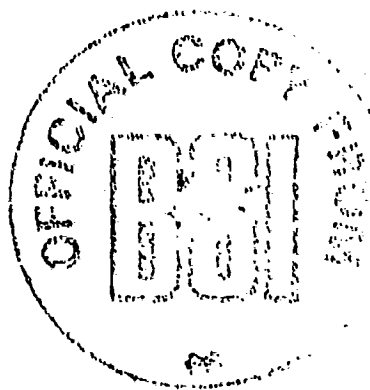


SPECIFICATION FOR
CRESYLIC ACID
OF SPECIFIED
META-CRESOL CONTENT

B.S. 521 : 1964



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THIS BRITISH STANDARD, having been approved by the Tar Products (other than Ammonia) Industry Standards Committee and endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the General Council on 29th December, 1964.

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Second revision September, 1948.

Third revision March, 1955.

Fourth revision December, 1964.

The Institution desires to call attention to the fact that this British Standard does not purport to include all the necessary provisions of a contract.

This standard makes reference to the following British Standards:

- B.S. 458/1. 3° xylene.
- B.S. 458/4. 5° xylene.
- B.S. 479/3. Coal-tar solvent naphtha (90/160).
- B.S. 572. Interchangeable conical ground glass joints.
- B.S. 593. Laboratory thermometers.
- B.S. 604. Graduated measuring cylinders.
- B.S. 612. Nessler cylinders.
- B.S. 616. Sampling of coal tar and its products.
- B.S. 658. Apparatus for the determination of distillation range (including flasks and receivers).
- B.S. 718. Density hydrometers and specific gravity hydrometers.
- B.S. 733. Density bottles.
- B.S. 756. Dean and Stark apparatus.
- B.S. 846. Burettes and bulb burettes.
- B.S. 1752. Laboratory sintered or fritted filters.
- B.S. 1848. Glass condensers.

British Standards are revised, when necessary, by the issue either of amendment slips or of revised editions. It is important that users of British Standards should ascertain that they are in possession of the latest amendments or editions.

The following B.S.I. references relate to the work on this standard:
Committee reference TPC/2 Draft for comment D63/9807

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CO-OPERATING ORGANIZATIONS

The Tar Products (other than Ammonia) Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

- *Association of British Chemical Manufacturers
- *Association of Tar Distillers
- *British Plastics Federation
- *Gas Council
- *Institution of Gas Engineers
 - Ministry of Defence, Army Department
 - Ministry of Defence, Navy Department
 - Ministry of Power
- *National Benzole and Allied Products Association
- Paintmakers Association of Great Britain
- *Standardization of Tar Products Test Committee

The industrial and scientific organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

- British Disinfectant Manufacturers' Association
- Low Temperature Coal Distillers Association of Great Britain
- Individual users and experts

BRITISH STANDARD SPECIFICATION FOR
CRESYLIC ACID
 OF SPECIFIED *META*-CRESOL CONTENT

FOREWORD

This specification forms one of a series of British Standards for phenol and cresylic acids, the preparation of which was authorized by the Chemical Divisional Council. It was first published in 1933, and revised in 1938, in 1948 and again in 1955.

In this present revision new requirements have been made in the specification to reduce the limit of *ortho*-cresol in Grade A material and the water content in all grades. New requirements have also been introduced in the distillation ranges and specific gravity. A more rapid and precise method for the determination of the *meta*-cresol content, based on the formation of a *meta*-cresol-urea complex, has been introduced.

By agreement with the Standardization of Tar Products Tests Committee, hereinafter referred to as S.T.P.T.C., the methods of testing and the descriptions of apparatus contained in the appendices have been reproduced, with editorial modifications, from their publication 'Standard methods for testing tar and its products' (5th edition, 1962).

It should be noted that British Standard specifications are available for many of the items of laboratory equipment used in testing, in addition to those which are described in the S.T.P.T.C. methods or which form the subject of British Standards referred to in the footnotes of this specification.

