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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1911

CRESYLIC ACID FOR INDUSTRIAL USE

DETERMINATION OF *m*-CRESOL CONTENT

1st EDITION

May 1971

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BRIEF HISTORY

The ISO Recommendation R 1911, *Cresylic acid for industrial use – Determination of* m-*cresol content,* 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. 1911, 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	Israel	Switzerland
Belgium	Italy	Thailand
Czechoslovakia	New Zealand	Turkey
France	Poland	U.A.R.
Germany	Portugal	United Kingdom
Greece	Romania	U.S.S.R.
Hungary	South Africa, Rep. of	
India	Spain	

The following Member Bodies opposed the approval of the Draft :

Japan
Netherlands

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 1911 May 1971

CRESYLIC ACID

FOR INDUSTRIAL USE

DETERMINATION OF *m*-CRESOL CONTENT

WARNING. This material burns 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.

1. SCOPE

This ISO Recommendation describes a method for the determination of *m*-cresol content of cresylic acid of high *m*-cresol content for industrial use.

2. FIELD OF APPLICATION

This method is applicable to cresylic acid containing 35 to 60 % (m/m) of *m*-cresol not more than 5 % (m/m) of phenol, and not more than 35 % (m/m) of *o*-cresol determined by the procedure described in ISO Recommendation R 1910, *Cresylic acid and xylenols for industrial use – Determination of o-cresol content*, of which not more than 5 % (V/V) distils below 190 °C and not more than 95 % (V/V) distils below 208 °C when tested by the procedure described in ISO Recommendation R 1906, *Cresylic acid and xylenols for industrial use – Determination of distillation range.*

3. 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.

4. PRINCIPLE

Formation of a complex between *m*-cresol and urea when excess urea is added to the dehydrated sample and determination of the crystallizing point of the complex.

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

5. REAGENTS

5.1 *Urea,* of analytical reagent quality.

Keep this reagent in an open bottle in a desiccator containing silica gel.

5.2 m-*Cresol/urea complex*

Heat and stir 11 g of pure *m*-cresol and 3 g of urea (5.1) in a small beaker until a clear melt is obtained; allow to cool, stirring until semi-solid (this may take some time). Leave the mixture to set. When cold add a little light petroleum (boiling range 40 to 60 °C), transfer to a sintered glass funnel, wash thoroughly with more light petroleum and dry by suction.

6. APPARATUS

Ordinary laboratory apparatus, and

6.1 *Crystallizing point apparatus* (see Figure 1) consisting of a glass test tube 150 mm × 25 mm nominal size placed inside a 160 mm × 38 mm test tube.

The latter tube is flanged so that it may be supported centrally by a metal cover plate, in a 1000 ml tall-form beaker filled with water to within 20 mm of the top.

The wider tube is weighted with lead shot or similar material and the inner tube is closed by means of a cork which carries a glass stirrer and through its centre a certified thermometer (6.2 or 6.3). The stirrer has a loop of outside diameter 18 mm, to surround the thermometer. The thermometer is so fixed in the cork that the bottom of the bulb is about 15 mm from the bottom of the inner tube. A thermometer (6.4) for the water bath (6.5) passes through a hole in the cover plate and is held by a rubber ring.

6.2 *Thermometer,* of the mercury-in-glass type, graduated for use at 100 mm immersion and covering the range 0 to 100 °C, certified for accuracy, graduated at intervals of 0.5 °C and of known scale error, not greater than ± 0.3 °C.

6.3 *Thermometer,* of the mercury-in-glass type, graduated for use at 100 mm immersion and covering the range 15.5 to 45 °C or 39.5 to 70.5 °C, certified for accuracy, graduated at intervals of 0.1 °C and of known scale error, not greater than ± 0.4 °C.

6.4 *Thermometer,* general purpose, graduated for use at 75 mm immersion and covering the range 0 to 50 °C, graduated at intervals of 1 °C and of known scale error, not greater than ± 0.5 °C.

6.5 *Water bath*

6.6 *Flask,* capacity 100 ml, short-necked, round-bottomed, fitted with a 24/29 conical ground glass socket (see ISO Recommendation R 383, *Interchangeable conical ground glass joints*).

6.7 *Air condenser,* as shown in Figure 2 with a conical ground glass joint to fit the flask (6.6).

