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Carbon disulphide for industrial use — Sampling and methods of test

ERRATUM

Page 3

In sub-clause 7.1.5, second paragraph, second line, replace "(H_2O)" by "(H_2S)".

In the left-hand side of the following equation, replace "m_2" by "m_3".

INTERNATIONAL STANDARD 3144

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

Carbon disulphide for industrial use — Sampling and methods of test

Sulfure de carbone à usage industriel — Échantillonnage et méthodes d'essai

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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 3144 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in June 1973.

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

Austria	Hungary	Spain
Belgium	India	Switzerland
Bulgaria	Israel	Thailand
Czechoslovakia	Italy	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	Poland	U.S.S.R.
Germany	South Africa, Rep. of	

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 ± 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 ± 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

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

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 ± 2 °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 ± 2 °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 °C, in grams per millilitre, of the test sample.

1) 1 kPa = 1 kN/m².

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 $[Zn(CH_3COO)_2.2H_2O]$ 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 ($Na_2S{\cdot}9H_2O$), 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 ($FeCl_3{\cdot}6H_2O$) in 100 ml of approximately 6 N hydrochloric acid solution.

7.1.2.5 Carbon disulphide, free from hydrogen sulphide.

Vigorously shake recently distilled carbon disulphide with approximately 20 % (*V/V*) of the zinc acetate solution (7.1.2.1). Separate the phases and distil the carbon disulphide phase on a water bath at 60 °C, discarding the first portions of the distillate.

7.1.3 *Apparatus*

Ordinary laboratory apparatus and

7.1.3.1 Spectrophotometer or

7.1.3.2 Photoelectric absorptiometer.

7.1.4 *Procedure*

7.1.4.1 TEST PORTION

Take 10 ± 0,1 ml of the test sample (i.e. 12,6 g), containing 0,005 to 0,05 mg of hydrogen sulphide, and weigh this test portion to the nearest 0,01 g.

7.1.4.2 BLANK TEST

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as those used for the determination.

7.1.4.3 PREPARATION OF THE CALIBRATION CURVE

7.1.4.3.1 Preparation of the standard matching solutions

Into a series of eight 100 ml separating funnels, pour the volumes of the standard hydrogen sulphide solution (7.1.2.3) and the carbon disulphide (7.1.2.5) indicated in table 1.

TABLE 1

7.1.4.3.2 Extraction of the hydrogen sulphide

To each of the separating funnels, add 25,0 ml of the zinc acetate solution (7.1.2.1) and shake vigorously for 5 min. Allow to stand, separate the two phases and transfer 20,0 ml of the aqueous phase to a 50 ml one-mark volumetric flask.

7.1.4.3.3 Colour development

Add 2 ml of the dimethyl-*p*-phenylenediamine solution (7.1.2.2) and 0,5 ml of the iron(III) chloride solution (7.1.2.4) and dilute to the mark.

Mix and leave undisturbed for 20 min.

7.1.4.3.4 Photometric measurements

WARNING. The compensation solution (**) shall have an absorbance equal to that of the matching test solution (*).

Carry out the photometric measurements with the spectrophotometer (7.1.3.1), at a wavelength of about 670 nm, or the photoelectric absorptiometer (7.1.3.2), fitted with suitable filters, after having adjusted the apparatus to zero absorbance against the matching test solution (*). Use a cell of optical path length suited to the characteristics of the measuring instrument.

7.1.4.3.5 Preparation of the calibration chart

Plot a graph having, for example, the number of milligrams of H_2S contained in 50 ml of the standard matching solutions as abscissae and the corresponding values of the absorbance as ordinates.

7.1.4.4 DETERMINATION

Transfer 25,0 ml of the zinc acetate solution (7.1.2.1) to a 100 ml separating funnel and add the test portion (7.1.4.1). Shake vigorously for 5 min. Allow to stand, separate the two phases and transfer 20,0 ml of the aqueous phase to a 50 ml one-mark volumetric flask. Proceed as described in

7.2 Determination of sulphur dioxide + sulphur trioxide content

7.2.1 *Principle*

Separation of an aqueous extract of carbon disulphide. Turbidimetric measurement of the barium sulphate obtained by precipitation, under well-defined conditions, with barium chloride, of the water-soluble sulphur compounds, after oxidation with hydrogen peroxide.

7.2.2 *Reagents*

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

7.2.2.1 Barium chloride dihydrate, standardized by screening, of uniform particle size ranging between 0,50 and 1,25 mm.

It is essential that all preparations concerning the determination and calibration should be carried out with a product having the same particle size distribution.