NOTE 1. These methods are intended to be carried out in accordance with the General Principles and apparatus requirements laid down in the S.T.P.T.C. publication mentioned.
 NOTE 2. The following definitions have been approved by the S.T.P.T.C. and have been adopted for the purposes of this British Standard:

Precision. The closeness of agreement among test results obtained under any definite set of conditions. Thus a method may be precise without being accurate. Repeatability and reproducibility are aspects of precision under two such defined sets of conditions.

Repeatability. A measure of the precision of the test results obtained by one operator using one set of apparatus. In this standard, repeatability, (R_1) is the difference between duplicate results on the same sample, that would be equalled or exceeded in the long run in only one case in twenty.

Reproducibility. A measure of the precision of test results obtained by different operators using different sets of the specified apparatus. Two measures of reproducibility are given in this standard. They are:

- R_{01} The difference, between a single result by one operator at one laboratory and a single result on the same sample by another operator at another laboratory, that would be equalled or exceeded in the long run in only one case in twenty.
- R_{02} The difference, between the mean of two results on one sample by one operator at one laboratory and the mean of two results by another operator on the same sample at another laboratory, that would be equalled or exceeded in the long run in only one case in twenty.

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SPECIFICATION

SCOPE

1. This British Standard covers cresylic acid in five grades according to *meta*-cresol content as specified in Clause 4.

DESCRIPTION

2. British Standard cresylic acid of specified *meta*-cresol content shall be a clear, colourless or very slightly tinted liquid.

NOTE. The material is liable to darken to a light brown colour on storing or on exposure to light.

COLOUR

3. The colour of the material shall not be more intense than Colour No. 2 (red tint) or Colour No. 3 (yellow tint), on the S.T.P.T.C. scale* when determined by the method described in Appendix A.

NOTE. This is approximately equal to 40 units (C) on the Lovibond† Scale, where $C = Y + 3R$, Y and R being respectively the number of yellow and red units required in combination to match the colour of the sample when measured in a 6 inch cell.

GRADES

4. The grades of the material are given in Table 1.

In establishing the grades, a range of 3 per cent on *meta*-cresol content has been adopted (except in Grade E, where the range is 4 per cent), in order to cover possible variations in sampling or in differences between analytical operators, and this shall be deemed to cover material specified for commercial

TABLE 1. GRADES

Grades	<i>meta</i> -cresol content (per cent)	
	above	not above
A	57	60
B	54	57
C	51	54
D	48	51
E	39	43

* 'Standard methods for testing tar and its products' (1962).

† The Tintometer Ltd., Salisbury.

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purposes within narrower limits of *meta*-cresol content. The *meta*-cresol content of the material shall be determined by the method described in Appendix B and shall not exceed the limits given in Table 1, unless otherwise agreed between purchaser and vendor.

ORTHO-CRESOL

5. The *ortho*-cresol content of the material shall not exceed the amounts given in Table 2, when determined by the method described in Appendix C.

TABLE 2. MAXIMUM ORTHO-CRESOL CONTENT

Grade	Maximum <i>ortho</i> -cresol content (per cent)
A	3
B	5
C	8
D	8
E	15

In the case of Grade E, the *ortho*-cresol content shall be within $(X \pm 2\frac{1}{2})$ per cent where X per cent is the figure agreed between purchaser and vendor.

WATER

6. The material shall not contain more than 0.3 per cent (w/w) of water when tested by the method described in Appendix D.

SPECIFIC GRAVITY*

7. The specific gravity, $S_{15.5/15.5^{\circ}\text{C}}$ of the material shall be not lower than 1.033 and not higher than 1.042 when determined by one of the methods described in Appendix E. In the event of dispute, Method 1 shall be used.

DISTILLATION RANGE

8. The material shall meet the requirements given in Table 3 when 100 ml is tested by the method described in Appendix F.

RESIDUE ON DISTILLATION

9. The material shall be free from pitch when tested by the method described in Appendix G.

* The term adopted by the International Organization for Standardization for this concept is 'relative density with reference to water'.

