International Standard 5279

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Toluene — Determination of hydrocarbon impurities — Gas chromatographic method

Toluène — Détermination des impuretés hydrocarbonées — Méthode par chromatographie en phase gazeuse

First edition — 1980-10-01

UDC 547.533 : 543.544 : 543.869 **Ref. No. ISO 5279-1980 (E)**

Descriptors : aromatic hydrocarbons, toluene, chemical analysis, determination of content, hydrocarbons, impurities, gas chromatographic analysis.

Price based on 4 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5279 was developed by Technical Committee ISO/TC 78, *Aromatic hydrocarbons*, and was circulated to the member bodies in July 1979.

It has been approved by the member bodies of the following countries :

- Australia
- Austria
- Chile
- Czechoslovakia
- Egypt, Arab Rep. of
- France
- Germany, F. R.
- Hungary
- India
- Italy
- Korea, Rep. of
- Libyan Arab Jamahiriya
- Mexico
- Netherlands
- Poland
- Romania
- South Africa, Rep. of
- United Kingdom
- USSR

No member body expressed disapproval of the document.

Printed in Switzerland

Toluene — Determination of hydrocarbon impurities — Gas chromatographic method

1 Scope and field of application

1.1 This International Standard specifies a gas chromatographic method for the quantitative determination of hydrocarbon impurities in commercial toluene. It is applicable to the determination of benzene, C_8 aromatics and non-aromatics up to *n*-nonane, in the range 0,01 to 1,00 % (m/m).

1.2 The materials and procedures described in this International Standard are satisfactory for the analysis, but alternatives that will give equivalent results may also be used. They must, however, give complete separation of the group of non-aromatics up to C_9, benzene, *n*-decane, toluene and the group of C_8 aromatics.

2 Reference

ISO 1995, *Aromatic hydrocarbons — Sampling*[1].

3 Principle

Addition of a known amount of internal standard to a test portion and introduction of an aliquot portion of this mixture into the separating column of a gas chromatograph by means of a syringe. Sweeping of the vaporized mixture through the column by a flow of carrier gas, detection of each component as it emerges by a flame ionization detector and recording as a peak on a chromatogram.

Identification of the impurities by their relative retention times and quantitative determination from their peak areas relative to the peak area of the internal standard.

The relative response of the detector to the various components is taken into consideration in the calculation.

4 Materials

NOTE — Materials required for preparation of the chromatography column are specified in 5.3.1.

4.1 Carrier gas, either helium or hydrogen, of low oxygen content [preferably 0,000 5 % (V/V) max.].

CAUTION — Hydrogen forms highly explosive mixtures with air. Its use should always be accompanied by appropriate precautions to ensure that leaks, etc., are not allowed to cause build up of dangerous concentrations of the gas.

4.2 Air supply.

4.3 Hydrogen supply.

4.4 *n*-Hexane, gas chromatography standard, free from components coeluting with benzene, *n*-decane, and ethyl benzene.

4.5 Internal standard, *n*-decane, purity 99 % (m/m) minimum.

4.6 Calibration materials, all having a purity of at least 99 % (m/m).

4.6.1 Benzene.

4.6.2 Ethylbenzene.

5 Apparatus

Usual laboratory equipment and in particular

5.1 Chromatograph.

Any gas chromatograph fitted with a flame ionization detector and capable of meeting the required operating conditions may be used. The instrument shall have sufficient sensitivity to obtain a peak height when using a mixture containing 0,005 % (m/m) ethylbenzene, under the specified operating conditions, of at least twice the noise level.

---

1) At present at the stage of draft.

5.2 Injection device.

On-column injection is recommended to obtain maximum resolution. If an inlet splitter is fitted to the chromatograph, poor resolution may result from blocking at the vent.

5.3 Column, made of stainless steel, copper, aluminium, or glass tubing, having a length of 4 m and a nominal internal diameter of 2 mm.

5.3.1 Materials (see also 1.2)

5.3.1.1 Packing.

5.3.1.2 Pink firebrick (the support), acid washed, particle size 180 to 250 µm.

5.3.1.3 Polyethylene glycol 1540 (stationary phase).

Dissolve 12,5 g of polyethylene glycol 1540 in 50 ml of a suitable solvent (for example methanol). To this solution, add 37,5 g of the firebrick (5.3.1.1), stir, and evaporate the solvent whilst stirring.

Resieving the coated material will generally improve the efficiency of the resulting column and is strongly recommended.

5.3.2 Preparation of the column

Plug one end of the tube with porous metal or glass wool and apply suction to this end. Pour the prepared packing into the tube through a funnel, continuously tapping or vibrating the tube to ensure even distribution. When the tube is full (see the note) tip out a small amount of packing and plug the open end of the tube. For on-column injection systems, a short length of column may need to be left unpacked at the inlet end, and porous metal plugs should be avoided.

NOTE — Columns may be packed before or after coiling. About 9 g of packing is normally required to fill the column. Differently packed columns, or commercially available prepacked columns, may be equally suitable.

5.3.3 Column performance

5.3.3.1 The column shall be capable of completely resolving the internal standard from all other peaks in the chromatogram when operated under the specified conditions.

