SLOVENSKI STANDARD
oSIST prEN 15938:2009

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Goriva za motorna vozila - Etanolna komponenta za dodajanje in gorivo etanol (E85) - Doloļ evanje elektriļ ne prevodnosti

Automotive fuels - Ethanol blending component and ethanol (E85) fuel - Determination of electrical conductivity

Kraftstoffe für Kraftfahrzeuge - Ethanol Mischungskomponent und Ethanolkraftstoff (E85) - Bestimmung der elektrischen Leitfähigkeit

Carburants pour automobiles - Ethanol comme base de mélange à l'essence et carburant éthanol (E85) - Détermination de la conductivité électrique

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English Version

Automotive fuels - Ethanol blending component and ethanol (E85) fuel - Determination of electrical conductivity

Carburants pour automobiles - Ethanol comme base de mélange à l'essence et carburant éthanol (E85) - Détermination de la conductivité électrique

Kraftstoffe für Kraftfahrzeuge - Ethanol Mischungskomponent und Ethanolkraftstoff (E85) - Bestimmung der elektrischen Leitfähigkeit

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document prEN 15938:2009 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 document is currently submitted to the CEN Enquiry.

This document was prepared by CEN/TC 19’s Ethanol Task Force under responsibility of its Working Group 21 and is based on DIN 51627-4 [1].

1 Scope

This document specifies a test method for the determination of the electrical conductivity in ethanol and ethanol fuel in the range from approximately (0,3 to 5) µS/cm at a temperature of 25 °C. The electrical conductivity is determined from the measured electrical conductance.

The electrical conductivity is an important analytical criterion for the ascertainment and control of anionic and cationic components in ethanol and ethanol fuel. Some of these components can exhibit corrosive properties.

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 3171, *Petroleum liquids — Automatic pipeline sampling (ISO 3171:1988)*

3 Terms and definitions

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

3.1
electrical conductance
G
reciprocal value of the electrical resistance

NOTE The electrical conductance is expressed in siemens (S) or Ω^{-1}.

3.2
electrical resistance
R
measurement value of a sample, directly determined by means of the conductivity meter, which increases with the sample length *l* and decreases with the sample cross-section *A*

NOTE The electrical resistance of a sample is expressed in ohms (Ω). The relation between *R* and the electrical conductance (3.1) is shown in equation (1).

$$G = \frac{1}{R} \tag{1}$$

3.3
electrical conductivity
σ_T
material-dependent measure of the electrical current conducted in an electrical field by the ions present in the solution

NOTE 1 The material-dependent electrical conductivity of a solution is related to the electrical current which is achieved by moving ions of the solution in an electrical field. It is therefore applicable as a method to limit ionic contamination in alcohols and alcohol based fuels. The electrical conductivity σ_T is determined in accordance with equation (2) from the determined electrical conductance *G* (3.1) and the geometrical dimensions of the measuring cell (length and effective cross-section) which are added to form the cell constant *K* (3.4).

$$\sigma_{\mathrm{T}} = G \cdot \frac{l}{A} = G \cdot K \qquad (2)$$

where

σ_{T} is the electrical conductivity of the sample, in µS cm^{-1} (1 S m^{-1} = 10^{4} µS cm^{-1}), at the measurement temperature *T*, in °C;

l is the length of the measured section (geometrical electrode gap), in m or, following the respective unit conversion, in cm, with 1 m = 100 cm;

A is the effective cross-section of the measuring cell, in m^{2} or, following the respective unit conversion, in cm^{2}, with 1 m^{2} = 10^{4} cm^{2};

K is the cell constant of the measuring cell in m^{-1} or, following the respective unit conversion, in cm^{-1}, with 1 m^{-1} = 0,01 cm^{-1}.

NOTE 2 The electrical conductivity of a solution depends on the temperature; therefore the temperature is given together with the measurement value. Hence the electrical conductivity, which is mostly determined at 25 °C, is designated as σ_{25}.

NOTE 3 The electrical conductivity depends on ion concentration, ion type, temperature and viscosity of the solution. Therefore its measurement range can be large. "Ultrapure" (deionized and demineralised) water for example, due to its self-dissociation at 25 °C, has an electrical conductivity of 0,054 83 µS cm^{-1} (5,483 µS m^{-1}).

