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Benzyl chloride for industrial use — Methods of test

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FOREWORD

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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 3362 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in January 1974.

It has been approved by the Member Bodies of the following countries :

Belgium	India	Spain
Chile	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep. of	Netherlands	Turkey
France	New Zealand	United Kingdom
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Benzyl chloride for industrial use — Methods of test

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies methods of test for benzyl chloride for industrial use.

2 REFERENCES

ISO/R 758, *Method for the determination of density of liquids at 20 °C.*

ISO/R 1392, *Determination of crystallizing point — General method.*

ISO 2209, *Liquid halogenated hydrocarbons for industrial use — Sampling.*

ISO 2718, *Standard layout for a method of chemical analysis by gas chromatography.*

3 CHARACTERIZATION OF PRODUCT

3.1 WARNING. Benzyl chloride is a lachrymatory substance; suitable precautions shall therefore be taken during all operations.

3.2 Components

The main impurities are benzal chloride, benzaldehyde, and chlorotoluenes. Samples may also contain toluene, 2,4-dichlorotoluene and unknown components.

4 SAMPLING

For the preparation of the laboratory sample, follow the method specified in ISO 2209 but using only apparatus made of glass or nickel.

NOTE — Benzyl chloride decomposes when heated in the presence of even a small amount of iron.

5 DETERMINATION OF CRYSTALLIZING POINT

Determine the crystallizing point of the undried sample by the method specified in ISO/R 1392, using a thermometer certified for accuracy and complying with the following requirements :

- Graduated at 0,1 °C intervals
- Accuracy : 0,1 °C
- Range : − 50 °C to 0 °C or some other suitable range
- Filling : liquid eutectic alloy of mercury and thallium

6 DETERMINATION OF DENSITY

Use the method specified in ISO/R 758.

7 GAS CHROMATOGRAPHIC ANALYSIS

7.1 Principle

Determination of the benzyl chloride content and of the impurities benzal chloride, benzaldehyde, toluene, chlorotoluenes, etc., by gas chromatography (ISO 2718 should be considered).

Under the specified conditions, benzaldehyde may be eluted together with unidentified components.

7.2 Materials required

7.2.1 *Carrier gas*

Helium, dried before use by passing through a freshly regenerated molecular sieve.

7.2.2 *Reference materials*

7.2.2.1 Benzyl chloride

7.2.2.2 Benzal chloride

7.2.2.3 Benzaldehyde

7.2.2.4 *o*- and *p*-Chlorotoluenes

7.2.2.5 Toluene

7.2.2.6 2,4-Dichlorotoluene

7.3 Apparatus

7.3.1 *Type*

Any commercially available type of gas chromatograph fitted with a thermal conductivity detector (katharometer).

7.3.2 Injection device

Any heatable type with which the commercially available apparatus is provided.

7.3.3 Column

7.3.3.1 Tube, glass, length 2 m, internal diameter approximately 4,5 mm, form and external diameter optional.

7.3.3.2 Packing

7.3.3.2.1 Support, flux-calcined diatomaceous earth[1]; particle size 180 to 250 μm, acid-washed, surface area 1 to 3,5 m^2/g, pore volume 2,78 cm^3/g, density 2,20 g/cm^3, packed density 0,3 to 0,4 g/cm^3.

7.3.3.2.2 Stationary phase, consisting of a mixture of polymeric methylphenyl ethers of relative molar mass up to 15 000, hydrocarbons and orthophosphoric acid.[2]

The column packing comprises 20 % (*m/m*) of the organic components[2] of the stationary phase and about 2 % (*m/m*) of orthophosphoric acid on 78 % (*m/m*) approximately of the support (7.3.3.2.1), and is prepared as follows :

Dissolve 1 g of concentrated orthophosphoric acid (ρ approximately 1,7 g/ml) in 200 ml of methanol in a porcelain dish, add 40 g of the support (7.3.3.2.1) and stir well. Evaporate the methanol, in a fume cupboard, on a water bath at 80 °C, with continuous stirring.

Then dissolve 10 g of the organic component[2] in 200 ml of toluene in a porcelain dish, add the orthophosphoric acid-loaded support and stir well. Evaporate the toluene, in a fume cupboard, on a water bath at 95 °C, with continuous stirring.

7.3.3.2.3 Mass of packing introduced. Approximately 8 g of the packing is required to fill the tube (7.3.3.1).

7.3.4 Detector, consisting of a thermal conductivity cell (katharometer), with electrodes, made of platinum or other suitable material, fastened in glass.

7.3.5 Recorder, full scale deflection 1 to 2,5 mV, with or without a suitable integrator.

7.4 Procedure

7.4.1 *Control of the apparatus*

a) injector temperature : 160 °C;

b) column temperature : 130 ± 5 °C;

c) carrier gas flow rate : approximately 90 cm^3/min, inlet pressure approximately 170 kPa*;

d) detector temperature : 130 ± 5 °C;

e) recorder chart speed : 10 mm/min.

7.4.2 *Calibration*

Use the internal calibration method.

For the determination of impurities in benzyl chloride for industrial use, the coefficients of proportionality (K_i, see 7.5) can be approximated to unity.

7.4.3 *Test*

7.4.3.1 Inject 0,002 5 ml of the test sample by means of a syringe of capacity 0,010 ml.

7.4.3.2 Run the chromatogram under the specified conditions for about 25 min, using optimum attenuation of the recorder, until all the components have been eluted.

7.4.4 *Examination of the chromatogram*

7.4.4.1 STANDARD CHROMATOGRAM

Prepare a standard chromatogram from a test mixture containing the reference materials listed in 7.2.2, using the procedure specified in 7.4.3.

This chromatogram is for identification purposes only.

7.4.4.2 ORDER OF ELUTION OF THE COMPONENTS

The components elute in the order

1) toluene;

2) benzaldehyde;

NOTE — Unidentified components may be included in the benzaldehyde peak.

3) chlorotoluenes;

4) benzyl chloride;

5) benzal chloride;

6) 2,4-dichlorotoluene.

Elution is completed within about 25 min. Identify the peaks by comparison with the standard chromatogram (7.4.4.1).

7.4.4.3 PEAK MEASUREMENT

Evaluate the area of each peak by using an appropriate method, for example by reading the peak area from an integrator.

1) Type chromosorb W-AW, for example, meets the specified requirements.

2) A suitable mixture of the organic components is available commercially under the trade name "Apiezon L".

* 1 Pa = 1 N/m^2

7.5 Expression of results

The content of component i, expressed as a percentage by mass, is given by the formula

$$\frac{K_i \times A_i}{\Sigma (K_i \times A_i)} \times 100$$

where

A_i is the peak area for component i;

K_i is the coefficient of proportionality for component i.

NOTE — All K_i's can be equated to unity (see 7.4.2).

Express the content of benzyl chloride to the nearest 0,1 % (*m/m*) and the contents of the impurities to the nearest 0,01 % (*m/m*).

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.