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TABLE 3. DISTILLATION RANGE

Grade	Requirements
A, B	At least 95 ml shall distil between 199°C and 204°C (running-points)
C, D	At least 92 ml shall distil between 199°C and 205°C (running-points)
E	At least 85 ml shall distil between 197°C and 205°C (running-points)

NEUTRAL OILS AND PYRIDINE BASES

10. The material shall not contain more than 0.1 per cent (v/v) of neutral oils and not more than 0.25 per cent (w/v) of pyridine bases, when tested by the method described in Appendix H.

HYDROGEN SULPHIDE

11. The material shall not impart more than a yellow coloration to lead acetate paper, when tested by the method described in Appendix J.

FREEDOM FROM ACIDS AND ALKALIS

12. The material shall be neutral to moistened litmus paper.

SAMPLING AND SIZE OF SAMPLE

13. A representative sample shall be taken from the bulk in accordance with the recommended methods given in B.S. 616, 'Sampling of coal tar and its products'. After thorough mixing, the requisite number of reference samples shall be prepared by transferring portions to clean, dry, amber- or blue-coloured glass stoppered bottles. For the purpose of examination for compliance with this specification, each one of these samples shall measure not less than 1 litre. The size of the sample bottle used shall be such that it is nearly filled by the sample, sufficient free space being left to allow for expansion and to allow the contents to be mixed by shaking. No rubber, sealing wax or similar material shall be allowed to come into contact with the sample and any plastics material used for sealing shall be applied in such a way that it cannot contaminate the samples when the bottles are opened.

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Phenolic substances burn the skin and can be absorbed into the system through the skin. It is essential for the sampler to wear gloves, preferably of polyvinyl chloride, and also a face shield. Inhalation of the vapours from hot material is to be avoided.

NOTE. The British Standards Institution is the owner of a registered certification trade mark. This is shown below, enclosed in the words 'Approved to British Standard'. This mark can be used only by manufacturers licensed under the certification mark scheme operated by the B.S.I. The presence of this mark on or in relation to a product is an assurance that the goods have been produced to comply with the requirements of the British Standard under a system of supervision, control and testing operated during manufacture and including periodical inspections at the manufacturer's works in accordance with the certification mark scheme of the B.S.I.

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APPENDIX A

METHOD FOR DETERMINATION OF COLOUR

(Technically identical with the relevant parts of S.T.P.T.C. Serial No. PC 1-62.)

A1. Outline of method. The colour of the sample is compared with that of standard solutions.

NOTE. The colour of cresylic acid is liable to darken on keeping and on exposure to light.

A2. Reagents. The reagents shall be of a recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used.

- a. *Cobalt sulphate*, crystalline heptahydrate ($\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$).
- b. *Copper sulphate*, crystalline pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$).
- c. *Potassium dichromate*, crystalline ($\text{K}_2\text{Cr}_2\text{O}_7$).

A3. Apparatus.

Nessler cylinders, two 50 ml cylinders complying with B.S. 612* and similar in every respect including colour, purchased as a matched pair.

A4. Colour standards. Prepare the following standard solutions to contain, per 1000 ml, the indicated quantities of reagents dissolved in distilled water, as given in Table 4.

TABLE 4. COLOUR STANDARDS SOLUTIONS

S.T.P.T.C. colour	grammes	Reagents
1	0.90 0.015	Cobalt sulphate Potassium dichromate
2	6.0 0.015	Cobalt sulphate Potassium dichromate
3	4.0 0.075 1.0	Cobalt sulphate Potassium dichromate Copper sulphate
4	22.5 0.06	Cobalt sulphate Potassium dichromate
5	10.0 0.18 2.5	Cobalt sulphate Potassium dichromate Copper sulphate

* B.S. 612, 'Nessler cylinders'.

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The tint of the even-numbered colours is red and that of the odd-numbered colours is yellow.

These solutions keep well and may be used up to one month from the date of preparation.

A5. Procedure.

Standard solutions. Compare 50 ml of the sample with 50 ml of standard solution, in the Nessler cylinders, held vertically 3 in (7.5 cm) above the surface of an opaque opal glass sheet reflecting diffused daylight. Report the colour of the samples as being darker than, equal to or not darker than that of the appropriate colour standard.

