UDC 661 : 547.562/563

Ref. No. : ISO/R 1910-1971 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1910

1597/VIII

CRESYLIC ACID AND XYLENOLS FOR INDUSTRIAL USE

DETERMINATION OF *m*-CRESOL CONTENT

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 1910, *Cresylic acid and xylenols for industrial use – Determination of* o-*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. 1910, 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	South Africa, Rep. of
Belgium	Israel	Spain
Chile	Italy	Switzerland
Czechoslovakia	Japan	Thailand
France	New Zealand	Turkey
Germany	Poland	U.A.R.
Greece	Portugal	United Kingdom
Hungary	Romania	U.S.S.R.

The following Member Body opposed the approval of the Draft :

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 1910 | May 1971

CRESYLIC ACID AND XYLENOLS

FOR INDUSTRIAL USE

DETERMINATION OF *o*-CRESOL CONTENT

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.

1. SCOPE AND FIELD OF APPLICATION

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

The method is primarily intended for samples containing 40 % or more of *o*-cresol but has been extended to lower ranges by fortifying the material with pure *o*-cresol.

NOTE. This method is the best one available at the present time; a gas chromatographic method can be used for *o*-cresol contents less than 40 % but the details of this have not yet been standardized.

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

Formation of a complex between *o*-cresol and cineole when excess cineole is added to the dehydrated sample and determination of the crystallizing point of the complex. Calculation of the *o*-cresol content from the water content of the sample and the crystallizing point of the complex.

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

4. REAGENTS

4.1 *Cineole,* having a crystallizing point not lower than 1.2 °C when determined in the apparatus shown in Figure 1, with suitable adjustment of the temperature of the bath (5.5). It should be kept quite dry and away from strong light, in amber-coloured bottles containing a little anhydrous calcium chloride. The crystallizing point should be checked before the material is used; if it is found to be lower than 1.2 °C, heat sufficient cineole for the test in a tube until the vapour ring reaches the top of the tube. Again determine the crystallizing point; if it is still lower than 1.2 °C the cineole should be discarded. In some circumstances cineole having a crystallizing point not lower than 1.35 °C is necessary (see Table, page 9).

4.2 o-*Cresol,* pure and dehydrated, having a crystallizing point not lower than 30.6 °C.

5. APPARATUS

Ordinary laboratory apparatus, and

5.1 *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 (5.2 or 5.3). 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 (5.4) for the water bath (5.5) passes through a hole in the cover plate and is held by a rubber ring.

5.2 *Thermometer* for testing the cineole (4.1), of the mercury-in-glass type, graduated for use at 100 mm immersion and covering the range − 10 to + 20 °C, certified for accuracy, graduated at intervals of 0.1 °C, and of known scale error, not greater than ± 0.2 °C.

5.3 *Thermometer* for use in determining the crystallizing point of the *o*-cresol/cineole complex, 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.

5.4 *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.

5.5 *Water bath*

5.6 *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*).

5.7 *Air condenser,* as shown in Figure 2, with a conical ground glass joint to fit the flask (5.6).

5.8 *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 (5.6). The tube is filled with anhydrous calcium chloride, which is held in place by cotton wool plugs at the ends.

6. PROCEDURE

6.1 Dehydration of the sample

Dry 50 ml of the laboratory sample by slowly heating it in the flask (5.6) fitted with the air condenser (5.7) until 1 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 (5.8) and allow to cool before taking the test portion.

6.2 Determination of crystallizing point

6.2.1 *Test portion.* Remove the inner tube of the assembled crystallizing point apparatus (5.1) from its jacket and weigh into the tube 8.40 g of the dry sample (6.1) and 12.00 g of cineole (4.1). If the *o*-cresol content of the sample is known to be less than 40 %, weigh into the tube 4.20 g of dry sample (6.1), 4.20 g of *o*-cresol (4.2) and 12.00 g of cineole (4.1).

NOTES

1. 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.

2. A molecular sieve may be added to the test portion to take up any atmospheric moisture that might contaminate the sample after dehydration.

6.2.2 *Determination.* Stir the mixture until homogeneous, avoiding exposure of the material to air for longer than necessary. Heat the mixture gently until it is completely fluid and then cool rapidly to determine the approximate crystallizing point. Warm the tube in the water bath (5.5) at a temperature about 5 °C above this point, so that the crystals melt, except for a trace necessary for seeding.

Replace the inner tube in its jacket with the water in the apparatus maintained at a temperature between 6 and 8 °C below the expected crystallizing point. Stir the sample *gently* and continuously and record thermometer readings at 30-second intervals.

The crystallizing point corresponds to the highest of the first five consecutive readings (corrected as in clause 6.2.3) during which the temperature remains constant within 0.05 °C.

