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Liquefied phenol for industrial use — Determination of phenols content — Bromination method

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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 1904 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in April 1970 by the Member Bodies of the following countries:

Australia	Hungary	Portugal
Belgium	India	Romania
Chile	Israel	South Africa, Rep. of
Czechoslovakia	Italy	Spain
Egypt, Arab Rep. of	Japan	Switzerland
France	Netherlands	Thailand
Germany	New Zealand	Turkey
Greece	Poland	U.S.S.R.

The Member Body of the following country expressed disapproval of the document on technical grounds:

United Kingdom

Printed in Switzerland

Liquefied phenol for industrial use — Determination of phenols content — Bromination method

WARNING. Phenols burn the skin and can be absorbed into the system through the skin. It is essential for the sampler to wear protective gloves, for example of polyvinyl chloride, and also a face shield. Inhalation of the vapours from hot material is to be avoided.

Phenols are extremely hygroscopic, and care should be taken to avoid contamination with atmospheric or other moisture.

1 SCOPE AND FIELD OF APPLICATION

This International Standard describes a method for the determination of the phenols content of "liquefied phenol" by bromination. The method is not specific for phenol but determines the total amount of material that can be brominated under the conditions of the test.

The method as described is applicable to mixtures of about 80 % phenol for industrial use and 20 % water (V/V) (commonly called "liquefied phenol"). It can be applied to any mixture of phenol in water, provided that suitable adjustments are made.

2 SAMPLING

Apply the principles given in ISO ...[1]. The following principles shall also be observed :

Place the laboratory sample representative of the material taken from the bulk in a clean, dry, dark-coloured, glass-stoppered bottle of such a size that it is nearly filled by the sample. If it is necessary to seal this bottle, care should be taken to avoid contaminating the contents.

3 PRINCIPLE

Reaction between phenol and bromine from a measured amount of standard bromide/bromate solution with the formation of tribromophenol, followed by iodometric determination and allowance for excess bromine using a standard volumetric solution of sodium thiosulphate.

4 REAGENTS

Distilled water or water of equivalent purity shall be used in the test.

4.1 Chloroform, ρ 1.49 g/ml approximately.

4.2 Hydrochloric acid, ρ 1.19 g/ml approximately 38 % (m/m) solution.

4.3 Potassium iodide, 150 g/l solution.

4.4 Potassium bromide/bromate, 0.1 N standard volumetric solution. Dissolve 10 g of potassium bromide and 2.784 g of potassium bromate in water, then transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark, and mix.

4.5 Sodium thiosulphate, 0.1 N standard volumetric solution.

4.6 Indicator. Use either:

4.6.1 Starch, 5 g/l solution, freshly prepared, or

4.6.2 Sodium starch glycollate, 5 g/l solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Iodine flask, 500 ml, as shown in the Figure.

Dimensions in millimetres

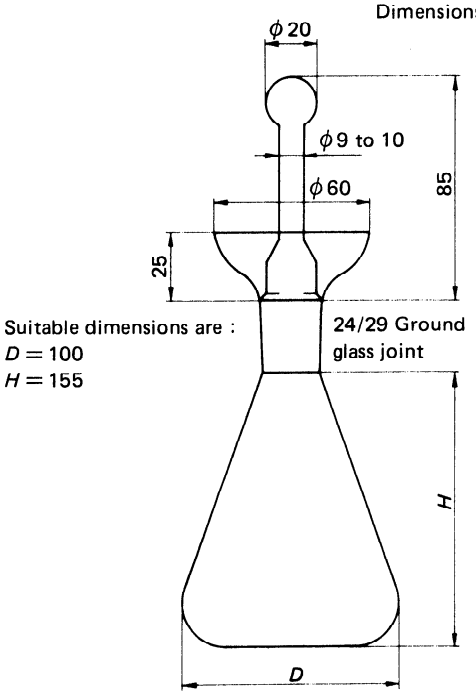

FIGURE — **500 ml iodine flask (5.1)**

1) Sampling of chemical products will form the subject of a future International Standard.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0.001 g, about 2.5 g of the laboratory sample from a weighing pipette into a 1 000 ml one-mark volumetric flask.