7.2.2.2 Hydrogen peroxide, 35 % (*m/m*) solution.

7.2.2.3 Hydrochloric acid, approximately N solution.

7.2.2.4 Sulphuric acid, standard solution containing 100 mg of SO_4 per litre.

Transfer 20,8 ml of exactly 0,1 N sulphuric acid solution to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,1 mg of SO_4.

7.2.3 *Apparatus*

Ordinary laboratory apparatus and

7.2.3.1 Spectrophotometer or

7.2.3.2 Photoelectric absorptiometer fitted with filters giving only negligible transmission below 450 nm and above 550 nm.

7.2.4 *Procedure*

7.2.4.1 TEST PORTION

Take 10 ± 0,1 ml of the test sample (i.e. 12,6 g), containing a mass of water-soluble sulphur compounds equivalent to 0,2 to 4 mg of SO_4. Weigh this test portion to the nearest 0,01 g.

7.2.4.2 BLANK TEST

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as those used for the determination.

7.2.4.3 PREPARATION OF THE CALIBRATION CURVE

7.2.4.3.1 Preparation of the standard matching solutions

Into a series of ten 50 ml one-mark volumetric flasks, place the volumes of the standard sulphuric acid solution (7.2.2.4) indicated in table 2.

TABLE 2

Standard sulphuric acid solution (7.2.2.4)	Corresponding mass of SO_4
ml	mg
0 *	0
2,0	0,2
5,0	0,5
10,0	1,0
15,0	1,5
20,0	2,0
25,0	2,5
30,0	3,0
35,0	3,5
40,0	4,0

* Compensation solution.

Add 5 ml of the hydrochloric acid solution (7.2.2.3) to each flask, stir, dilute to the mark and mix.

7.2.4.3.2 Turbidimetric reaction

Pour the contents of each volumetric flask rapidly into a dry 100 ml beaker containing approximately 0,3 g of the barium chloride (7.2.2.1). Stir by hand for 1 min at a rate of 2 rev/s. Leave undisturbed for 15 min at 20 ± 2 °C.

NOTE — Stagger the tests in such a way as to adhere to the contact times indicated.

7.2.4.3.3 Turbidimetric measurements

Stir the solution which is to be subjected to photometry by hand, transfer it to a cell of suitable optical path length and carry out the measurement with the aid of the spectrophotometer (7.2.3.1), at a wavelength of about 470 nm, or by means of the photoelectric absorptiometer (7.2.3.2), fitted with suitable filters, after having adjusted the instrument to zero absorbance against the compensation solution.

7.2.4.3.4 Preparation of the calibration chart

Plot a graph having, for example, the number of milligrams of SO_4 contained in 50 ml of the standard matching solutions as abscissae and the corresponding values of the absorbance as ordinates.

7.2.4.4 DETERMINATION

7.2.4.4.1 Preparation of the test solution

Place the test portion (7.2.4.1) and 25 ± 0,1 ml of water in a dry 100 ml separating funnel. Shake vigorously for 3 min.

Leave undisturbed until the phases have separated and transfer the aqueous phase to a dry vessel, filtering it through dry filter paper.

Pour 20 ± 0,1 ml of the filtered aqueous phase into a 50 ml one-mark volumetric flask, add 5 ml of the hydrogen peroxide solution (7.2.2.2) and 5 ml of the hydrochloric acid solution (7.2.2.3), shake, leave undisturbed for 10 min, dilute to the mark and mix.

7.2.4.4.2 Turbidimetric reaction

Take 25,0 ml of the test solution (7.2.4.4.1) and transfer it rapidly to a dry 100 ml beaker containing approximately 0,15 g of the barium chloride (7.2.2.1). Stir by hand for 1 min at a rate of 2 rev/s. At this point, the barium chloride should be completely in solution. Allow to stand for 15 min.

7.2.4.4.3 Turbidimetric measurement

Transfer a sufficient quantity of the test solution (7.2.4.4.1) to a cell of the same optical path length as that used for preparing the calibration curve (7.2.4.3) and use this solution as compensation solution for adjusting the spectrophotometer (7.2.3.1) or the photoelectric absorptiometer (7.2.3.2) to zero absorbance.

Stir the solution (7.2.4.4.2) by hand, transfer it to a cell of the same optical path length and proceed with the turbidimetric measurement by the method described in 7.2.4.3.3.

