



**SLOVENSKI STANDARD**  
**SIST EN 15484:2007**  
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Ethanol as a blending component for petrol - Determination of inorganic chloride - Potentiometric method

Ethanol zur Verwendung als Blendkomponente in Ottokraftstoff - Bestimmung des gehalts an anorganischen Chloriden aus dem Eindampfrückstand - Potentiometrisches Verfahren

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Ethanol comme base de mélange a l'essence - Dosage du chlorure inorganique - Méthode potentiométrique

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EUROPEAN STANDARD

EN 15484

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ICS 71.080.60

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## Ethanol as a blending component for petrol - Determination of inorganic chloride - Potentiometric method

Ethanol comme base de mélange à l'essence - Dosage du chlorure minéral - Méthode potentiométrique

Ethanol zur Verwendung als Blendkomponente in Ottokraftstoff - Bestimmung des Gehalts an anorganischen Chloriden aus dem Eindampfrückstand - Potentiometrisches Verfahren

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

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre or to any CEN member.

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  
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## Foreword

This document (EN 15484:2007) 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of an identical text or by endorsement, at the latest by February 2008, and conflicting national standards shall be withdrawn at the latest by February 2008.

The method described in this document is based on ISO 6227 [1] and a method from a European Regulation on wine [2].

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 standard specifies a potentiometric method for the determination of inorganic chloride content in ethanol from 4 mg/l to 30 mg/l. The chloride content is determined in aqueous solution after dissolution of the evaporation residue of the ethanol sample.

**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 to determine the applicability of regulatory limitations prior to use.**

NOTE For the purposes of this document, the terms “% (m/m)” and “% (V/V)” are used to represent respectively the mass fraction and 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 Principle

A weighed test portion of ethanol sample is evaporated on a water bath. The dry residue is dissolved in deionised water. Inorganic chloride content is determined by potentiometric titration either manually or using automated equipment.

## 4 Reagents and materials

All reagents shall be of analytical reagent grade or of higher purity.

**4.1 Acetone.**

**4.2 Nitric acid**,  $\rho$  approximately 1,40 g/ml, about 68 % (m/m) solution.

**4.3 Potassium nitrate**, solution saturated at room temperature.

**4.4 Silver nitrate solution 1**,  $c(\text{AgNO}_3) = 0,1 \text{ mol/l}$  approximately. Dissolve 8,5 g of silver nitrate in a 500 ml one-mark volumetric flask, dilute to the mark and mix. Store in a dark glass bottle.

**4.5 Silver nitrate solution 2**,  $c(\text{AgNO}_3) = 0,004 \text{ mol/l}$  approximately. Prepare the solution at the time of use from silver nitrate solution 1 (4.4), diluting when required in a one-mark volumetric flask.

**4.6 Potassium chloride standard reference solution 1**,  $c(\text{KCl}) = 0,100 \text{ mol/l}$ . Weigh 3,727 6 g of potassium chloride to the nearest 0,000 1 g. Potassium chloride was previously dried for 1 h at about 130 °C and cooled in a desiccator. Dissolve in a little water and transfer the solution quantitatively into a 500 ml one-mark volumetric flask. Dilute to the mark and mix. This solution shall not be kept for more than one month.

**4.7 Potassium chloride standard reference solution 2**,  $c(\text{KCl}) = 0,004 \text{ mol/l}$ . Prepare the solution at the time of use from the standard reference potassium chloride solution 1 (4.6), diluting when required in a one-mark volumetric flask.

**4.8 Water**, for analytical laboratory use, conforming to grade 3 of EN ISO 3696.

## 5 Apparatus

**5.1 Evaporating dish**, capacity 100 ml to 250 ml.

**5.2 Water bath**

**5.3 Drying oven**, thermostatically controlled at  $(105 \pm 2) ^\circ\text{C}$ .

**5.4 Dessicator**, containing freshly activated silica gel (or equivalent desiccant) with moisture a content indicator.

**5.5 Analytical balance**, capable of weighing to the nearest 0,1 mg.

**5.6 Pipette**, volumetric, class A, capable of delivering 25 ml.

**5.7 Potentiometric titration apparatus**, comprising:

**5.7.1 Potentiometer**, sensitivity at least 2 mV, covering the range  $-500 \text{ mV}$  to  $+500 \text{ mV}$ .

**5.7.2 Reference electrodes.** (standards.iteh.ai)

**5.7.2.1 Calomel electrode**, fitted with a reservoir filled with saturated potassium chloride solution, or

[SIST EN 15484:2007](#)

**5.7.2.2 Double junction silver/silver chloride electrode.** The outer electrolyte must be saturated potassium nitrate solution (4.3), the inner electrolyte may be potassium chloride 3 mol/l or saturated potassium nitrate (4.3).

**5.7.3 Bridge**, containing saturated potassium nitrate solution (4.3), to be connected to the calomel electrode (5.7.2.1) and fitted with porous diaphragms at the ends.

**5.7.4 Measuring electrodes**, being either

**5.7.4.1 Silver electrode**, or

**5.7.4.2 Chloride or silver ion-selective electrode.**

**5.8 One-mark flasks**, 25 ml volume.

**5.9 Magnetic stirrer**, with a polytetrafluorethylene (PFTE)-coated rod.

**5.10 Burettes**, with fine point tip, graduated in 0,02 ml or 0,01 ml divisions.

## 6 Samples and sampling

Unless otherwise specified, laboratory samples shall be obtained by the procedures described in EN ISO 3170. High density polyethylene containers shall be used. The containers should be carefully cleaned and rinsed with pure water before use to avoid contamination.

