# SLOVENSKI STANDARD
# SIST EN 238:1998

**01-maj-1998**

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**Tekoči naftni proizvodi - Bencin - Določevanje benzena z infrardečo spektrometrijo**

Liquid petroleum products - Petrol - Determination of the benzene content by infrared spectrometry

Flüssige Mineralölerzeugnisse - Ottokraftstoff - Bestimmung des Benzolgehaltes durch Infrarotspektrometrie



Produits pétroliers liquides - Essence - Détermination de la teneur en benzene par spectrométrie infrarouge



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English version

# Liquid petroleum products - Petrol - Determination of the benzene content by infrared spectrometry

Produits pétroliers liquides - Essence - Détermination de la teneur en benzène par spectrométrie infrarouge

Flüssige Mineralölerzeugnisse - Ottokraftstoff - Bestimmung des Benzolgehaltes durch Infrarotspektrometrie



This European Standard was approved by CEN on 1995-10-06. 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 Central Secretariat or to any CEN member.

The European Standards exist 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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**CEN**

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

**Central Secretariat: rue de Stassart,36 B-1050 Brussels**

**Foreword**

This European Standard has been prepared by the 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of an identical text or by endorsement, at the latest by July 1996, and conflicting national standards shall be withdrawn at the latest by July 1996.

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

This European Standard is based on DIN 51 414: "Testing of petroleum products; determination of the benzene content of gasolines; determination by infrared spectrometry" (June 1985) in connection with DIN 51 451: "Testing of petroleum products and related products; analysis by infrared spectrometry; general working principles" (September 1988).



In this standard annex A is normative.

# 1 Scope

This European Standard specifies an infrared spectrometric method for the determination of benzene content in petrol in the range 0,1 % (*V/V*) to 20 % (*V/V*). The presence of cyclopentadiene in the sample will interfere with the benzene determination when it exceeds 5 % (*V/V*).

Contents of ethanol less than 10 % (*V/V*) and toluene less than 25 % (*V/V*) do not interfere with the benzene determination.

**WARNING :** The use of this European 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 European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.



| | |
|---|---|
| ISO 595-1 | Reusable all-glass or metal-and-glass syringes for medical use - Part 1: Dimensions |
| ISO 595-2 | Reusable all-glass or metal-and-glass syringes for medical use - Part 2: Design, performance requirements and tests |
| ISO 648 | Laboratory glassware - One-mark pipettes |
| ISO 1042 | Laboratory glassware - One-mark volumetric flasks |
| ISO 3170 | Petroleum liquids - Manual sampling |
| ISO 3171 | Petroleum liquids - Automatic pipeline sampling |

# 3 Principle

After dilution of the sample with cyclohexane an infrared spectrum is recorded from 730 $cm^{-1}$ to 630 $cm^{-1}$. The absorbance is measured at 673 $cm^{-1}$ and compared with the absorbance of standard benzene solutions. The benzene content is then given in g/100 ml and calculated as a percentage by volume or by mass.

# 4 Reagents and materials

Use only reagents of recognized analytical grade.

**WARNING :** The reagents are toxic. Special precautions shall be taken when handling them to avoid inhaling the vapour. If some liquid is accidentally spilled on the skin the affected part shall be washed immediately with soap and water.

**4.1 Benzene**, minimum purity 99,9 % (*m/m*).

**4.2 Benzene standard stock solution**, 1 g/100 ml.

Weigh to the nearest 0,1 mg, approximately 1 g of benzene (4.1) into a volumetric flask (5.5). Dilute to the mark with cyclohexane (4.4) and shake.

**4.3 Benzene standard stock solution**, 3 g/100 ml.

Weigh to the nearest 0,1 mg, approximately 3 g of benzene (4.1) into a volumetric flask (5.5). Dilute to the mark with cyclohexane (4.4) and shake.

**4.4 Cyclohexane**, giving no absorbance at approximately 673 $cm^{-1}$.

**4.5 Solvent**, for cleaning purposes, giving no absorbance at approximately 673 $cm^{-1}$.

NOTE : 1,1,1-Trichloroethane has been found suitable.



## 5 Apparatus



Usual laboratory apparatus and glassware, together with the following:

**5.1 Infrared spectrometer**, either a double beam or fourier-transform (FTIR) type, enabling a minimum slit width of 2 $cm^{-1}$ and a recording speed of 60 $cm^{-1}$/min.

**5.2 Cells** (two), one for test solutions and one for the reference solution. The cells shall have windows made from potassium bromide and shall provide accurately known path lengths.

NOTE : With sample dilutions of 1:50 to 1:100 and a benzene content of 2 % (*V/V*) the optimal path length is approximately 0,50 mm.

**5.3 Glass syringes**, of the appropriate capacities (see ISO 595-1) and conforming to ISO 595-2.

**5.4 One-mark pipettes**, conforming to class B of ISO 648.

**5.5 One-mark volumetric flasks**, capacity 100 ml, conforming to class B of ISO 1042.

## 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 General

Measure all volumes at a temperature within 5 °C of the temperature at which the volumetric glassware was calibrated.