6.8 *Tube,* having a shank length of approximately 115 mm and minimum bore of 16.5 mm, provided at one end with a conical ground glass joint to fit the flask (6.6). The tube is fitted with anhydrous calcium chloride, held in place by cotton wool plugs at the ends.

7. PROCEDURE

7.1 Dehydration of the sample

Dry 100 ml of the laboratory sample by slowly heating it in the flask (6.6) fitted with the air condenser (6.7) until 2 ml of distillate has been collected in a 10 ml measuring cylinder. (The purpose of this distillation is to remove all traces of water without materially altering the composition of the sample.) Replace the condenser by the tube (6.8) and allow to cool, before taking the test portion.

7.2 Determination of crystallizing point

7.2.1 *Test portion.* Weigh 20.0 g of the dehydrated sample (7.1) into the inner tube of the crystallizing point apparatus (6.1) and add 4.0 g of urea (5.1). Insert the cork carrying the thermometer (6.2) and stirrer.

NOTE. – The stirrer may be omitted and the stirring carried out by hand using the thermometer but care should be taken that the thermometer does not touch the walls of the tube.

7.2.2 *Determination.* Heat the tube with a Bunsen flame and stir the mixture until the thermometer indicates a temperature between 80 and 85 °C and a clear melt is obtained. Stir steadily while allowing the tube to cool in air. When crystals first begin to form, add a few crystals of the *m*-cresol/urea complex (5.2), continue cooling and stirring and note the approximate temperature at which silky crystals of the complex separate.

Warm the water in the crystallizing point apparatus (6.1) and maintain it at a temperature 4 to 5 °C below the approximate crystallizing point. Replace the thermometer (6.2) by the thermometer (6.3).

Warm the contents of the inner tube by means of a small Bunsen flame to a temperature 4 to 5 °C above the approximate crystallizing point. Place the tube in its jacket. Stir *gently* and continuously and record thermometer readings at 30-second intervals. When the temperature drops to the approximate crystallizing point, add a few crystals of the complex (5.2). The temperature should fall at a fairly steady rate to minimum value, then rise and pass through a maximum value before falling again at a steady rate. If no such supercooling occurs, repeat the test using a fresh portion of the dehydrated sample (7.1).

The crystallizing point corresponds to the maximum value referred to in the preceding paragraph, corrected as described in clause 7.2.3.

7.2.3 *Temperature correction.* Obtain the corrected temperature referred to above by applying the correction for thermometer error.

8. EXPRESSION OF RESULTS

8.1 Method of calculation

Ascertain the percentage by mass of *m*-cresol in the dry sample by reference to the Table.

The *m*-cresol content is given, as a percentage by mass, by the following formula :

$$\frac{D \times (100 - W)}{100}$$

where

D is the *m*-cresol content of the dry sample, expressed as a percentage by mass and ascertained by reference to the Table;

W is the water content of the test portion, expressed as a percentage by mass and determined by one of the procedures described in ISO Recommendation R 1897, *Phenol,* o-*cresol,* m-*cresol,* p-*cresol, cresylic acid and xylenols for industrial use – Determination of water by the Karl Fischer method* and ISO Recommendation R 1898, *Phenol,* o-*cresol,* m-*cresol,* p-*cresol, cresylic acid and xylenols for industrial use – Determination of water by the Dean and Stark method.*

TABLE – Relation between crystallizing point and *m*-cresol content (percentage by mass) of *m*-cresol/urea mixtures

Crystallizing point °C	0.0	0.2	0.4	0.6	0.8
27	34.8	35.0	35.1	35.3	35.4
28	35.6	35.8	35.9	36.1	36.2
29	36.4	36.6	36.7	36.9	37.0
30	37.2	37.4	37.6	37.7	37.9
31	38.1	38.3	38.5	38.6	38.8
32	39.0	39.2	39.4	39.5	39.7
33	39.9	40.1	40.3	40.5	40.7
34	40.9	41.1	41.3	41.5	41.7
35	41.9	42.1	42.3	42.5	42.7
36	42.9	43.1	43.3	43.6	43.8
37	44.0	44.2	44.4	44.7	44.9
38	45.1	45.3	45.5	45.8	46.0
39	46.2	46.4	46.7	46.9	47.2
40	47.4	47.7	47.9	48.2	48.4
41	48.7	49.0	49.3	49.5	49.8
42	50.1	50.4	50.7	51.0	51.3
43	51.6	51.9	52.2	52.5	52.8
44	53.1	53.4	53.7	54.1	54.4
45	54.7	55.0	55.4	55.7	56.1
46	56.4	56.7	57.1	57.4	57.8
47	58.1	58.5	58.8	59.2	59.5
48	59.9	60.3	—	—	—

8.2 **Repeatability and reproducibility**

8.2.1 *Repeatability*. Duplicate results by the same operator should be considered suspect if they differ by more then the value in the Table given below.