Samples should be analysed as soon as possible after removal from bulk supplies, to prevent loss of chloride.

Thoroughly mix samples in their containers immediately prior to drawing test portions.

## 7 Calibration

### 7.1 Preparation of the blank test solution

At the same time as the determination, carry out a blank test, using 25 ml of water (4.8).

### 7.2 Preparation of a 5,00 ml standard solution

**7.2.1** Place the blank test solution in a beaker of suitable capacity. Add, using one of the burettes (5.10), 5,00 ml of the potassium chloride solution (4.7).

**7.2.2** Add a quantity of the acetone (4.1) to obtain a ratio of acetone to aqueous solution preferably equal to 80/10 (V/V) but, in any case, not lower than 50/50 (V/V).

### 7.3 Titration

Introduce into the beaker a magnetic stirrer rod (5.9), place the beaker on the stirrer and set it in motion. Introduce in the solution the measuring electrode (5.7.4) and the reference electrode (5.7.2). If, however, a calomel electrode (5.7.2.1) is used, introduce one end of the bridge (5.7.3) instead of the reference electrode and immerse the other end in a beaker of water containing the calomel electrode. Connect the electrodes to the potentiometer (5.7.1) and record the initial value of the potential, after having checked the zero setting of the instruments.

Execute the titration by adding silver nitrate solution (4.5) in successive increments of 0,1 ml each. After each addition, wait for the potential to reach the steady value.

In the first two columns of a table, note the successive volumes added and the corresponding potentials.

In a third column, note the successive increments ( $\Delta_1 E$ ) of the potential  $E$ .

In a fourth column, note the differences ( $\Delta_2 E$ ), positive or negative, between the successive increments ( $\Delta_1 E$ ) of the potential  $E$ .

The end of the titration corresponds to the addition of the 0,1 ml ( $V_1$ ) of the silver nitrate solution, which gives the maximum value of  $\Delta_1 E$ .

The exact volume ( $V_{eq}$ ), in ml, of silver nitrate solution (4.5) corresponding to the end of the reaction is given by the equation (for better comprehension see the example in Annex A):

$$V_{eq} = V_0 + \left( V_1 \cdot \frac{b}{B} \right) \quad (1)$$

where

$V_0$  is the volume, in ml, of the silver nitrate solution (4.5) which gives the maximum increment of  $\Delta_1 E$ ;

$V_1$  is the volume, in millilitres, of the silver nitrate solution (4.5);

$b$  is the first negative increment of  $\Delta_2 E$ ;

$B$  is the sum of the absolute values of the last positive increment of  $\Delta_2 E$  and the first negative increment of  $\Delta_2 E$ .



#### 7.4 Standardisation of 10,00 ml potassium chloride solution.

Repeat the operations specified in **Error! Reference source not found.** and 7.3, this time using 10,00 ml instead of 5,00 ml, of potassium chloride solution (4.7).

#### 7.5 Calculation of concentration of silver nitrate solution

The concentration of the silver nitrate solution,  $c(\text{AgNO}_3)$ , expressed as moles of  $\text{AgNO}_3$  per litre, is given by the equation:

$$c(\text{AgNO}_3) = c(\text{KCl}) \cdot \frac{5}{(V_2 - V_3)} \quad (2)$$

where

$c(\text{KCl})$  is the concentration, in mol/l, of potassium chloride solution (4.7);

$V_2$  is the value, in ml, of  $V_{\text{eq}}$  corresponding to the titration of 10 ml of potassium chloride solution (4.7), in the presence of blank test solution;

$V_3$  is the value, in ml, of  $V_{\text{eq}}$  corresponding to the titration of 5 ml of potassium chloride solution (4.7), in the presence of blank test solution;

5 being the difference, in ml, between the two volumes of potassium chloride solution (4.7).

#### 7.6 Calculation of value of blank test

The value of the reagent blank test,  $V_4$ , is given, in ml, by the equation:

$$V_4 = 2V_3 - V_2 \quad (3)$$

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where  $V_2$  and  $V_3$  are as defined in 7.5.

## 8 Sample analysis

**IMPORTANT — One should always be aware of interferences originating from specific elements in the sample. Some known interferences are indicated in Annex B.**

### 8.1 Preparation of the dry residue

Add 25 ml ( $V_E$ ) of test portion to the evaporating dish (5.1). Place the dish with sample on the boiling water bath (5.2) and allow to dry. Place the dish in the oven (5.3) at 105 °C for 30 min and then transfer the dish to a desiccator (5.4). Allow the dish to cool for 30 min.

Add 10 ml of water (4.8), to the evaporating dish and heat gently until complete dissolution of the dry residue. Transfer the solution to a 25 ml one-mark flask. Repeat the treatment with 10 ml water. Add water to the mark.

### 8.2 Preparation of the sample solution

Transfer the sample solution (8.1) in to a suitable beaker. Further proceed as specified in **Error! Reference source not found.**2.

If the chloride ion content is very low, proceed from 8.1 using a larger volume of test portion ( $V_E$ ).