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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1905

PHENOL FOR INDUSTRIAL USE

TEST FOR IMPURITIES INSOLUBLE IN WATER

VISUAL TEST

1st EDITION

May 1971

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 1905, *Phenol for industrial use – Test for impurities insoluble in water – Visual test*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1905, which was circulated to all the ISO Member Bodies for enquiry in November 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1905 May 1971

PHENOL FOR INDUSTRIAL USE

TEST FOR IMPURITIES INSOLUBLE IN WATER

VISUAL TEST

WARNING. These materials 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 ISO Recommendation describes a visual test for impurities insoluble in water and is applicable to phenol for industrial use.

This is a simple empirical test of no great precision.

2. SAMPLING

Apply the principles given in ISO Recommendation R . . . *. The following principles should 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

Shaking with water 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 water.

* Sampling of chemical products will form the subject of a future ISO Recommendation.

4. REAGENTS

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

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

4.2 *Barium chloride*, crystalline ($BaCl_2.2H_2O$).

4.3 *Sulphuric acid*, 0.01 N standard volumetric solution.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *Water bath*, capable of being controlled at 20 ± 0.5 °C.

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

5.3 *Black shield*, with an opalescent glass sheet as base.

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

5.5 *Magnetic stirrer*

NOTE. – The apparatus is shown assembled in the Figure, page 6.

6. PROCEDURE

6.1 Test portion

Weigh, to the nearest 0.1 g, approximately 10 g of the laboratory sample in a weighed ground glass-stoppered conical flask of 250 ml capacity.

6.2 Comparison with an agreed turbidimetric solution

6.2.1 *Preparation of sample solution*. Add to the conical flask containing the test portion (6.1) a volume V of water at a temperature between 20 and 30 °C calculated from the following formula :

$$V = m \times \frac{150}{10}$$

where m is the mass, in grammes, of the test portion.

Shake the stoppered flask until solution is complete, and place it in the water bath (5.1), maintained at 20 ± 0.5 °C, for 30 minutes.

6.2.2 *Preparation of standard turbidimetric matching solution*. Place 0.23 g of barium chloride (4.2) in a weighed 250 ml ground glass-stoppered conical flask; add a volume of water equal to (V – 30) ml (see clause 6.2.1) and 30 ml of the ethanol/glycerol mixture (4.1). Mix the contents of the flask using a magnetic stirrer (5.5) until the barium chloride has completely dissolved. Add the volume agreed (between the parties) of sulphuric acid solution (4.3) and stir for 1 minute.

6.2.3 *Comparison*. Pour the standard turbidimetric matching solution (6.2.2) into one of the Nessler cylinders (5.2) and pour the sample solution (6.2.1) into the other one. Place the Nessler cylinders in the black shield (5.3) as shown in the Figure, page 6.

Compare the turbidity of the two solutions viewing vertically and with the electric lamp (5.4) switched on.

6.3 Comparison with water

6.3.1 *Preparation of sample solution*. Add to the conical flask containing the test portion (6.1) a volume V of water at a temperature between 20 and 30 °C, caulated from the following formula :

$$V = m \times R$$

where

m is the mass, in grammes, of the test portion;

R is the ratio (agreed between the parties) of water to the mass of the test portion.

6.3.2 *Comparison*. Shake the stoppered flask until solution is complete. Pour the sample solution (6.3.1) into one of the Nessler cylinders (5.2) and pour a volume, in millilitres, numerically equal to $(V + m)$ (see clause 6.3.1) of water into the other one.

Place the two Nessler cylinders in the water bath (5.1), maintained at 20 ± 0.5 °C for 30 minutes. Then transfer them to the black shield (5.3) as shown in the Figure, page 6.

Compare the turbidity of the two solutions viewing vertically and with the electric lamp (5.4) switched on.

7. EXPRESSION OF RESULTS

7.1 Comparison with agreed turbidimetric solution

Report the sample solution (6.2.1) as "clear" or report the turbidity produced as greater than, equal to, or less than that of the standard turbidimetric matching solution (6.2.2). State also the agreed volume of sulphuric acid solution (4.3) used in preparing the standard turbidimetric matching solution (6.2.2).

7.2 Comparison with water

Report the sample solution as "clear" or "turbid". State also the agreed ratio of water to the mass of the test portion (see clause 6.3.1).

8. TEST REPORT

The test report should give 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 ISO Recommendation or regarded as optional.

