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Phenol, *o*-cresol, *m*-cresol, *p*-cresol, cresylic acid and xlenols for industrial use — Methods of test — Part VIII : Determination of *o*-cresol content (Cresylic acid and xlenols only)

*Phénol, o-crésol, m-crésol, p-crésol, acide crésylique et xylénols à usage industriel — Méthodes d'essai —
Partie VIII : Détermination de la teneur en o-crésol (Acide crésylique et xylénols uniquement)*

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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.

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 1910-1971 and found it technically suitable for transformation. Number 1910, however, has been changed to 1897/VIII. International Standard ISO 1897/VIII therefore replaces ISO Recommendation R 1910-1971, to which it is technically identical.

ISO Recommendation R 1910 had been approved by the member bodies of the following countries :

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

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

Netherlands

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

Netherlands

Phenol, *o*-cresol, *m*-cresol, *p*-cresol, cresylic acid and xylenols for industrial use — Methods of test —

Part VIII : Determination of *o*-cresol content (Cresylic acid and xylenols only)

1 SCOPE AND FIELD OF APPLICATION

This part of ISO 1897 specifies a method for the determination of the *o*-cresol content of cresylic acid of high *o*-cresol content, cresylic acid of high *m*-cresol content and xylenols for industrial use.

The method was primarily intended for samples containing 40 % (*m/m*) or more of *o*-cresol but has been extended to lower ranges by enriching the material with pure *o*-cresol.

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

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

2 PRINCIPLE

Dehydration of a quantity of the test sample. Formation of a complex between *o*-cresol and cineole by addition of excess cineole to a test portion of the dehydrated sample. Determination of the crystallizing point of the complex. Calculation of the *o*-cresol content from the water content of the test sample and the crystallizing point of the complex.

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade.

3.1 Cineole ($C_{10}H_{18}O$), having a crystallizing point not lower than $1,2^{\circ}\text{C}$ when determined in the apparatus (4.1), with suitable adjustment of the temperature of the bath (4.5).

It shall be kept completely dry and away from strong light, in amber-coloured bottles containing a little anhydrous calcium chloride. The crystallizing point shall be checked before the material is used; if it is found to be lower than $1,2^{\circ}\text{C}$, 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 $1,2^{\circ}\text{C}$ the cineole shall be discarded. In some circumstances cineole having a crystallizing point not lower than $1,35^{\circ}\text{C}$ is necessary (see the table).

3.2 *o*-Cresol ($\text{CH}_3\text{—C}_6\text{H}_4\text{—OH}$), dehydrated, having a crystallizing point not lower than $30,6^{\circ}\text{C}$.

4 APPARATUS

Ordinary laboratory apparatus, and

4.1 Crystallizing point apparatus (see figure 1) consisting of a glass test tube, nominal size 150 mm $\times$ 25 mm, placed inside a 160 mm $\times$ 38 mm test tube.

The latter tube is flanged so that it may be supported centrally by a metal cover plate, in a 1 000 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 a centrally mounted certified thermometer (4.2 or 4.3). The stirrer has a loop of outside diameter approximately 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 (4.4) for the water bath (4.5) passes through a hole in the cover plate and is held by a rubber ring.

4.2 Thermometer for testing the cineole (3.1), of the mercury-in-glass type, graduated for use at 100 mm immersion and covering the range -10 to $+20^{\circ}\text{C}$, certified for accuracy, graduated at intervals of $0,1^{\circ}\text{C}$ and of known scale error, not greater than $\pm 0,2^{\circ}\text{C}$.

4.3 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 $70,5^{\circ}\text{C}$, certified for accuracy, graduated at intervals of $0,1^{\circ}\text{C}$ and of known scale error, not greater than $\pm 0,4^{\circ}\text{C}$.

4.4 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 $\pm 0,5^{\circ}\text{C}$.

4.5 Water bath with thermostat.

4.6 Flask, capacity 100 ml, short-necked, round-bottomed, fitted with a 24/29 conical ground glass socket complying with ISO 383.

4.7 Air condenser, as shown in figure 2, with a conical ground glass joint to fit the flask (4.6).

4.8 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 (4.6). The tube is filled with anhydrous calcium chloride, which is held in place by cotton wool plugs at the ends.

5 PROCEDURE

5.1 Dehydration of the test sample

Dry 50 ml of the test sample by slowly heating it in the flask (4.6) fitted with the air condenser (4.7) 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 (4.8) and allow to cool before taking the test portion (5.2.1).

5.2 Determination of crystallizing point

5.2.1 Test portion

Remove the inner tube of the assembled crystallizing point apparatus (4.1) from its jacket and weigh accurately into the tube 8,40 g of the dry sample (5.1) and 12,00 g of the cineole (3.1). If the *o*-cresol content of the sample is known to be less than 40 % (*m/m*), weigh into the tube 4,20 g of the dry sample (5.1), 4,20 g of the *o*-cresol (3.2) and 12,00 g of the cineole (3.1).

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.

5.2.2 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 (4.5) 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 intervals of 30 s.

The crystallizing point corresponds to the highest of the first five consecutive readings (corrected as in 5.2.3) 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 graph 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 graph before the temperature rise.

Report the temperature corresponding to the point of intersection (corrected as in 5.2.3) as the crystallizing point.