If supercooling occurs, as shown by a rise in temperature, observe the constant temperature after the rise. A temperature rise of 1 °C is the maximum allowable. If a constant temperature is not obtained over the first five readings after the rise in temperature, record six readings commencing with the point at which the maximum temperature is first attained.

Plot the complete cooling curve of temperature against time and draw a straight line to lie evenly between the first and second and between the fifth and sixth points mentioned above. Extend this line to meet the section of the cooling curve before the temperature rise.

Report the temperature corresponding to the point of intersection (corrected as in clause 6.2.3) as the crystallizing point.

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

7. EXPRESSION OF RESULTS

Ascertain, by reference to the Table, the percentage by mass of *o*-cresol in the dry sample or in the mixture of dry sample and pure *o*-cresol.

7.1 If no pure *o*-cresol (4.2) was added before determining the crystallizing point, *o*-cresol content is given, as a percentage by mass, by the following formula :

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

where

A is the *o*-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 sample, 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.*

7.2 If pure *o*-cresol (4.2) was added before determining the crystallizing point, *o*-cresol content is given, as a percentage by mass, by the following formula :

$$\frac{(2B - C) \times (100 - W)}{100}$$

where

B is the *o*-cresol content of the mixture of dry sample and pure *o*-cresol (4.2), expressed as a percentage by mass and ascertained by reference to the Table;

C is the *o*-cresol content, expressed as a percentage by mass, of the pure *o*-cresol (4.2) added;

W is the water content of the test portion, expressed as a percentage by mass and determined by one of the procedures described in the ISO Recommendations referred to in clause 7.1 above.

NOTE. – The purity of the added *o*-cresol may be ascertained from the following :

Crystallizing point °C	Purity %
31.0	100
30.8	99.5
30.6	99.0

TABLE Relation between crystallizing point and *o*-cresol content (percentage by mass) of *o*-cresol/cineole mixtures

Crystallizing point °C	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
31	40.0	40.2	40.3	40.5	40.6	40.8	40.9	41.1	41.2	41.4
32	41.6	41.7	41.9	42.0	42.2	42.4	42.5	42.7	42.9	43.0
33	43.2	43.4	43.5	43.7	43.9	44.0	44.2	44.4	44.5	44.7
34	44.9	45.0	45.2	45.4	45.6	45.8	46.0	46.2	46.3	46.5
35	46.7	46.9	47.1	47.3	47.5	47.7	47.9	48.0	48.3	48.4
36	48.6	48.8	49.0	49.2	49.4	49.6	49.8	50.0	50.2	50.4
37	50.6	50.8	51.0	51.2	51.4	51.6	51.8	52.0	52.2	52.4
38	52.6	52.8	53.0	53.2	53.4	53.6	53.8	54.0	54.2	54.5
39	54.7	54.9	55.1	55.3	55.5	55.7	56.0	56.2	56.4	56.6
40	56.8	57.0	57.2	57.4	57.6	57.8	58.0	58.2	58.4	58.6
41	58.8	59.0	59.3	59.5	59.7	60.0	60.2	60.4	60.6	60.8
42	61.0	61.3	61.5	61.7	62.0	62.2	62.4	62.6	62.9	63.1
43	63.3	63.6	63.8	64.0	64.3	64.5	64.8	65.0	65.2	65.5
44	65.8	66.0	66.2	66.4	66.7	66.9	67.2	67.4	67.6	67.8
45	68.0	68.3	68.5	68.8	69.0	69.3	69.5	69.8	70.0	70.3
46	70.5	70.8	71.0	71.3	71.6	71.8	72.1	72.4	72.6	72.9
47	73.1	73.4	73.7	73.9	74.2	74.5	74.7	75.0	75.2	75.5
48	75.7	76.0	76.3	76.5	76.8	77.0	77.3	77.6	77.8	78.1
49	78.3	78.6	78.9	79.2	79.4	79.7	80.0	80.2	80.5	80.8
50	81.1	81.4	81.7	82.0	82.3	82.6	82.8	83.2	83.4	83.7
51	84.0	84.3	84.6	84.9	85.2	85.5	85.8	86.1	86.4	86.7
52	87.0	87.3	87.6	87.9	88.2	88.5	88.8	89.1	89.4	89.7
53	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7
54	92.9	93.2	93.5	93.8	94.1	94.4	94.7	95.0	95.3	95.0
55	95.9	96.2	96.5	96.8	97.1	97.4	97.7	98.0	98.2	98.5
56	98.8	99.1	99.4	99.7	100.0	–	–	–	–	–

NOTE. – The portion of the Table above the double horizontal line is applicable if the cineole used has a crystallizing point above 1.2 °C. The portion of the Table below the double horizontal line is applicable only if the cineole used has a crystallizing point not lower than 1.35 °C.

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.