BRITISH STANDARD

BS 662-3:1979

Carbon disulphide for industrial use —

Part 3: Additional methods of test

Confirmed
January 2011

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Cooperating organizations

The Chemicals Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Association of Fatty Acid Distillers
British Tar Industry Association
Chemical Industries' Association*
Chemical Society, Analytical Division
Department of Health and Social Security
Department of Industry, Laboratory of the Government Chemist
Fertiliser Manufacturers' Association Ltd.
Hydrocarbon Solvents Association
Institution of Gas Engineers
Ministry of Agriculture Fisheries and Food
Ministry of Defence
National Sulphuric Acid Association
Paintmakers' Association of Great Britain Ltd.
Royal Institute of Public Health and Hygiene
Soap and Detergent Industry Association
Standardization of Tar Products Tests Committee

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

British Man-made Fibres Federation
British Plastics Federation
Oil and Colour Chemists Association
Pharmaceutical Society of Great Britain
Royal Institute of Chemistry
Individual Company

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Executive Board on 31 January 1979

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The following BSI references relate to the work on this standard:
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Amendments issued since publication

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Contents

Foreword

This British Standard has been prepared under the direction of the Chemicals Standards Committee to provide a specification and methods of test for carbon disulphide for industrial use.

The first edition of this standard was issued in 1936. A revision, published in 1950, was amended in 1954 to include the determination of sulphur dioxide, and was confirmed in 1960. The present edition extends the scope of the specification and incorporates ISO 3144.

This standard is presented in three Parts:

— *Part 1: Specification;*

— *Part 2: Sampling and methods of test;*

— *Part 3: Additional methods of test.*

Part 2 is identical with ISO 3144 "*Carbon disulphide for industrial use — Sampling and methods of test*" and includes the following test methods:

Determination of density

Determination of distillation characteristics

Determination of residue on evaporation

Determination of inorganic sulphur content

Determination of alkalinity or acidity

Part 3 specifies methods of test for thiophene, benzene, mercaptans (thiols) and hydrocarbons, which are not included in the ISO publication, and gives an alternative method for the determination of hydrogen sulphide and inorganic sulphur which avoids the use of the extremely deliquescent disodium sulphide nonahydrate.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This Part of BS 662 specifies methods for the determination of sulphur compounds, benzene and hydrocarbons in carbon disulphide for industrial use.

WARNING NOTE. Carbon disulphide is highly volatile and gives off vapour at normal room temperature. It is harmful by inhalation of the vapour, by prolonged or repeated contact with the liquid or if taken into the mouth.

Further information is available in Technical Data note 2 published by the Health and Safety Executive on an annual basis.

Carbon disulphide is a highly flammable volatile liquid with a low flash point, a wide explosive range and an auto-ignition temperature lower than 120 °C. Contact with exposed steam lines or the surface of an ordinary electric light bulb can cause ignition.

Because of its hazardous nature, carbon disulphide should always be handled in accordance with the supplier's safety recommendations.

2 References

The titles of the publications referred to in this standard are listed on the inside back cover.

3 Determination of hydrogen sulphide and sulphur dioxide

3.1 Principle. Titration of an aliquot portion of the laboratory sample with a standard volumetric iodine solution to a permanent pink colouration.

3.2 Reagents. Use only reagents of a recognized analytical quality and water complying with the requirements of BS 3978.

3.2.1 *Iodine*. 0.01N standard volumetric solution.

3.3 Apparatus. In addition to ordinary laboratory apparatus the following is required.

3.3.1 *Burette,* complying with the requirements of class A of BS 846, capable of being read to 0.05 ml.

3.4 Procedure. Place in a clean dry 250 ml glass stoppered flask a 100 ml aliquot portion of the test sample which has been previously filtered through a clean dry filter paper. Add from the burette (**3.3.1**) the standard volumetric iodine solution (**3.2.1**), in 0.1 ml portions. Stopper the flask and shake it for 15 s after each addition.

Continue the titration until there is a permanent pink coloration in the carbon disulphide.

Record the volume of standard volumetric iodine solution required to give the permanent pink coloration. Repeat the procedure on a fresh aliquot portion.

3.5 Expression of results. The hydrogen sulphide plus sulphur dioxide content, expressed as milligrams of hydrogen sulphide per kilogram, is given by the expression:

$$\frac{1.7\ V}{\rho}$$

where

V is the volume (in ml) of the standard volumetric iodine solution (**3.2.1**) required to give the permanent pink coloration;

ρ is the density (in g/ml) of the carbon disulphide

3.6 Precision. The precision of the method, as obtained by statistical examination of interlaboratory test results is as follows.

3.6.1 *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 0.05 mg/kg only in one case in twenty.

3.6.2 *Reproducibility.* No data are available for the reproducibility of the method.

3.7 Test report. The test report shall include:

a) a reference to this British Standard;

b) the results and method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this British Standard.

4 Determination of thiophene, benzene and hydrocarbons

4.1 Principle. Spectrophotometric measurement at the appropriate wavelength and determination of the concentration of the impurity from a calibration curve.