5.2.3 Temperature correction

Obtain the corrected temperature referred to above by applying the correction for thermometer error (see 4.3).

6 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 *o*-cresol (3.2).

6.1 If no *o*-cresol (3.2) was added before determining the crystallizing point, the *o*-cresol content, expressed as a percentage by mass, is given by the formula

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

where

A is the *o*-cresol content of the dry sample (5.1), expressed as a percentage by mass and ascertained by reference to the table on page 3;

W is the water content of the test sample, expressed as a percentage by mass and determined by one of the methods specified in parts I and II of ISO 1897 (see the annex).

6.2 If *o*-cresol (3.2) was added before determining the crystallizing point, the *o*-cresol content, expressed as a percentage by mass, is given by the formula

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

where

B is the *o*-cresol content of the mixture of dry sample (5.1) and *o*-cresol (3.2), expressed as a percentage by mass and ascertained by reference to the table on page 3;

C is the *o*-cresol content, expressed as a percentage by mass, of the *o*-cresol (3.2) added;

W has the same meaning as in 6.1.

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

Crystallizing point	Purity
°C	%
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.

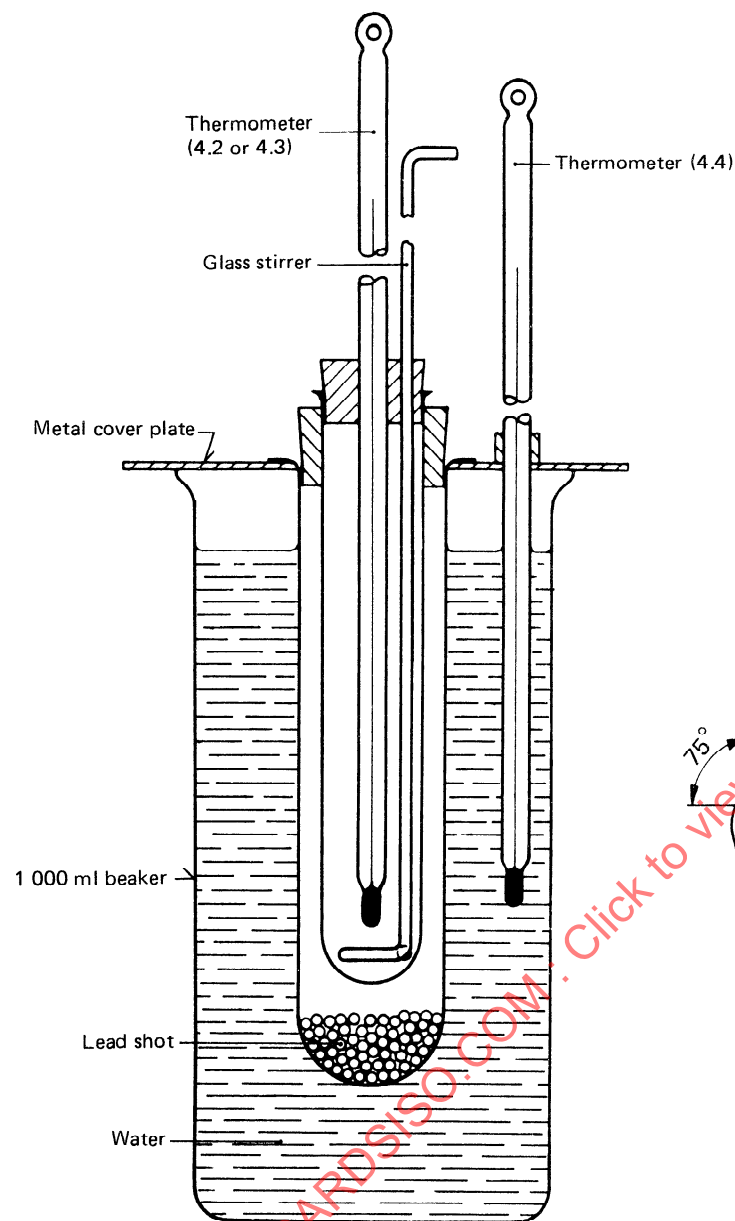


FIGURE 1 — Apparatus for the determination of the crystallizing point (4.1)

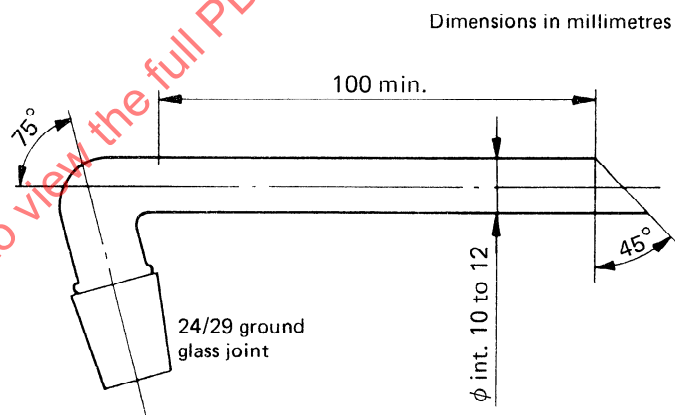


FIGURE 2 — Air condenser (4.7)