NOTE : This temperature is usually 20 °C.

If results are required as a percentage by mass and the density of the sample is unknown, it shall be determined in kilograms per cubic metre.

## 7.2 Preparation of the calibration graph

### 7.2.1 Preparation of a set of calibration solutions

Prepare a set of calibration solutions with benzene contents of 0,01 g/100 ml, 0,02 g/100 ml, 0,04 g/100 ml, 0,06 g/100 ml, 0,10 g/100 ml, 0,12 g/100 ml and 0,15 g/100 ml as follows.

Introduce by means of syringes (5.3) or pipettes (5.4) the following volumes of the benzene standard stock solutions into a series of volumetric flasks (5.5): 1,0 ml, 2,0 ml, 4,0 ml and 10,0 ml of the 1 g/100 ml standard stock solution (4.2) and 2,0 ml, 4,0 ml and 5,0 ml of the 3 g/100 ml standard stock solution (4.3). Dilute to the mark with cyclohexane (4.4) and shake.



### 7.2.2 Spectrometric measurements



Introduce the calibration solutions successively into the clean and dry sample cell (5.2) and cyclohexane (4.4) into the clean and dry reference cell (5.2). Record the infrared spectrum of each of the calibration solutions against cyclohexane from 730 $cm^{-1}$ to 630 $cm^{-1}$.

Apply the procedure for the base line correction given in annex A, taking 710 $cm^{-1}$ and 640 $cm^{-1}$ as base line end points. Determine the absorbances of the calibration solutions at 673 $cm^{-1}$.

NOTE : If the benzene contents are lower than 1 g/100 ml, it is advised to shift the base line end point of 640 $cm^{-1}$ as necessary to provide the best tangent as the base line.

### 7.2.3 Plotting the graph

Plot a graph having the concentrations, in g/100 ml, of benzene of the calibration solutions as abscissae and the corresponding base line corrected absorbances as ordinates. Determine the calibration line using linear regression. This calibration line is given by equation 1 (see clause 8).

Use the calibration graph only for measurements where the same test and reference cells are used as for measuring the calibration solutions themselves.

Repeat the calibration when the correlation coefficient given by the linear regression analysis of the calibration graph is less than 0,999. Check the calibration graph regularly.

### 7.3 Preparation of the test solution

Ensure that during the preparation of the test solution no losses occur due to evaporation.

Dilute the sample with cyclohexane (4.4) at a temperature of 20 °C. Apply dilution factors as given in table 1 in order to achieve a test solution with a benzene content falling in the range of the calibration graph.

**Table 1 : Dilution factors for preparation of the test solution**

| Estimated benzene content, $x$, of the sample % (V/V) | Dilution factor |
|---|---|
| $x < 1$ | 1 : 25 |
| $1 \leq x < 2$ | 1 : 50 |
| $2 \leq x$ | 1 : 100 |

### 7.4 Determination



Measure the absorbance of the test solution as specified in 7.2.2 for the calibration solutions. Ensure that the test solution is placed in the sample cell (see 7.2.2).



## 8 Calculation

**8.1** Determine the benzene content of the test solution, $B$, in g/100 ml, either by reading the concentration corresponding to the absorbance of the test solution from the calibration line (7.2.3), or by calculation using the following equation:

$$B = \frac{(A - b)}{m} \quad \ldots (1)$$

where :

$A$ is the absorbance of the test solution;

$b$ is the ordinate intercept of the calibration graph;

$m$ is the slope of the calibration graph.

**8.2** Calculate the benzene content of the sample, $B_v$, expressed as a percentage by volume, using the equation:

$$B_v = \frac{B \times f}{\rho b} \times 1000 \quad \ldots (2)$$

where :

$B$ is the benzene content of the test solution, in g/100 ml;

$f$ is the dilution factor used for the preparation of the test solution (e.g. with dilution factor 1:50 (see table 1), $f$ will be 50);

$\rho b$ is the density of benzene at 20 °C (878,6 kg/m$^3$).

**8.3** If results are required as a percentage by mass, calculate the content of the benzene, $B_M$, using the equation:

$$B_M = \frac{B_v \times \rho b}{\rho s} \quad \ldots (3)$$

where :

$\rho s$ is the density of the sample at 20 °C, in kilograms per cubic metre.

## 9 Expression of results



Report the benzene content of the sample to the nearest 0,1 % (*V/V*).



If required, report the benzene content of the sample to the nearest 0,1 % (*m/m*).

## 10 Precision

### 10.1 Repeatability

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 following values only in one case in twenty: 0,1 % (*V/V*) or 0,1 % (*m/m*).

### 10.2 Reproducibility

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 following values only in one case in twenty: 0,3 % (*V/V*) or 0,3 % (*m/m*).

## 11 Test report

The test report shall contain at least the following information:

a) the type and identification of the product under test;

b) a reference to this European Standard;