4.2 Reagents. Use only reagents of a recognized analytical quality and water complying with the requirements of BS 3978.

4.2.1 *Carbon disulphide,* spectrometric quality, free from thiophene, benzene and hydrocarbons.

4.2.2 *Thiophene*

4.2.3 *Benzene*

4.2.4 *Hydrocarbon oil,* boiling range 160 °C to 250 °C.

4.3 Apparatus. In addition to ordinary laboratory apparatus the following are required.

4.3.1 *Infra-red spectrophotometer*

4.3.2 *Sodium chloride cells,* one matched pair, path length 1 cm.

4.4 Procedure. Using the carbon disulphide (**4.2.1**) and the matched cells (**4.3.2**) set the spectrophotometer (**4.3.1**) to zero in accordance with the manufacturer's instructions.

Using the carbon disulphide (**4.2.1**), prepare three series of standard solutions of the thiophene (**4.2.2**), the benzene (**4.2.3**) and the hydrocarbon oil (**4.2.4**) of the following concentrations:

10 20 30 40 50 60 70 80 90 100 mg/kg

Measure the absorbance of each series of solutions using the spectrophotometer (**4.3.1**) set at the appropriate wavenumber (wavelength) given in Table 1 and with carbon disulphide in the reference cell.

Table 1 — Wavenumbers for the determination of thiophene, benzene and hydrocarbons

Solute	Wavenumber (wavelength)	
	cm^{-1}	μm
Thiophene	125	(8.00)
Benzene	180.8	(5.53)
Hydrocarbons	293.3	(3.41)

Plot calibration curves from each series of measurements.

Charge one of the matched cells (**4.3.2**) with a filtered portion of the test sample and measure and record the absorbance at each of the wavenumbers (wavelengths) given in Table 1.

4.5 Expression of results. For each of the absorbance values obtained for the test sample as described, read off from the appropriate calibration curve the concentration, in mg/kg, of thiophene, benzene and hydrocarbons present in the test sample.

4.6 Precision. The precision of the method as obtained by statistical examination of interlaboratory test results is as follows.

4.6.1 *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 values given in one case in twenty.

thiophene	10 mg/kg
benzene	10 mg/kg
hydrocarbons	10 mg/kg

4.6.2 *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 values given in one case in twenty.

thiophene	20 mg/kg
benzene	20 mg/kg
hydrocarbons	20 mg/kg

4.7 Test report. The test report shall include:

a) a reference to this British Standard;

b) the results and method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this British Standard.

5 Determination of mercaptans (thiols)

5.1 Principle. Treatment with silver nitrate solution followed by titration of an aqueous extract with 0.01N standard volumetric sodium hydroxide solution.

5.2 Reagents. Use only reagents of a recognized analytical quality and water complying with the requirements of BS 3978.

5.2.1 *Silver nitrate,* approximately 10 % (*m*/*m*) solution.

5.2.2 *Sodium hydroxide,* 0.01N standard volumetric solution.

5.2.3 *Methyl red indicator solution,* prepared in accordance with the requirements of BS 4123.

5.2.4 *Carbon disulphide,* free from mercaptans (thiols), prepared by shaking, in a separating funnel, with the silver nitrate solution (**5.2.1**) until no coloration is produced and then washing with distilled water.

5.3 Apparatus. Ordinary laboratory apparatus.

5.4 Procedure

WARNING NOTE. During the procedure vent the separating funnel frequently.

Test for the presence of hydrogen sulphide and, if present, remove it by shaking the test portion with 50 ml of a 5 % solution of acidified cadmium chloride. Separate the carbon disulphide layer and wash with water until the washings are neutral.

To a 300 ml aliquot portion of the test sample in a clean, dry 500 ml separating funnel, add 25 ml of the silver nitrate solution (**5.2.1**) and 50 ml of water. Stopper and shake the funnel, allow the layers to separate, collect the carbon disulphide layer in a second separating funnel and wash with 50 ml of water.

Combine the aqueous layers in a 250 ml conical flask. Add a few drops of the methyl red indicator solution (**5.2.3**) and titrate with the sodium hydroxide solution (**5.2.2**) to the green end point.

Carry out a blank determination using the carbon disulphide free from mercaptans (**5.2.4**) in place of the test portion.

5.5 Expression of results. The mercaptan content, expressed in milligrams of mercaptan sulphur per kilogram, is given by the expression:

$$0.85\ (V_1 - V_2)$$

where

V_1 is the volume (in ml) of the sodium hydroxide solution (**5.2.2**) required for the test portion;

V_2 is the volume (in ml) of the sodium hydroxide solution (**5.2.2**) required for the blank.

5.6 Precision. No data are available for the precision of the method.

5.7 Test report. The test report shall include:

a) a reference to this British Standard;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this British Standard.

blank

Publications referred to

BS 846, *Burettes and bulb burettes.*
BS 3978, *Water for laboratory use.*
BS 4123, *Schedule of preferred chemical indicators.*

BSI — British Standards Institution

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