8.2.2 *Reproducibility*. Single results submitted by each of two operators should be considered suspect if they differ by more than the value in the Table given below.

m-cresol content, % (m/m)	35	40	45	50	55
Repeatability	0.5	0.6	0.7	0.9	1.1
Reproducibility	0.7	0.9	1.1	1.4	1.6

The repeatability and the reproducibility in terms of the crystallizing point of the *m*-cresol/urea complex is constant over the range covered by the test.

9. 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.

Dimensions in millimetres

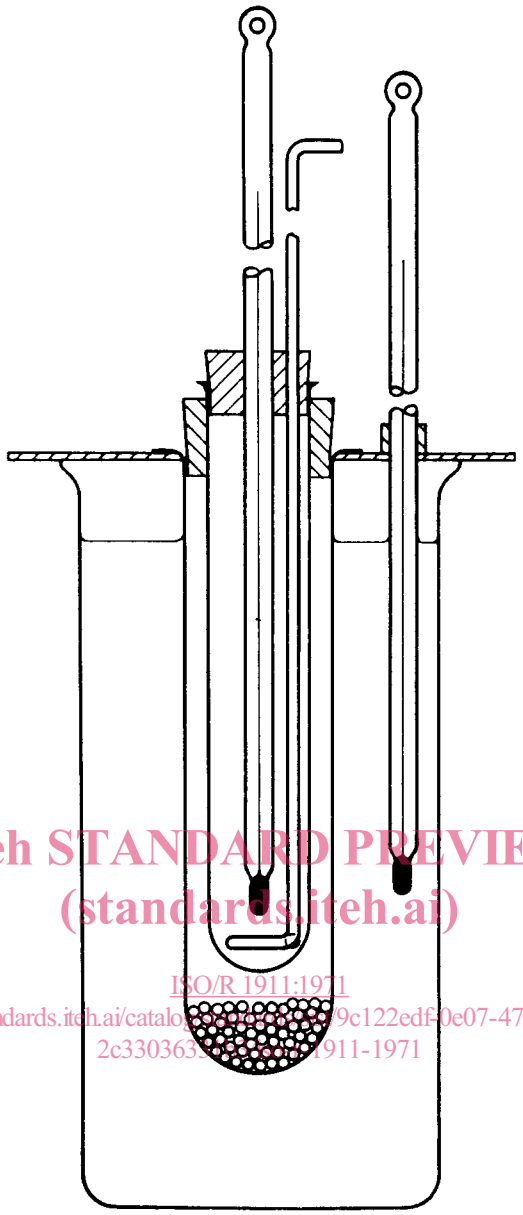

FIG. 1 – **Apparatus for the determination of the crystallizing point** (6.1)

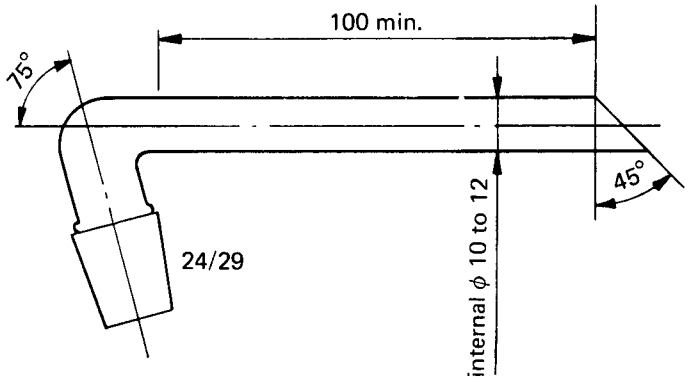

FIG. 2 – **Air condenser** (6.7)

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.**

LIQUEFIED 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 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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