7.2.5 *Expression of results*

By means of the calibration curve (see 7.2.4.3.4), determine the quantity of SO_4 corresponding to the value of the turbidimetric measurements. The $SO_2 + SO_3$ content, expressed as milligrams of SO_4 per kilogram of product, is given by the formula

$$\frac{(m_4 - m_5) \times 1\,000}{m_6} \times \frac{20}{25} = \frac{m_4 - m_5}{m_6} \times 800$$

where

m_4 is the mass, in milligrams, of SO_4 found in the test solution;

m_5 is the mass, in milligrams, of SO_4 found in the blank test solution;

m_6 is the mass, in grams, of the test portion.

8 DETERMINATION OF ALKALINITY OR ACIDITY

8.1 Principle

Separation of an aqueous extract of carbon disulphide. Titration of the acidity or alkalinity of this aqueous extract with a standardized solution of sodium hydroxide or hydrochloric acid in the presence of bromocresol green as indicator.

8.2 Reagents

During the analysis, use only reagents of recognized analytical reagent grade.

8.2.1 Distilled water, neutral to bromocresol green.

Pour distilled water into a conical flask with a ground glass stopper, add 1 % (*V/V*) of the bromocresol green solution (8.2.4), and neutralize with the solution hydroxide solution (8.2.3) until a colour change to pure blue occurs.

8.2.2 Hydrochloric acid, 0,01 N standard volumetric solution.

8.2.3 Sodium hydroxide, 0,01 N standard volumetric solution.

8.2.4 Bromocresol green, 1 g/l ethanolic solution.

Dissolve 0,1 g of bromocresol green in 95 % (*V/V*) ethanol and dilute to 100 ml with the same ethanol.

8.3 Apparatus

Ordinary laboratory apparatus and

8.3.1 Stopwatch

8.4 Procedure

8.4.1 *Test portion*

Take 50,0 ml of the test sample.

8.4.2 *Preparation of the test solution*

Transfer the test portion (8.4.1) to a separating funnel of suitable capacity (250 ml, for example) containing 100 ml of the water (8.2.1). Shake for exactly 3 min, timed by the stopwatch (8.3.1). Allow the two phases to separate, filter about 60 ml of the aqueous phase through a dry filter and collect the filtrate in a dry vessel. If the aqueous phase is coloured yellow, the sample is acid; if it is coloured blue, the sample is neutral or alkaline.

8.4.3 *Titration*

Transfer 50,0 ml of the test solution (8.4.2) to a beaker of suitable capacity (250 ml, for example) and titrate with the standard volumetric sodium hydroxide solution (8.2.3) or with the standard volumetric hydrochloric acid solution (8.2.2), as indicated below.

8.4.3.1 DETERMINATION OF ACIDITY

Titrate the test solution (8.4.3) with the standard volumetric sodium hydroxide solution (8.2.3) until a colour change to pure blue occurs, using the water (8.2.1) as standard end-point matching solution.

8.4.3.1 DETERMINATION OF ALKALINITY

Titrate the test solution (8.4.3) with the standard volumetric hydrochloric acid solution (8.2.2) until a yellow colour appears, then back-titrate with the standard volumetric sodium hydroxide solution (8.2.3) until a colour change to pure blue occurs, using the water (8.2.1) as standard end-point matching solution.

8.5 Expression of results

According to the case, calculate the acidity or alkalinity as indicated below.

8.5.1 *Calculation of the acidity*

The acidity, expressed as a percentage by mass (*m/m*) of sulphuric acid (H_2SO_4), is given by the formula

$$\frac{V_0 \times 0{,}000\ 49}{\rho} \times 4 = \frac{V_0}{\rho} \times 0{,}001\ 96$$

where

V_0 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (8.2.3) used for the titration;

ρ is the density, in grams per millilitre, of the test sample;

0,000 49 is the mass, in grams, of sulphuric acid corresponding to 1 ml of 0,01 N standard volumetric sodium hydroxide solution.

8.5.2 *Calculation of the alkalinity*

The alkalinity, expressed as a percentage by mass (*m/m*) of sodium hydroxide (NaOH), is given by the formula

$$\frac{(V_1 - V_2) \times 0{,}000\ 40}{\rho} \times 4 = \frac{(V_1 - V_2)}{\rho} \times 0{,}001\ 6$$

where

V_1 is the volume, in millilitres, of the standard volumetric hydrochloric acid solution (8.2.2) used to obtain the yellow colour;

V_2 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (8.2.3) used in the back-titration (pure blue colour);

ρ is the density, in grams per millilitre, of the test sample;

0,000 40 is the mass, in grams, of sodium hydroxide corresponding to 1 ml of 0,01 N standard volumetric hydrochloric acid solution.