NOTE — If necessary, resolution of the internal standard from other components may be achieved by adjusting the column temperature by up to 20 °C either above or below the recommended temperature of 100 °C.

5.3.3.2 Test the performance of the column by preparing a mixture of 0,25 % (*V*/*V*) ethylbenzene in toluene. Inject a sample of the mixture, of the same size as used in the procedure (see clause 7), into the chromatograph and record the chromatogram. The height of the valley between the two peaks measured from the base-line shall be no more than 2 % of the ethylbenzene peak if integration is to be used, and no more than 10 % if measurement is to be made by hand.

The base-line is defined as the line the recorder pen would have traced, had the peaks not been present.

5.4 Integrator.

Use of an integrator is recommended. Electronic integrators should have a count rate of at least 10 000 counts/min (full scale) and a linearity of at least 0,5 % of the full-scale counts. Other methods of recording and measuring peak areas such as computer systems may be used, provided that the sensitivity requirements are met.

5.5 Strip chart recorder, with a scale of at least 200 mm and a response time of not more than 1 s for full-scale deflection.

6 Sampling

Take a representative sample of the product to be tested in accordance with ISO 1995.

7 Procedure

7.1 Experimental conditions

The experimental conditions are set out in table 1.

Table 1 — Experimental conditions

Column temperature	100 °C (see note to 5.3.3.1).
Carrier gas flow-rate	Adjusted to give a retention time for toluene of between 10 and 15 min.
Fuel gases	Hydrogen and air flow-rates for the flame ionization detector should be adjusted to the levels recommended by the instrument manufacturers.
Size of test portion	1 µl is recommended, but larger test portions may be used provided that satisfactory resolution is maintained.
Chart speed	Not less than 20 mm/min if peak areas are measured manually.

7.2 Calibration

7.2.1 Prepare a calibration mixture of *n*-decane (4.5), benzene (4.6.1) and ethylbenzene (4.6.2) in *n*-hexane (4.4) according to the following procedure.

Place 10 ml of the *n*-hexane in a clean, dry, stoppered 25 ml volumetric flask. Using a syringe, add 50 µl of each of the calibration materials, taking care to ensure each component is taken into the hexane phase before the next addition, and make up to 25 ml with *n*-hexane.

7.2.2 Inject the mixture into the chromatograph under the conditions specified in table 1 and record the chromatogram.

7.2.3 Calculate the relative response factors from the formula

$$F_i = \frac{A_i}{A_0} \times \frac{0{,}730}{\varrho_i}$$

where

F_i is the relative response factor;

A_i is the area of the peak for component *i*;

A_0 is the area of the *n*-decane peak;

ϱ_i is the density of component *i* (for benzene, ϱ = 0,879 g/ml; for ethylbenzene, ϱ = 0,867 g/ml).

A list of typical relative response factors is given in table 2.

7.2.4 If the relative response factors obtained by the above procedure differ from the values given in table 2 by more than 10 %, check them by means of a second calibration mixture. The results shall agree to within 10 % of the values given in table 2.

Table 2 — Typical relative response factors

Calibration material	Response factor
n-decane	1,00
benzene	1,17
ethylbenzene	1,02

7.3 Determination

7.3.1 Add a volume of the internal standard (4.5) to the test portion, in the volume ratio of 2 to 1 000, using minimum quantities of 20 µl and 10 ml respectively. Weigh both the test portion and internal standard in a flask to within ± 0,2 mg.

7.3.2 Use the experimental conditions given in table 1. Inject the test portion into the column and record the chromatogram.

7.3.3 Determine the peak areas of the impurities and internal standard, i.e. non-aromatics, benzene, *n*-decane and C_8 aromatics.

The peak areas may be determined either by integration or manually. Integrators or computers are recommended, particularly for measurement of the non-aromatic peaks. Tailing of the main component may preclude their use with components eluting after it under the conditions specified in table 1. Manual measurement of peak areas may be made either from peak height multiplied by peak width at half height for sufficiently resolved peaks, or by triangulation.

The components may be identified from the typical chromatogram given in the figure and from the relative retention times given in table 3.

Table 3 — Typical relative retention times from injection

Compound	Retention time
benzene	0,87
n-decane	1,00
toluene	1,50
ethylbenzene	2,33
m- and *p*-xylenes	2,42 to 2,48
o-xylene	3,18

8 Calculation

The concentrations of impurities, expressed as percentages by mass, are given by the formula

$$X_i = \frac{100 \times A_i \times m_1 \times F_1}{A_1 \times m_0 \times F_i}$$

where

X_i is the content, expressed as a percentage by mass, of substance *i* in the test portion;

A_i is the peak area for substance *i*;

A_1 is the peak area for the internal standard;

F_i is the relative response factor for substance *i*;

F_1 is the relative response factor for the internal standard;

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of internal standard used.

NOTE — All non-aromatic impurities have been assumed to have the same relative response factor as *n*-decane, and all C_8 aromatics the same as ethylbenzene.

