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INTERNATIONAL STANDARD ISO 1897/IV

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

Phenol, *o*-cresol, *m*-cresol, *p*-cresol, cresylic acid and xylenols for industrial use — Methods of test — Part IV : Visual test for impurities insoluble in sodium hydroxide solution (Excluding cresylic acid and xylenols)

Phénol, o-crésol, m-crésol, p-crésol, acide crésylique et xylénols à usage industriel — Méthodes d'essai — Partie IV : Essai visuel de contrôle des matières insolubles dans une solution d'hydroxyde de sodium (Acide crésylique et xylénols exclus)

First edition — 1977-04-15

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Descriptors : phenols, phenol, cresols, xylenol, chemical analysis, determination of content, water, volumetric analysis, Karl Fischer reagent.

Price based on 4 pages

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.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 1902-1971 and found it technically suitable for transformation. Number 1902, however, has been changed to 1897/IV. International Standard ISO 1897/IV therefore replaces ISO Recommendation R 1902-1971, to which it is technically identical.

ISO Recommendation R 1902 had been approved by the member bodies of the following countries :

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

The member body of the following country had expressed disapproval of the Recommendation on technical grounds :

Japan

The member body of the following country disapproved the transformation of the Recommendation into an International Standard :

Netherlands

Printed in Switzerland

Phenol, *o*-cresol, *m*-cresol, *p*-cresol, cresylic acid and xylenols for industrial use — Methods of test — Part IV : Visual test for impurities insoluble in sodium hydroxide solution (Excluding cresylic acid and xylenols)

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1897 specifies a visual test for impurities insoluble in sodium hydroxide solution and is applicable to phenol, *o*-cresol, *m*-cresol and *p*-cresol for industrial use.

NOTE — This is a simple empirical test of no great precision.

This document should be read in conjunction with part I (see the annex).

2 PRINCIPLE

Shaking a test portion with sodium hydroxide solution under specified conditions and assessment of any insoluble matter present in suspension in the mixture by comparison either with an agreed standard turbidimetric solution or with sodium hydroxide solution.

3 REAGENTS

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

3.1 Ethanol/glycerol mixture containing 2 volumes of 95 % (*V/V*) ethanol to 1 volume of glycerol.

3.2 Barium chloride dihydrate ($BaCl_2.2H_2O$).

3.3 Sodium hydroxide, 50 g/l solution.

3.4 Sulphuric acid, 0,005 M (= 0,01 N) standard volumetric solution.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Water bath, capable of being controlled at 20 ± 0,5 °C.

4.2 Two matched Nessler cylinders, each having a volume not greater than 150 ml and a length not less than 100 mm.

4.3 Black shield, with an opalescent glass sheet as base.

4.4 Electric lamp, equipped with a light blue "daylight" bulb, 60 W approximately.

4.5 Electromagnetic stirrer.

NOTE — The apparatus is shown assembled in the figure.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,1 g, approximately 10 g of the test sample in a weighed, ground glass-stoppered conical flask of 250 ml capacity.

5.2 Comparison with an agreed standard turbidimetric solution

5.2.1 *Preparation of test solution*

Add to the conical flask containing the test portion (5.1) a volume V_1 of the sodium hydroxide solution (3.3) calculated, in millilitres, from the formula

$$V_1 = m \times \frac{150}{10} = 15\,m$$

where m is the mass, in grams, of the test portion (5.1).

Shake the stoppered flask until solution is complete, place it in the water bath (4.1), controlled at 20 ± 0,5 °C, and leave it for 30 min.

5.2.2 *Preparation of standard turbidimetric solution*

Place 0,23 g of the barium chloride (3.2) in a second weighed 250 ml ground glass-stoppered conical flask; add a volume of water equal to ($V_1 - 30$) ml (see 5.2.1) and 30 ml of the ethanol/glycerol mixture (3.1). Mix the contents of the flask using the electromagnetic stirrer (4.5) until the barium chloride has completely dissolved. Add the volume agreed (between the parties) of the sulphuric acid solution (3.4) and stir for 1 min.