3.4
cell constant

K

the geometrical dimensions of the measuring cell which are added to form one value

NOTE The electrical conductivity σ (3.3) cannot be calculated simply from the electrical resistance *R* (3.2) and the geometrical cell dimensions because its functional relationship to the cell dimensions is very complex, especially in cases where the geometry is not cubical. Therefore, the measuring cell is calibrated by means of a calibration solution of known electrical conductivity σ^{*}. The cell constant *K* is determined in accordance with equation (3) from the determined electrical conductance *G** caused by the calibration solution and its known electrical conductivity σ^{*}.

$$K = \frac{\sigma_{25}^{*}}{G_{25}^{*}} \qquad (3)$$

where

K is the cell constant of the measuring cell in m^{-1} or, following the respective unit conversion, in cm^{-1} (1 m^{-1} = 0,01 cm^{-1});

G_{25}^{*} is the electrical conductance at 25 °C in the measuring cell filled with the calibration solution, expressed in S;

σ_{25}^{*} is the electrical conductivity of the calibration solution at 25 °C, in S m^{-1} or µS cm^{-1} (1 S m^{-1} = 10^{4} µS cm^{-1}).

4 Principle

The determination of the electrical conductivity in ethanol or ethanol fuel is carried out by means of a direct conductometer (6.1) measurement, using a measuring cell (6.2) suitable for a measurement range of approximately (0,01 to 5) µS/cm. The measurement is carried out at a sample temperature of (25 ± 0,1) °C. A selection of another measurement temperature and the subsequent conversion of the electrical conductance to a target temperature of 25 °C is not allowed.

5 Reagents

5.1 Ethanol, absolute, of analytical grade or a similar quality, for cleaning and rinsing the measuring cell (6.2), the sample vessel (6.4) and all the other vessels/containers with which the sample can come into contact.

5.2 Calibration solution, KCl solution having a conductivity σ_{25} of max.100 µS cm^{-1}.

5.3 Water, deionized or distilled with a maximum conductivity of 1 µS cm^{-1}, for cleaning and rinsing the sample vessel (6.4) and the measuring cell (6.2).

6 Apparatus

6.1 Conductometer, suitable of being operated with measuring cells (6.2) having a cell constant of approximately (0,1 to 1) cm^{-1}, with an alternating voltage of a frequency between 50 Hz and 500 Hz in the measuring cell (6.2).

The conductometer measures the resulting current in the solution to be measured for the purpose of determining the electrical conductance. A single-point calibration using a single KCl calibration solution (5.2) is sufficient.

6.2 Measuring cell, manufactured from a material which does not react chemically with ethanol or ethanol fuel. This can be a high-grade steel measuring cell, for example.

The cell constant should be in the range (0,1 and 1) cm^{-1}, preferably close to 0,1 cm^{-1}. The measuring cell is introduced from above into the sample vessel (6.4), through a cover, and fixed in a way it is completely immersed in the solution to be measured.

6.3 Temperature sensor, suitable for recording the temperature of the solution subjected to measurement at approximately 25 °C with an accuracy of ± 0,1 °C or better.

This sensor can also be integrated in the measuring cell (6.2). The temperature signal will be indicated on a display to the nearest ± 0,1 °C.

6.4 Thermostatable glass sample vessel, having a capacity of 20 ml to 500 ml, into which the sample to be measured is filled.

The sample vessel may be designed as a double-walled glass vessel, for example, which is thermostated by means of a circulating liquid. Alternatively, a thermostatable flow-through vessel may be used, provided it is ensured that the substance to be measured can be maintained at a temperature of (25 ± 0,1) °C, the measuring cell (6.2) is completely immersed, and all requirements for cleaning can also be complied with.

6.5 Thermostat with thermometer, suitable for controlling the temperature to (25 ± 0,1) °C for the purpose of thermostating the sample vessel (6.4).

6.6 Stirrer for ensuring a homogeneous temperature in the sample by stirring it with a magnetic stirrer and a teflon-coated stirring magnet.

7 Sampling

Unless specified otherwise, samples shall be taken in accordance with EN ISO 3170 or EN ISO 3171 and/or the requirements of national standards regarding the sampling of fuels.

Only glass bottles shall be used for samples. The glass bottles shall be cleaned very thoroughly and rinsed at least twice using the product to be sampled prior to the actual sampling.

For any other handling of the samples particular attention shall be paid to avoid any risk of further contamination.

8 Procedure

8.1 Preparation of the measuring device

8.1.1 General

The device manufacturer's instructions shall be followed for the preparation and operation of the conductometer (6.1) and the measuring cell (6.2). For adjusting the measurement temperature, the thermostat (6.5) is and connected with the sample vessel (6.4) and set to achieve a temperature of (25 ± 0,1) °C in the sample vessel.