6.2 Blank test

Carry out a blank test on the reagents as follows:

Place 50.0 ml of the potassium bromide/bromate solution (4.4) in the iodine flask (5.1). Add 15 ml of the potassium iodide solution (4.3) and 10 ml of chloroform (4.1), followed by 5 ml of the hydrochloric acid solution (4.2). Stopper immediately and shake thoroughly. Titrate the liberated iodine with the sodium thiosulphate solution (4.5) until the contents of the flask become only faintly yellow, add a few drops of the indicator (4.6.1 or 4.6.2) and continue the titration until the last traces of the blue colour have disappeared.

6.3 Preparation of the sample solution and determination

Dilute the test portion (6.1) in the 1 000 ml one-mark volumetric flask to the mark with water and mix.

Transfer 25.0 ml of this solution into the iodine flask (5.1). Add 50.0 ml of the bromide/bromate solution (4.4) followed by 5 ml of the hydrochloric acid solution (4.2). Stopper immediately and seal by running a little of the potassium iodide solution (4.3) into the annular space between the stopper and the funnel-shaped neck of the flask. Mix the contents of the flask by occasional swirling during 30 min and then leave to stand for a further 15 min.

Add 20 ml of the potassium iodide solution (4.3), in small portions, to the contents of the flask by pouring it into the annular space on the neck and gently easing the stopper. Shake the flask thoroughly and titrate the contents by means of the sodium thiosulphate solution (4.5) until only a faint yellow colour remains. Add 10 ml of the chloroform (4.1) to dissolve the bulky precipitate of bromophenol which otherwise is liable to absorb iodine, and follow by a few drops of the indicator (4.6.1 or 4.6.2).

Continue titrating and shaking until the last traces of the blue colour have disappeared.

7 EXPRESSION OF RESULTS

Phenols content, expressed as phenol, is given, as a percentage by mass, by the formula :

$$\frac{156.8\,(V_1 - V_2)}{25\,m}$$

where

V_1 is the volume, in millilitres, of the sodium thiosulphate solution (4.5) used in the blank test;

V_2 is the volume, in millilitres, of the sodium thiosulphate solution (4.5) used for the determination;

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

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 usual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.

ANNEX

This document forms one of a series on methods of test for phenol, cresols, cresylic acid and xylenols for industrial use.

The complete list of those documents already prepared or in course of preparation is as follows:

PHENOL, *o*-CRESOL, *m*-CRESOL, *p*-CRESOL, CRESYLIC ACID, XYLENOLS

ISO/R 1897, *Determination of water by the Karl Fischer method.*
ISO/R 1898, *Determination of water by the Dean and Stark method.*
ISO/R 1899, *Determination of neutral oils and pyridine bases.*

PHENOL, *o*-CRESOL, *m*-CRESOL, *p*-CRESOL

ISO/R 1900, *Determination of residue on evaporation.*
ISO/R 1901, *Determination of crystallizing point.*
ISO 2208, *Determination of crystallizing point after drying with a molecular sieve.*[1)]
ISO/R 1902, *Test for impurities insoluble in sodium hydroxide solution – Visual test.*
ISO 2273, *Determination, after combustion, of total sulphur (conductimetric method) and chlorine content (potentiometric or spectrophotometric method).*[1)]

LIQUEFIED PHENOL, *m*-CRESOL, CRESYLIC ACID, XYLENOLS

ISO/R 1903, *Determination of density at 20° C.*

LIQUEFIED PHENOL

ISO 1904, *Determination of phenols content – Bromination method.*

PHENOL

ISO/R 1905, *Test for impurities insoluble in water – Visual test.*

CRESYLIC ACID AND XYLENOLS

ISO/R 1906, *Determination of distillation range.*
ISO/R 1907, *Determination of residue on distillation.*
ISO/R 1908, *Test for absence of hydrogen sulphide.*
ISO/R 1909, *Measurement of colour.*
ISO/R 1910, *Determination of o-cresol content.*

CRESYLIC ACID

ISO/R 1911, *Determination of m-cresol content.*

NOTE – A laboratory sample of not less than 500 ml (for phenol and cresols) or 1 000 ml (for cresylic acid and xylenols) is necessary to carry out the whole series of tests described in these documents.

1) At present at the stage of Draft.

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