5.2.3 *Comparison*

Pour the standard turbidimetric solution (5.2.2) into one of the Nessler cylinders (4.2) and pour the test solution (5.2.1) into the other cylinder. Place the Nessler cylinders in the black shield (4.3) as shown in figure.

Compare the turbidity of the two solutions viewing vertically and with the electric lamp (4.4) switched on.

5.3 Comparison with sodium hydroxide solution

5.3.1 *Preparation of test solution*

Add to the conical flask containing the test portion (5.1) a volume V_2 of the sodium hydroxide solution (3.3) calculated, in millilitres, from the formula

$$V_2 = m \times R$$

where

m is the mass, in grams, of the test portion (5.1);

R is the ratio (agreed between the parties) of the volume of the sodium hydroxide solution (3.3) to the mass of the test portion.

Shake the stoppered flask until solution is complete.

5.3.2 *Comparison*

Pour the test solution (5.3.1) into one of the Nessler cylinders (4.2) and pour a volume, in millilitres, numerically equal to $(V_2 + m)$ (see 5.3.1) of the sodium hydroxide solution (3.3) into the other cylinder.

Place the two Nessler cylinders in the water bath (4.1), controlled at 20 ± 0,5 °C, and leave them for 30 min. Then transfer them to the black shield (4.3) as shown in the figure.

Compare the turbidity of the two solutions viewing vertically and with the electric lamp (4.4) swtiched on.

6 EXPRESSION OF RESULTS

6.1 Comparison with an agreed standard turbidimetric solution.

Report the test solution (5.2.1) as "clear" or report the turbidity produced as greater than, equal to, or less than that of the standard turbidimetric solution (5.2.2). State also the agreed volume of the sulphuric acid solution (3.4) used in preparing the standard turbidimetric solution.

6.2 Comparison with sodium hydroxide solution

Report the test solution (5.3.1) as "clear" or "turbid". State also the agreed ratio of the volume of the sodium hydroxide solution (3.3) to the mass of the test portion (5.1) (see clause 5.3.1).

