



ISO 1897 PT*IX 77 ■ 4851903 0091752 9 ■

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 — P - IX : Determination of *m*-cresol content (Cresylic acid only)

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

First edition — 1977-04-15

UDC 661 : 547.562/563 : 543.8

Ref. No. ISO 1897/IX-1977 (E)

Descriptors : phenols, phenol, cresols, xyleneol, 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 1911-1971 and found it technically suitable for transformation. Number 1911, however, has been changed to 1897/IX. International Standard ISO 1897/IX therefore replaces ISO Recommendation R 1911-1971, to which it is technically identical.

ISO Recommendation R 1911 had been approved by the member bodies of the following countries :

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

The member bodies of the following countries had expressed disapproval of the Recommendation on technical grounds :

Japan
Netherlands

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

Netherlands

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Printed in Switzerland

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

Part IX : Determination of *m*-cresol content (Cresylic acid only)

1 SCOPE AND FIELD OF APPLICATION

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

The 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 method specified in part VIII (see the annex), and 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 method specified in ISO/R 1906 (see the annex).

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 *m*-cresol and urea by addition of excess urea to a test portion of the dehydrated sample. Determination of the crystallizing point of the complex. Calculation of the *m*-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 Urea (NH₂ – CO – NH₂).

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

3.2 *m*-Cresol/urea complex.

Heat and stir 11 g of *m*-cresol and 3 g of urea (3.1) 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 funnel fitted with a sintered glass filter, wash thoroughly with more light petroleum and dry by suction.

4 APPARATUS

Ordinary laboratory apparatus, and

4.1 Crystallizing point apparatus (see figure 1) consisting of a glass test tube, nominal size 150 mm × 25 mm, 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 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, 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.

4.3 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.

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 ± 0,5 °C.

4.5 Water bath fitted with a thermoregulator.

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 100 ml of the test sample by slowly heating it in the flask (4.6) fitted with the air condenser (4.7) 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 (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 20,0 g of the dehydrated sample (5.1) and 4,0 g of the urea (3.1). Insert the cork carrying the thermometer (4.2) 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.

5.2.2 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 (3.2), 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 (4.1) and maintain it at a temperature 4 to 5° C below the approximate crystallizing point. Replace the thermometer (4.2) by the thermometer (4.3).

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 intervals of 30 s. When the temperature drops to the approximate crystallizing point, add a few crystals of the *m*-cresol/urea complex (3.2). The temperature should fall at a fairly steady rate to a 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 test portion (5.2.1).

The crystallizing point corresponds to the maximum value referred to in the preceding paragraph, corrected as specified in 5.2.3.

5.2.3 Temperature correction

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

6 EXPRESSION OF RESULTS

6.1 Method of calculation

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

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

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

where

D is the *m*-cresol content of the dry sample (5.1), expressed as a percentage by mass and ascertained by reference to the table below;

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).

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	--	--	--

6.2 Repeatability and reproducibility

6.2.1 Repeatability

Duplicate results by the same operator should be considered suspect if they differ by more than the appropriate value in the following table.

6.2.2 Reproducibility

Single results submitted by each of two operators should be considered suspect if they differ by more than the appropriate value in the following table.

<i>m</i> -Cresol content, % (<i>m/m</i>)	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.

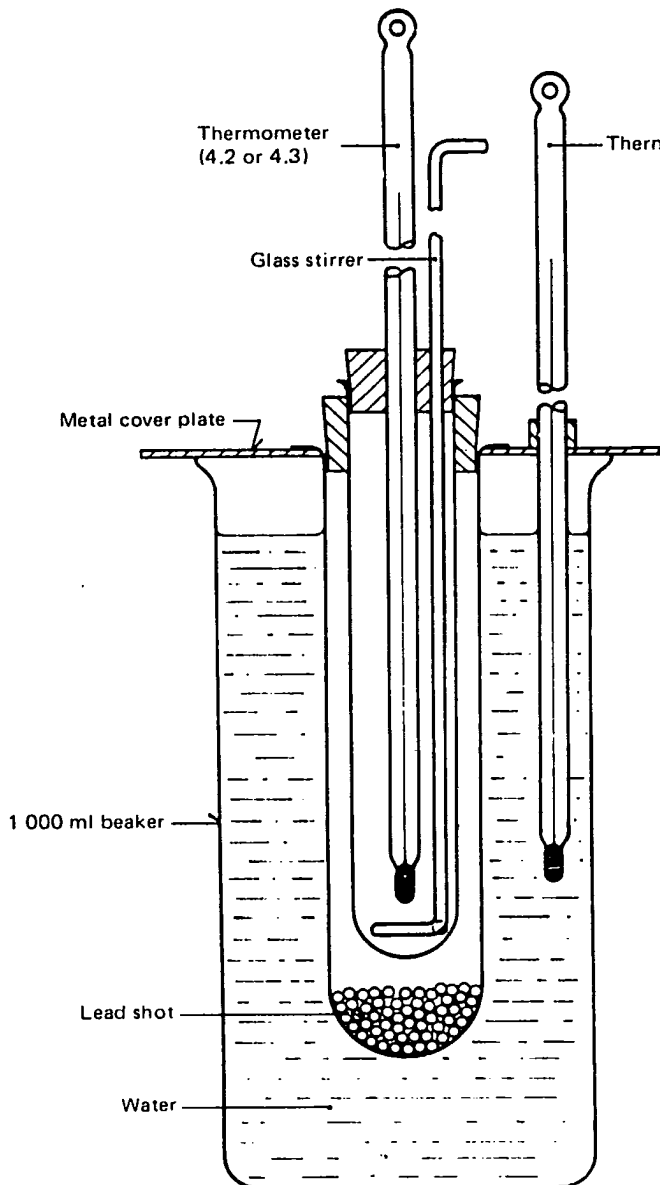


FIGURE 1 – Apparatus for the determination of the crystallizing point (4.1)

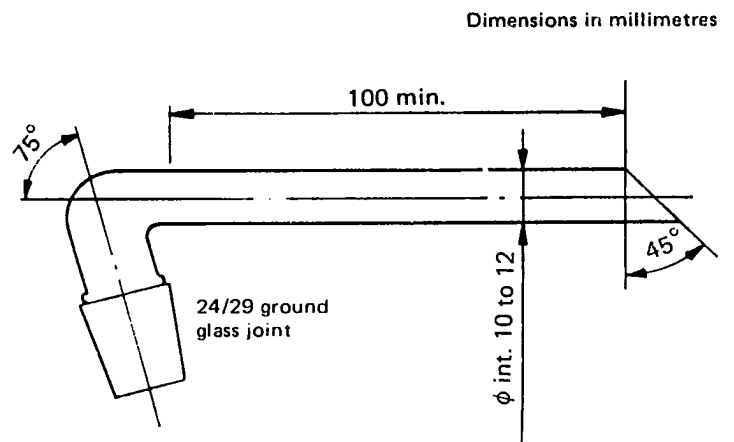


FIGURE 2 – Air condenser (4.7)

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 <i>o</i> -cresol content.
				E		ISO 1897/IX – Determination of <i>m</i> -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.

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- 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.