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Carbon disulphide for industrial use — Sampling and methods of test

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FOREWORD

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

Austria	Hungary	Spain
Belgium	India	Switzerland
Bulgaria	Israel	Thailand
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This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

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Carbon disulphide for industrial use — Sampling and methods of test

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies methods of sampling and test for carbon disulphide for industrial use.

2 REFERENCES

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

ISO/R 918, *Test method for distillation (distillation yield and distillation range)*.

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

ISO 2211, *Liquid chemical products — Measurement of colour in Hazen units (platinum-cobalt scale)*.

3 SAMPLING

For the preparation of the laboratory sample, use the method specified in ISO 2209.

NOTE — The aqueous phase present shall be proportionally sampled.

In all cases, unless instructions are given to the contrary, three portions of the same laboratory sample shall be taken, each portion being sufficient to carry out all the analyses. Before making this division, stir the sample carefully in order to obtain a homogeneous mixture and pour this immediately into three dark glass bottles fitted with ground glass stoppers. These bottles shall be clean, dry, air-tight and of such capacity that they are almost completely filled by the sample. If it is necessary to seal the bottles, take every precaution to avoid contamination of the contents.

4 DETERMINATION OF DENSITY

Use the method specified in ISO/R 758.

5 DETERMINATION OF DISTILLATION CHARACTERISTICS

Use the method specified in ISO/R 918, subject to the additional details appropriate for carbon disulphide, as follows :

5.1 Distillation flask (see 3.1 of ISO/R 918). Short-necked, round-bottomed flask of 150 ml nominal capacity, of borosilicate glass, and a connecting tube with conical ground glass joints for connection to the condenser and supporting the thermometer, as shown in figures 2 and 3.

5.2 Thermometer (see 3.2 of ISO/R 918) with conical joint; scale including the range 20 to 51 °C, or any other suitable interval.

5.3 Thermostatic bath, electrically heated and capable of being controlled at $60 \pm 0,5$ °C, fitted with a stirrer. The dimensions of the bath shall be as shown in figure 4.

5.4 Procedure

Place 100 ml of the test sample in a measuring cylinder (see 3.3 of ISO/R 918). Transfer the liquid as completely as possible to the distillation flask (5.1). Connect the distillation flask, with thermometer (5.2) inserted, to the condenser specified in 3.4 of ISO/R 918. The temperature of the water circulating in the condenser shall not exceed 20 °C. Immerse the distillation flask in the thermostatic bath (5.3), controlled at $60 \pm 0,5$ °C, taking care that the liquid in the bath and that in the flask are at the same level. Collect the condensate in a measuring cylinder placed in a transparent glass vessel filled with water and pieces of ice (see figure 1).

NOTES

1 Avoid the use of any grease for lubrication of the ground glass joints of the apparatus.

2 Seal the condenser inlet at the moment of separating the distillation flask.

Record either :

- the temperatures corresponding to the volumes of condensates defined in the specification of the test product; these temperatures should be corrected as indicated in clause 7 of ISO/R 918, unless the specification of the product provides only for the determination of a difference of temperature between two volumes of condensate;

or

- the volumes of condensate at the moment when the thermometer indicates the distillation temperatures (corrected as indicated in clause 5 of ISO/R 918) laid down in the specification for the product.

5.5 Temperature correction

If the barometric pressure (corrected as indicated in clause 8 of ISO/R 918) differs from 760 mmHg, correct the observed temperatures.

The correction, which should be added algebraically to the observed distillation temperature, is equal to

$$0,040 (760 - p_1) ^\circ\text{C} \text{ or } \\ 0,030 (1\,013 - p_2) ^\circ\text{C}$$

where

p_1 is the barometric pressure in millimetres of mercury;

p_2 is the barometric pressure in kilopascals.¹⁾

6 DETERMINATION OF RESIDUE ON EVAPORATION

6.1 Principle

Determination of the mass of the residue after evaporation to dryness of a test portion at $60 \pm 2 ^\circ\text{C}$.

6.2 Procedure

For this determination use the same apparatus and the same procedure as in the determination of distillation characteristics, taking care to tare the distillation flask (5.1).

After completion of the distillation (see clause 5), disconnect the distillation flask and carefully clean the part of the flask which was immersed in the water bath. Place the flask horizontally in an electric oven, controlled at $60 \pm 2 ^\circ\text{C}$, for 1 h. Cool the flask and its contents in a desiccator and weigh.

Repeat the operations of heating in the oven, of cooling in the desiccator and of weighing until the mass is constant.

6.3 Expression of results

The residue on evaporation, expressed as a percentage by mass, is given by the formula

$$\frac{m_2 - m_1}{\rho}$$

where

m_1 is the mass, in grams, of the distillation flask;

m_2 is the mass, in grams, of the distillation flask and the residue after evaporation;

ρ is the density at $20 ^\circ\text{C}$, in grams per millilitre, of the test sample.

1) $1 \text{ kPa} = 1 \text{ kN/m}^2$.

7 DETERMINATION OF INORGANIC SULPHUR CONTENT

7.1 Determination of hydrogen sulphide content

7.1.1 Principle

Extraction of the hydrogen sulphide with a solution of zinc acetate. Formation of a coloured complex between the hydrogen sulphide and dimethyl-*p*-phenylenediamine. Photometric measurement of the coloured complex at a wavelength of about 670 nm.

7.1.2 Reagents

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water, or water of equivalent purity.

7.1.2.1 Zinc acetate dihydrate, 15 g/l in dilute acetic acid solution.

Dissolve 15 g of zinc acetate dihydrate $[\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}]$ in water, add glacial acetic acid (approximately 17 N solution) until the solution is clear, dilute to 1 000 ml with water and mix.

7.1.2.2 Dimethyl-*p*-phenylenediamine, 4 g/l solution in dilute hydrochloric acid.

Dissolve 0,4 g of dimethyl-*p*-phenylenediamine in 100 ml of approximately 6 N hydrochloric acid solution; shake with about 1 g of activated carbon and filter. Repeat the treatment with activated carbon until the filtrate becomes colourless.

7.1.2.3 Hydrogen sulphide, standard solution corresponding to 5 mg of H_2S per litre.

Weigh, to the nearest 0,001 g, 0,882 g of sodium sulphide hydrate ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$), dissolve in recently boiled and cooled water, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

Take 10,0 ml of the resultant solution, transfer it to a 250 ml one-mark volumetric flask, dilute to the mark with recently boiled and cooled water and mix.

1 ml of this standard solution corresponds to 0,005 mg of hydrogen sulphide (H_2S).

Prepare the two solutions at the time of use.

NOTE — If necessary, check iodometrically the concentration of this solution.

7.1.2.4 Iron(III) chloride, 25 g/l solution in dilute hydrochloric acid.

Dissolve approximately 2,50 g of iron(III) chloride hydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) in 100 ml of approximately 6 N hydrochloric acid solution.