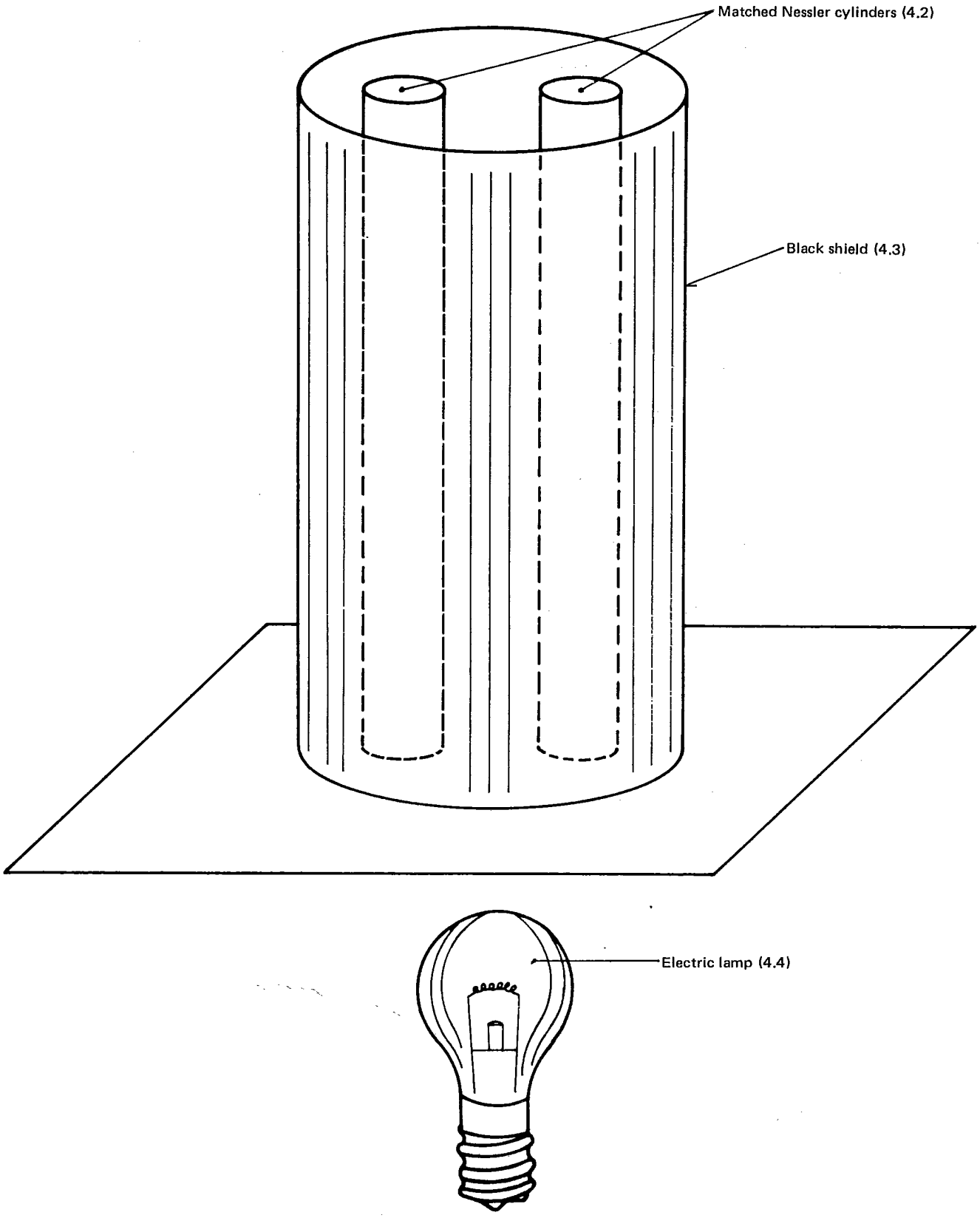

FIGURE — **Apparatus for use in the visual test for impurities insoluble in sodium hydroxide solution**

ANNEX

ISO PUBLICATIONS RELATING TO (A) PHENOL, (B) *o*-CRESOL, (C) *m*-CRESOL, (D) *p*-CRESOL, (E) CRESYLIC ACID, AND (F) XYLENOLS, FOR INDUSTRIAL USE

Applicability						
A[1]	B[2]	C	D[2]	E	F	ISO 1897/I — General.
A	B	C	D	E	F	ISO 1897/II — Determination of water — Dean and Stark method.
A	B	C	D	E	F	ISO 1897/III — Determination of neutral oils and pyridine bases.
A	B	C	D			ISO 1897/IV — Visual test for impurities insoluble in sodium hydroxide solution.
A						ISO 1897/V — Visual test for impurities insoluble in water.
				E	F	ISO 1897/VI — Test for absence of hydrogen sulphide.
				E	F	ISO 1897/VII — Measurement of colour.
				E	F	ISO 1897/VIII — Determination of *o*-cresol content.
				E		ISO 1897/IX — Determination of *m*-cresol content.
A	B	C	D			ISO/R 1900 — Determination of residue on evaporation.
A	B	C	D			ISO/R 1901 — Determination of crystallizing point.
A[3]						ISO 1904 — Determination of phenols content — Bromination method.
				E	F	ISO/R 1906 — Determination of distillation range.
				E	F	ISO/R 1907 — Determination of residue on distillation.
A	B	C	D			ISO 2208 — Determination of crystallizing point after drying with a molecular sieve.

1) In the case of phenol, the determination of density at 20 °C specified in ISO 1897/I is applicable only to liquefied phenol.

2) The determination of density at 20 °C specified in ISO 1897/I is not applicable to these products.

3) Applicable only to liquefied phenol.