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Carbon tetrachloride for industrial use — Methods of test

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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 2312 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in October 1971 by the Member Bodies of the following countries :

Austria	India	Switzerland
Belgium	Ireland	Thailand
Chile	Italy	Turkey
Czechoslovakia	Korea, Dem. P. Rep. of	United Kingdom
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The Member Body of the following country expressed disapproval of the document on technical grounds :

New Zealand

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Carbon tetrachloride for industrial use — Methods of test

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies methods of test of carbon tetrachloride for industrial use.

2 REFERENCES

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

ISO/R 760, *Determination of water by the Karl Fischer method.*

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

ISO/R 1393, *Liquid halogenated hydrocarbons for industrial use — Determination of the acidity.*

ISO/R 1394, *Liquid halogenated hydrocarbons for industrial use — Determination of the cloud point.*

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

ISO 2210, *Liquid halogenated hydrocarbons for industrial use — Determination of residue on evaporation.*

ISO 2211, *Liquid chemical products — Measurement of colour in Hazen units (Platinum-cobalt scale).*[1)]

3 SAMPLING

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

NOTE — The sampling and handling of the samples shall be carried out as rapidly as possible and in a way that reduces to a minimum contact of the sample with the atmosphere and exposure to light.

The samples taken both for analysis and for reference purposes shall be kept in clean and dry, glass-stoppered, dark-coloured bottles.

4 MEASUREMENT OF COLOUR IN HAZEN UNITS

Use the method specified in ISO 2211.

1) At present at the stage of draft.

5 DETERMINATION OF DISTILLATION CHARACTERISTICS

Use the method specified in ISO/R 918.

The following particulars and modifications, specific for carbon tetrachloride, shall be introduced in the above-mentioned document.

5.1 Scope (see section 1 in ISO/R 918)

This determination indicates

a) either the temperatures corresponding to the collection of two volumes of distillate, A and B,

b) or the difference between these two temperatures.

The two volumes, A and B, shall be indicated in the specifications for the product agreed between the interested parties.

5.2 Thermometer (see 3.2 in ISO/R 918)

Use a thermometer conforming to the requirements of ISO/R 918, with a scale from 69 to 81 °C or some other suitable scale (for example, 48 to 102 °C).

5.3 Distillation rate (see 6.2 in ISO/R 918)

4 to 5 ml/min.

5.4 Correction to be applied to the temperatures (see section 7 in ISO/R 918)

This correction is necessary only for case a). It is equal to $0.043\ (760 - p)$ °C, where p is the barometric pressure in millimetres of mercury, and shall be added to the distillation temperatures.

6 DETERMINATION OF DENSITY AT 20 °C

Use the method specified in ISO/R 758.

7 DETERMINATION OF RESIDUE ON EVAPORATION

Use the method specified in ISO 2210.

8 DETERMINATION OF WATER CONTENT

Use the method specified in ISO/R 760.

9 DETERMINATION OF THE CLOUD POINT

Use the method specified in ISO/R 1394.

10 DETERMINATION OF ACIDITY

Use the method specified in ISO/R 1393.

11 DETERMINATION OF CARBON DISULPHIDE CONTENT

11.1 Principle

Reaction of the carbon disulphide, in the presence of Cu(II) ions and diethylamine, to form copper diethyldithiocarbamate. This compound is soluble in carbon tetrachloride giving a yellow colour the intensity of which is a function of the carbon disulphide content. Photometric measurement of the intensity of the colour at about 425 nm.

11.2 Reagents

11.2.1 *Colour reagent*

Place 50 ml of water in a 1 000 ml one-mark volumetric flask, add 0.060 g of copper (II) acetate monohydrate $[Cu(CH_3 \cdot COO)_2 \cdot H_2O]$ and shake until dissolution is complete. Add 10.0 ml of diethylamine and 20.0 ml of triethanolamine.

Dilute to the mark with 95 % (V/V) ethanol and mix. Store the solution in the dark, in a closed flask, preferably closed by means of a polyethylene stopper.

11.2.2 *Carbon tetrachloride,* free from carbon disulphide.

