.1 General

The precision given was derived from statistical analysis by EN ISO 4259 [3] of the results of interlaboratory testing of a matrix of ethanol liquids, produced from biomaterials such as wine (fermentation result), molasses, pulp and corn.

EN 15691:2009 (E)

NOTE The interlaboratory testing and the statistical evaluation are detailed in [2]. In some occasions the repeatability is larger than the reproducibility, caused by outliers, but the evaluation did not show major mistakes in the set up of the interlaboratory testing.

The term “single test result” means the value obtained when the standardized test method is applied fully once to a single sample. Unless otherwise stated, the probability shall be 95 %.

9.2 Repeatability, r

The repeatability limit is the value below which the absolute difference between two single test results obtained under the same conditions (same operator, same apparatus, same laboratory and a short interval of time) should exceed the following value in mg/100 ml in one case in twenty:

$$r = 0,191 X + 0,000 55 \quad (2)$$

where

X is the average value of results being compared.

9.3 Reproducibility, R

The reproducibility limit is the value below which the absolute difference between two single test results obtained under different conditions (different operators, different apparatus and/or different laboratories and/or different time) should not exceed the following value in mg/100 ml in one case in twenty:

$$R = 0,184 8 X + 0,000 81 \quad (3)$$

where

X is the average value of results being compared.

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10 Test report

The test report shall contain at least the following information:

- a) reference to this European Standard, i.e. EN 15691;
- b) all information necessary for the complete identification of the sample;
- c) applied sampling procedure (see 5.1);
- d) results of the test (see 8);
- e) any deviation, by agreement or otherwise, from the procedure specified in this European Standard, or regarded as optional;
- f) details of any incidents which may have influenced the test results;
- g) date of the test.