SLOVENSKI STANDARD
SIST EN 12177:1999

01-november-1999

Tekoļ i naftni proizvodi - Neosvinļ eni motorni bencini ï Doloļ evanje benzena s plinsko kromatografijo

Liquid petroleum products - Unleaded petrol - Determination of benzene content by gas chromatography

Flüssige Mineralölerzeugnisse - Unverbleite Ottokraftstoffe - Bestimmung des Benzolgehaltes mittels Gaschromatographie

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Produits pétroliers liquides - Essence sans plomb - Détermination de la teneur en benzene par chromatographie en phase gazeuse

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Ta slovenski standard je istoveten z: **EN 12177:1998**

ICS:

75.160.20	Tekoļ a goriva	Liquid fuels

SIST EN 12177:1999 **en**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 12177

April 1998

ICS 75.160.20

Descriptors: petroleum products, gasoline, chemical analysis, determination of content, benzene, chromatographic analysis, gas chromatography

English version

Liquid petroleum products - Unleaded petrol - Determination of benzene content by gas chromatography

Produits pétroliers liquides - Essence sans plomb - Détermination de la teneur en benzène par chromatographie en phase gazeuse

Flüssige Mineralölerzeugnisse - Unverbleite Ottokraftstoffe - Bestimmung des Benzolgehaltes mittels Gaschromatographie

This European Standard was approved by CEN on 23 March 1998.

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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 Central Secretariat has the same status as the official versions.

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

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Ref. No. EN 12177:1998 E

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NNI.

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 October 1998, and conflicting national standards shall be withdrawn at the latest by October 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

In this standard annex A is informative.

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1 Scope

This European standard specifies a column switching gas chromatographic method for the quantitative determination of benzene content in the range 0,05 % (*V/V*) to 6 % (*V/V*) in unleaded petrol having a final boiling point not greater than 220 °C.

The method described in this standard is suitable for determining benzene in petrol, including petrol containing oxygenates, in line with the relevant EC Directives[1)].

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

WARNING : The 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

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

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EN ISO 3675	Crude petroleum and liquid petroleum products - Laboratory determination of density or relative density - Hydrometer method (ISO 3675:1993)
EN ISO 3838	Crude petroleum and liquid or solid petroleum products - Determination of density or relative density - Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods (ISO 3838:1983)
EN ISO 12185	Crude petroleum and petroleum products - Determination of density - Oscillating U-tube method (ISO 12185:1996)
ISO 3170	Petroleum liquids - Manual sampling
ISO 3171	Petroleum liquids - Automatic pipeline sampling

3 Principle

The benzene-containing fraction is isolated from the sample using a capillary column and, in a second capillary column, the benzene is separated and detected using a flame ionization detector.

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1) EC Directive 85/210/EEC, Council Directive on the approximation of the laws of the Member States concerning the lead content of petrol. EC Directive 85/536/EEC, Council Directive on crude-oil savings through the use of substitute fuel components in petrol.

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NOTE 1 : Some oxygenates are known to interfere with the determination of benzene using a single column gas chromatographic method.

NOTE 2 : Guidance on the column switching technique is given in annex A.

4 Reagents and materials

Use only reagents of recognized analytical grade.

4.1 Carrier gas

Hydrogen, helium, or nitrogen, free of hydrocarbons.

WARNING : Hydrogen is explosive in mixtures with air at concentrations of hydrogen ranging approximately from 4 % (*V/V*) to 75 % (*V/V*). All joints and lines carrying hydrogen shall be made gas tight to prevent leakage of hydrogen into a confined space.

4.2 Reagents for the preparation of calibration samples

NOTE: Calibration samples will normally be combinations of benzene, solvent and an internal standard.

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4.2.1 Benzene

Not less than 99,0 % (*m/m*) pure.

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WARNING : Benzene is toxic and carcinogenic.

4.2.2 Solvent

Not containing benzene or internal standard, e.g. heptane.

4.3 Internal standard

Reagent for which it has been established that it is not present in the sample.

NOTE : Isobutyl methyl ketone is preferred as the internal standard.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Gas chromatographic assembly

5.1.1 **Gas chromatograph**, provided with a means for column switching, equipped with a programmable oven temperature controller, or controllers in the case of dual oven gas chromatographs, and two flame ionization detectors (FID).

NOTE : It is recommended that a system constructed entirely of glass from the sample injection port up to the detector system is used since petrol may contain oxygenates which can give rise to corrosion and changes of retention times in systems constructed partly using metals.