8.5.3 Alternatively, the acidity or alkalinity can be expressed in milligram equivalents per litre.

NOTE – If the concentrations of the standard volumetric solutions used are not exactly as specified in the list of reagents, an appropriate correction should be made.

9 DETERMINATION OF COLOUR

Use the method specified in ISO 2211.

NOTE – If the sample is stored under water, separate the carbon disulphide from water by means of a separating funnel.

The sample to be tested should be taken after decantation of any solid matter in suspension and should be perfectly clear.

10 TEST REPORT

The test report shall include, for each test carried out, 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 regarded as optional.

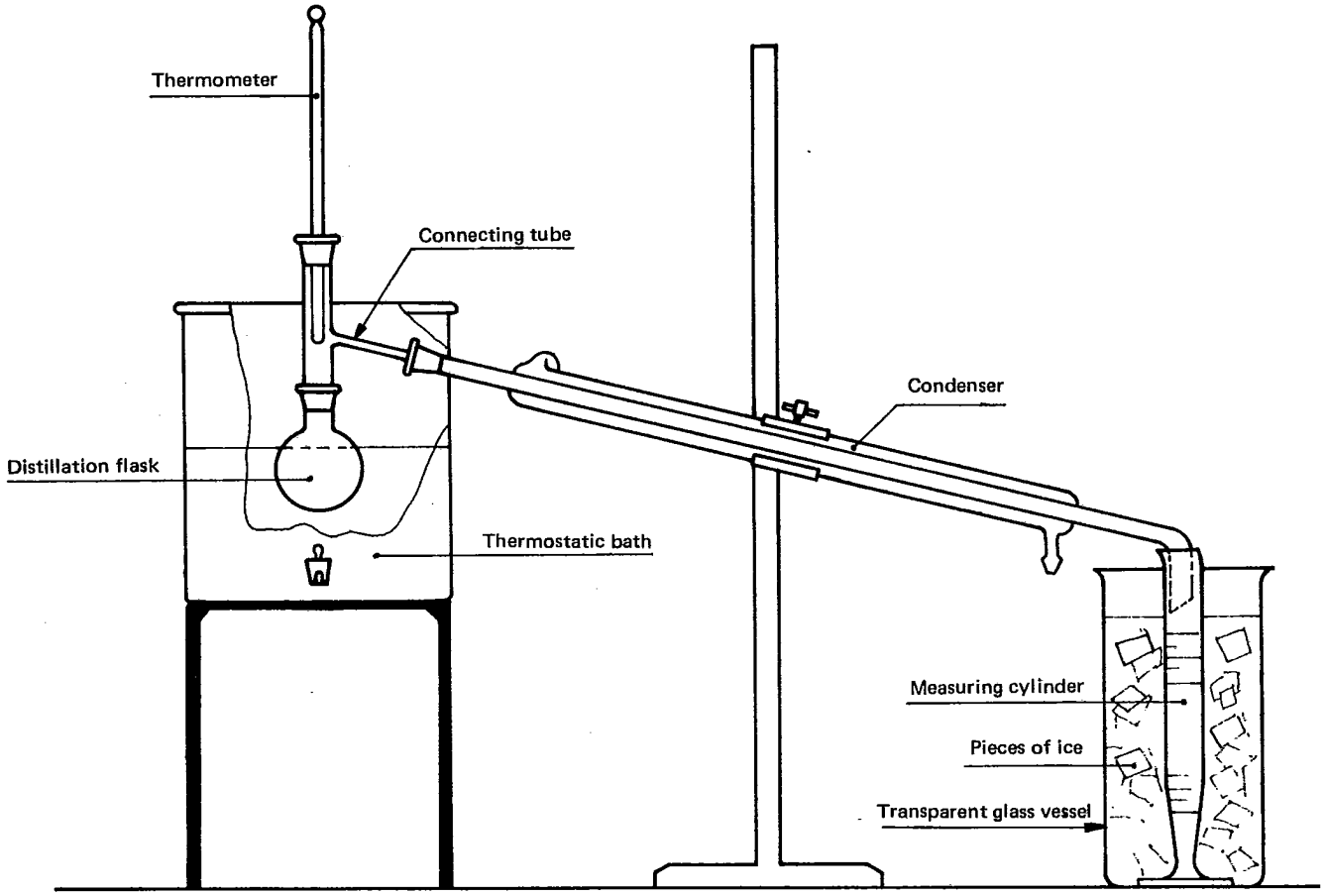

FIGURE 1 — **Assembled distillation apparatus**

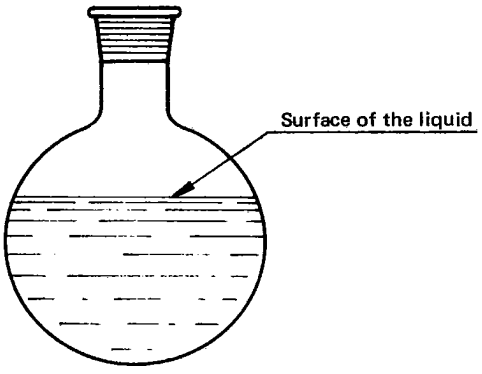

FIGURE 2 — **Distillation flask**

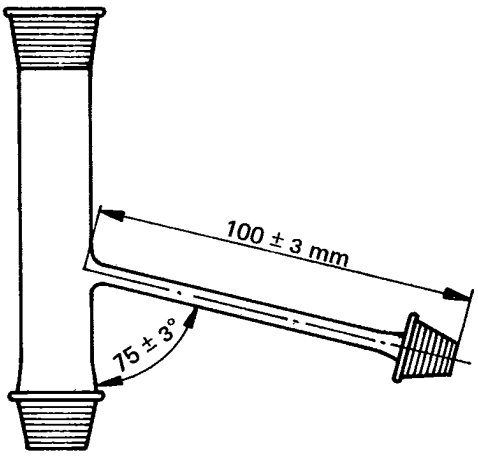

FIGURE 3 — **Connecting tube with conical ground glass joints**

Dimensions in millimetres

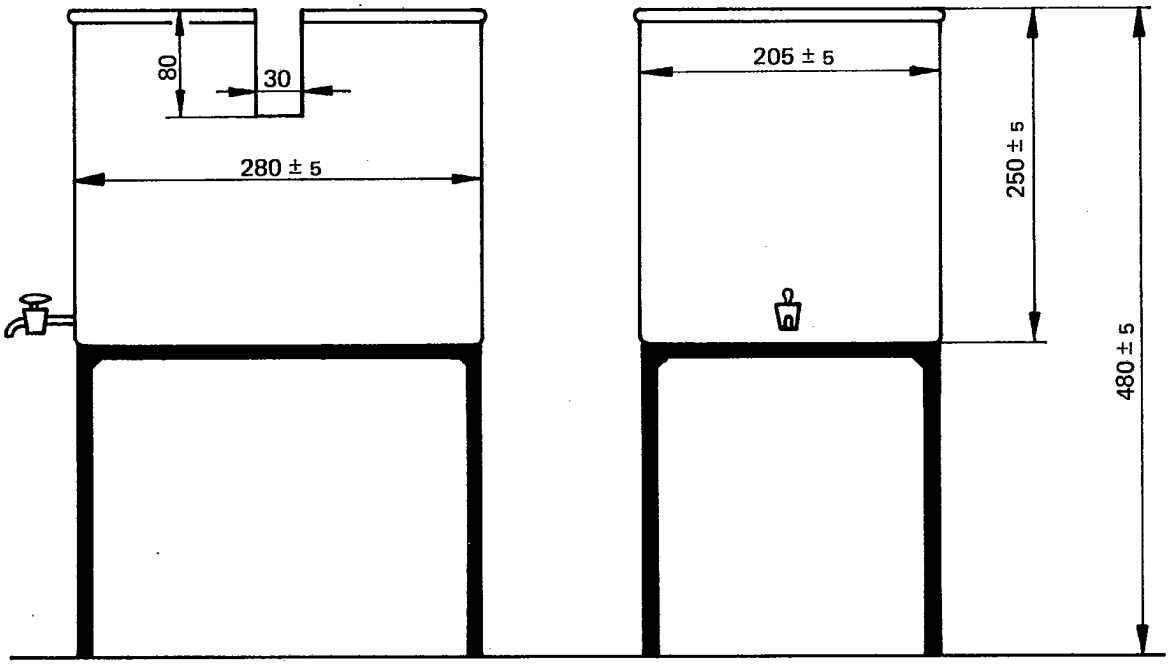

FIGURE 4 — **Electrically heated thermostatic bath**