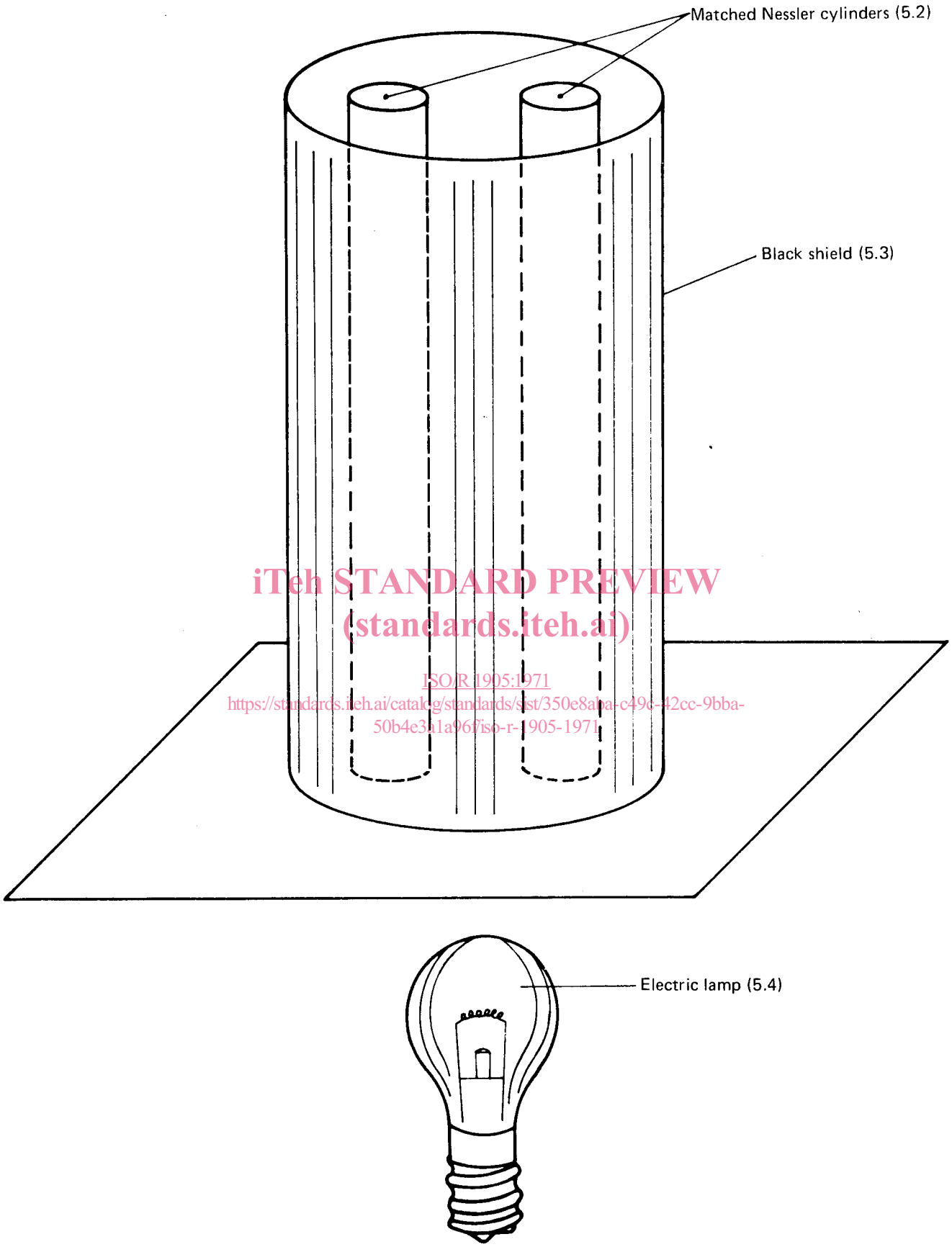

FIGURE – **Apparatus for use in the visual test for impurities insoluble in water**

ANNEX

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

The complete list of the Recommendations 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/R 2208, *Determination of crystallizing point after drying with a molecular sieve.**
ISO/R 1902, *Test for impurities insoluble in sodium hydroxide solution – Visual test.*
ISO/R 2273, *Determination, after combustion, of total sulphur (conductimetric method) and chlorine content (potentiometric or spectrophotometric method).**

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

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

PHENOL

ISO/R 1904, *Determination of phenol content – Bromination method.**
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 1000 ml (for cresylic acid and xylenols) is necessary to carry out the whole series of tests described in these documents.

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* At present at the stage of Draft ISO Recommendation.

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