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5.1.2 **Two capillary columns**, of appropriate dimensions, each internally coated with a substance of different polarity, such that the resolution between the benzene peak and the matrix is at least 1 after elution from the second column.
The resolution, R, between peaks A and B (see figure 1) is calculated using the equation:

$$R = 1{,}18 \frac{t'_B - t'_A}{w_A + w_B}$$

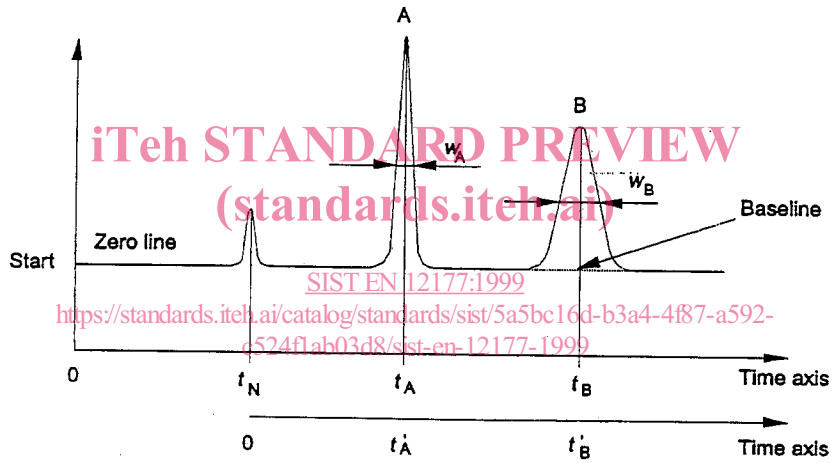
where :

t'_A is the reduced retention time of component A;

t'_B is the reduced retention time of component B;

w_A is the peak width at half-height of component A;

w_B is the peak width at half-height of component B.



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NOTE : t_N is the hold-up time zero of the column, i.e. the time taken for an inert component, such as methane, to travel through the column without chromatography taking place.

Figure 1 - Calculation of the resolution between peaks A and B

5.1.3 **Device for the control of the flow of carrier gas.**

5.1.4 **Recorder and/or integrator**

An amplifier and potentiometric recording device, or an integrator or data processor system, giving area values corresponding to the peak area in square millimetres.

5.2 **Injection device**

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5.3 **Test sample container**, normally with a capacity of between 10 ml and 100 ml, fitted with a self-sealing rubber septum coated with polytetrafluoroethylene (PTFE).

6 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in ISO 3170 or ISO 3171, and/or in accordance with the requirements of national standards or regulations for the sampling of petrol.

7 Procedure

7.1 Setting up the apparatus

7.1.1 General

Prepare the equipment and set the test conditions in accordance with the manufacturer's instructions.

7.1.2 Carrier gas

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Adjust the pressure and flow rate of the carrier gas to levels such that the resolution is in accordance with 5.1.2.

7.2 Calibration

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Prepare the calibration sample by combining known masses of benzene (4.2.1) and internal standard (4.3) in a suitable solvent (4.2.2).

Inject a suitable quantity of the prepared calibration sample into the gas chromatograph such that the capacity of the columns and other components is not exceeded and the linearity of the detector is not impaired.

Determine and record the retention times for benzene and for the internal standard. Calculate the calibration factor, f, for benzene using the following equation:

$$f = \frac{m_1 \times A_2}{A_1 \times m_2}$$

where :

m_1 is the mass of benzene in the calibration sample, in grams;

A_2 is the peak area of the internal standard, in square millimetres;

A_1 is the peak area of benzene, in square millimetres;

m_2 is the mass of the internal standard in the calibration sample, in grams.

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7.3 Determination of density

Determine the density at 15 °C, ρ_s , of the sample in accordance with either EN ISO 3675, EN ISO 3838 or EN ISO 12185 and record the result to the nearest 0,1 kg/m³.

7.4 Preparation of test sample

Cool the sample to between 5 °C and 10 °C. Weigh, to the nearest 0,1 mg, the test sample container (5.3) and its rubber septum without sealing it.

Transfer a quantity of the internal standard (4.3) to the test sample container and weigh, to the nearest 0,1 mg, the test sample container with contents and septum, without sealing the sample container. The mass, m_{st}, in grams, of the internal standard shall amount to between 2 % (*m/m*) and 5 % (*m/m*) of the sample, m_s, but shall not be less than 0,050 g.

Transfer a quantity, normally between 5 ml and 100 ml, of the cooled sample to the test sample container and seal immediately with the septum. Weigh, to the nearest 0,1 mg, the test sample container and contents. Record the mass, m_s, in grams, of the test sample taken, to the nearest 0,1 mg.

Record the amount of internal standard in the prepared test sample as a percentage by mass. Mix the contents of the test sample container by shaking until homogeneous.

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7.5 Introduction of test portion

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Inject a suitable quantity of the prepared test sample (7.4) into the gas chromatograph. Ensure that the test portion size is such that the capacity of the columns and other components of the gas chromatograph is not exceeded and that the linearity of the detector is not impaired.

7.6 Examination of the gas chromatogram

Examine the gas chromatogram and identify the benzene and the internal standard by means of their retention times (see 7.2).

8 Calculation

8.1 Calculation of mass of benzene in the test sample

Calculate the mass, m_3, in grams, of benzene in the test sample using the following equation:

$$m_3 = \frac{f \times A_3 \times m_4}{A_4}$$

where :

f is the calibration factor for benzene;

A_3 is the peak area of benzene, in square millimetres;