9 Precision

9.1 Repeatability

The difference between the results obtained by one operator within a short time interval with the same apparatus under constant operating conditions on identical test material shall, at the 95 % confidence level, not exceed 10 % of the concentration being determined or 0,01 % (*m/m*), whichever is the greater.

9.2 Reproducibility

The difference between the results obtained by different operators in different laboratories on identical test material shall, at the 95 % confidence level, not exceed 20 % of the concentration being determined or 0,02 % (*m/m*), whichever is the greater.

10 Test report

The test report shall contain at least the following information :

a) the type and identification of the product tested;

b) the reference of this International Standard;

c) the results for benzene, the group of C_8 aromatics, and non-aromatics, as appropriate, to the nearest 0,01 % (*m*/*m*);

d) any deviation by agreement or otherwise, from the procedure specified;

e) the date of the test.

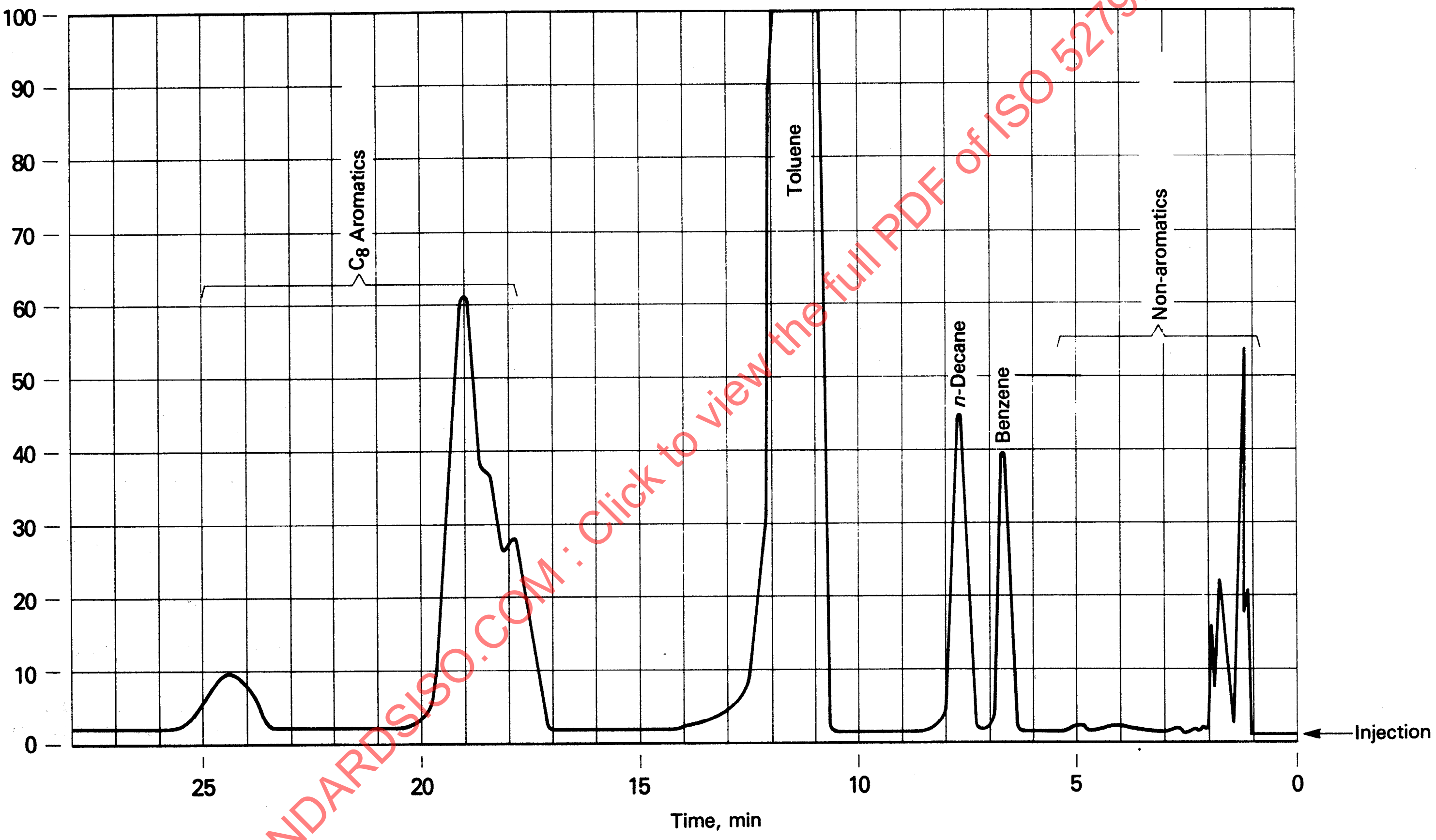

Figure — Typical chromatogram for toluene and hydrocarbon impurities

INTERNATIONAL STANDARD ISO 5279-1980 (E)/ERRATUM

Published 1981-12-01

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Toluene — Determination of hydrocarbon impurities — Gas chromatographic method

ERRATUM

Page 2

Sub-clause 5.3.1.3, third line : The reference to the firebrick should read "(5.3.1.2)".

This page intentionally left blank