11.2.3 *Carbon disulphide.*

11.2.4 *Carbon disulphide,* standard solution.

11.2.4.1 Introduce about 50 ml of the carbon tetrachloride (11.2.2) into a 100 ml one-mark volumetric flask and add 10.0 ml of the carbon disulphide (11.2.3). Dilute to the mark with the carbon tetrachloride (11.2.2) and mix.

The solution A thus obtained has a concentration of 8.09 % (m/m) of carbon disulphide (CS_2) in carbon tetrachloride.

11.2.4.2 Take 10.0 ml of solution A (11.2.4.1) and transfer to a 100 ml one-mark volumetric flask. Dilute to the mark with the carbon tetrachloride (11.2.2) and mix.

The solution B thus obtained has a concentration of 0.79 % (m/m) of carbon disulphide in carbon tetrachloride.

11.2.4.3 Repeat the operation described in 11.2.4.2 twice more.

The solution C thus obtained has a concentration of 0.007 9 % (m/m) of carbon disulphide in carbon tetrachloride.

11.3 Apparatus

Ordinary laboratory apparatus and

11.3.1 *Spectrophotometer*

or

11.3.2 *Photoelectric absorptiometer,* fitted with appropriate filters.

11.3.3 *Chronometer,* for measuring times up to 5 min.

11.4 Procedure

11.4.1 *Preparation of calibration curve*

11.4.1.1 Preparation of standard matching solutions

Into a series of four 100 ml one-mark volumetric flasks, place respectively 5.0 — 7.5 — 10.0 — 12.5 ml of the standard carbon disulphide solution C (11.2.4.3). Dilute to the mark with carbon tetrachloride (11.2.2) and mix.

The standard matching solutions thus prepared contain respectively the following contents by mass of carbon disulphide (CS_2) in carbon tetrachloride, rounded to the fourth decimal place :

0.000 4 — 0.000 6 — 0.000 8 — 0.001 0

11.4.1.2 Development of colour

By means of a pipette, place 50 ml of the colour reagent (11.2.1) into each of two 100 ml conical flasks. To one of the flasks add, by means of a pipette, 5.0 ml of the standard matching solution (11.4.1.1) and to the other 5.0 ml of the carbon tetrachloride (11.2.2) (compensating solution).

Start the chronometer (11.3.3), mix and transfer the solutions to the two cells of the spectrophotometer with appropriate optical path length.

11.4.1.3 Photometric measurements

Carry out the measurements 5 min after the start of colour development using the spectrophotometer (11.3.1) at a wavelength in the region of 425 nm or the photoelectric absorptiometer (11.3.2) fitted with appropriate filters, adjusting the instrument to zero absorbance against the compensating solution.

Carry out the operations of colour development and photometric measurement for each of the standard matching solutions.

11.4.1.4 Preparation of calibration chart

Prepare a calibration chart having, for example, the carbon disulphide contents as percentages by mass as abscissae and the corresponding values of the photometric measurements as ordinates.

11.4.2 *Determination*

11.4.2.1 Development of colour

By means of a pipette, place 50 ml of the colour reagent (11.2.1) in two 100 ml conical flasks. To one of the flasks add, by means of a pipette, 5.0 ml of the sample and to the other 5.0 ml of the carbon tetrachloride (11.2.2) (compensating solution).

Start the chronometer (11.3.3), mix and transfer the solutions to the cells of the apparatus.

11.4.2.2 Photometric measurements

Carry out the photometric measurements 5 min after the start of the colour development following the directions given in 11.4.1.3.

NOTE – If the carbon disulphide content of the sample is greater than the upper limit of the calibration chart, the sample may be suitably diluted for the determination using carbon tetrachloride (11.2.2). Take account of this dilution in calculating the results.

11.5 Expression of results

Determine, by means of the calibration chart (11.4.1.4), the carbon disulphide (CS_2) content, expressed as a percentage by mass. If the sample was diluted with carbon tetrachloride (11.2.2), take this dilution into account in calculating the results.

12 TEST REPORT

The test report shall include, for each test, 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 those ISO documents to which reference is made, or any operation regarded as optional.

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