8.1.2 Cleaning of the sample vessel

Before the measurement, the sample vessel (6.4) is rinsed several times with ethanol (5.1). The rest of the ethanol used for rinsing shall be dripped off. If the sample vessel (6.4) had been used before for saline samples, it shall additionally be cleaned thoroughly with water (5.3) prior to using it for ethanol or ethanol fuel. Subsequently, it is dried in the drying oven at min. 80 °C. Afterwards it is rinsed once more using ethanol (5.1). If there are any visible contaminations (e.g. stains) at the surface of the glass on the inside of the sample vessel (6.4), the vessel shall not be used.

8.1.3 Cleaning of the measuring cell

The measuring cell (6.2) is also cleaned by rinsing it thoroughly with ethanol (5.1). If the measuring cell (6.2) had been used before for saline samples, it shall be cleaned thoroughly with water (5.3) prior to using it for ethanol or ethanol fuel. Subsequently, it is thoroughly rinsed with ethanol (5.1). If there are any visible contaminations (e.g. stains) at the surface of the measuring cell (6.2), the cell shall not be used.

8.1.4 Filling the sample into the sample vessel

The ethanol or ethanol fuel, respectively, is filled into the sample vessel (6.4) by pouring it directly from the sample container into the vessel. Should an intermediate vessel be required for transferring, this vessel shall first be cleaned thoroughly using water (5.3), and dried in the drying oven at min. 80 °C. Before the intermediate vessel is used, it is rinsed with ethanol (5.1). Subsequently it shall be dripped off. Rubbing its inside with a fibrous cloth shall be avoided. The size of the intermediate vessel shall be chosen such that it is filled up to at least half its height. If there are any visible contaminations (e.g. stains) at the surface of the glass on the inside of the vessel, the vessel shall not be used.

8.2 Calibration

8.2.1 Interferences

The measurement values of the electrical conductivity can very strongly be influenced by contaminations in the sample as well as by pollutions of the measuring cell (6.2) (suspended matter, grease or oil). Such interferences cannot easily be detected in routine operation; therefore, the cell constant should regularly be checked by means of a reference solution or the calibration solution (5.2).

8.2.2 Checking the cell constant

The cell constant is determined at (25 ± 0,1) °C using the calibration solution (5.2), and stored in the device and recorded. In order to enable a fast measurement, the calibration solution (5.2) should be pre-adjust to a temperature of 25 °C. The measuring cell (6.2) and the sample vessel (6.4) are cleaned of any adhering rinsing ethanol (5.1). To do so, the sample vessel (6.4) is filled with the calibration solution (5.2) and, after the measuring cell (6.2) has been immersed in the solution, the solution is stirred for at least one minute.

Subsequently, the sample vessel (6.4) is emptied und refilled with the calibration solution (5.2). The measuring cell (6.2) is immersed in the solution, and the lid is closed. In order to achieve a homogeneous temperature distribution, the solution is gently stirred. As soon as a constant temperature of (25 ± 0,1) °C has been achieved in the solution, the calibration measurement can be started. The determined cell constant is stored in the device in accordance with the manufacturer's instructions and recorded rounded to the nearest 0,001 cm^{-1}.

Following the calibration of the conductometer (6.1) the first measurement using the calibration solution (5.2) is carried out. The sample vessel (6.4) is emptied and refilled with the calibration solution (5.2) without another intermediate cleaning step. The measurement result shall not deviate by more than 2 % relative from the conductivity σ_{25}^{*} of the calibration solution (5.2).

8.3 Measurement procedure

After cleaning the sample vessel (6.4) and the measuring cell (6.2) in accordance with 8.1.2 and 8.1.3, the sample to be measured is filled into the sample vessel (6.4) in accordance with the specifications of 8.1.4, the measuring cell (6.2) is immersed, the lid is closed, and the solution is stirred for at least one minute.

Once a constant temperature of (25 ± 0,1) °C has been obtained in the solution, the measured value is recorded.

9 Expression of results

Report the electrical conductivity of the sample, expressed in µS/cm, rounded to the nearest 0,01 µS/cm.

Report the measurement temperature, expressed in °C, rounded to the nearest 0,1 °C.

10 Precision

10.1 General

The precision statements have been developed in a round robin study according to EN ISO 4259 [2].

10.2 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 value given in Table 1 in only one case in twenty.

10.3 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 value given in Table 1 only in one case in twenty.