Use of permanent colour standards. For convenience, the colour of the sample may be compared with that of a suitable series of permanent colour standards using a Lovibond special purposes comparator or tintometer, but in cases of dispute the test is to be carried out by comparison with standard solutions as described above.

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APPENDIX B

METHOD FOR DETERMINATION OF *META*-CRESOL IN CRESYLIC ACID

(Technically identical with S.T.P.T.C. Serial No. PC 18-62.)

B1. Outline of method. This method is based on the fact that *m*-cresol forms a *m*-cresol-urea complex when excess of urea is added to the cresylic acid.

The water content is first determined by the method described in Appendix D. The sample is then dehydrated by distillation. The *m*-cresol content is determined from the crystallizing-point of the *m*-cresol/urea mixture.

B2. Reagents.

a. Urea. Analytical reagent grade urea, kept in an open bottle in a desiccator containing silica gel.

b. m-cresol, pure.

c. Light petroleum, 40–60°C.

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

B3. Apparatus.

a. Flask. A 250 ml short-necked round-bottomed flask with a B24 socket*.

b. Air condenser (S.T.P.T.C. Schedule No. C5)† as illustrated in Fig. 1.

c. Measuring cylinders, 100 ml and 10 ml, complying with B.S. 604‡.

d. Crystallizing-point apparatus. The apparatus is illustrated in Fig. 2. A glass test tube 150 × 25 mm nominal size is placed inside a 160 × 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 standard thermometer. 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 for the water bath passes through a hole in the cover plate and is held by a rubber ring.

* B.S. 572, 'Interchangeable conical ground glass joints'.

† 'Standard methods for testing tar and its products' (1962).

‡ B.S. 604, 'Graduated measuring cylinders'.

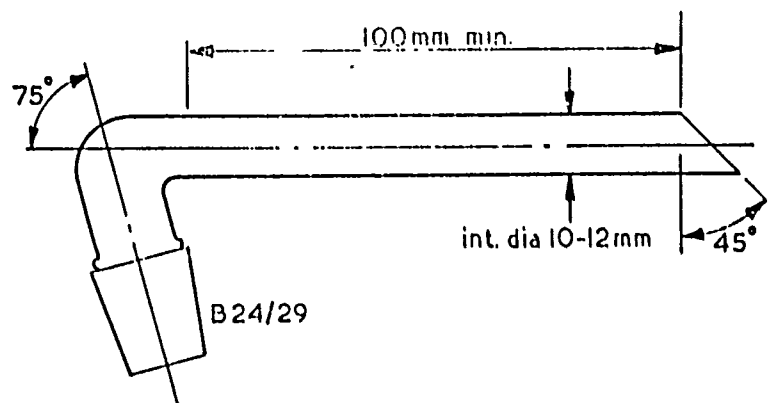


Fig. 1. Air condenser

e. *Thermometers*, S.T.P.T.C.* Schedule T3c and T5c or T17c as described in Appendix K, depending upon whether the *m*-cresol content is below or above 50 per cent.

f. *Calcium chloride tube* (S.T.P.T.C.* Schedule No. A3), consisting of a plain-end single B24 cone†, shank length approximately 115 mm, minimum bore 16.5 mm. The dehydrating agent is held in place by cotton wool plugs at the ends.

g. *Beaker*, small.

h. *Sintered glass funnel*, porosity Grade 2 complying with B.S. 1752‡.

j. *Bunsen burner*.

B4. Procedure.

Determination of water content. The water content by weight (W) is that determined by the method described in Appendix D.

Dehydration of the sample. Dehydrate the sample by slowly heating about 100 ml in the round-bottomed flask fitted with the air condenser, until 2 ml of distillate have been collected in the 10 ml measuring cylinder. The purpose of this distillation is to remove all traces of water without materially changing the composition of the sample. Replace the air condenser by the calcium chloride tube and allow to cool.

Determination of the crystallizing-point. Weigh 20.0 g of the dehydrated

* 'Standard methods for testing tar and its products' (1962).

† B.S. 572, 'Interchangeable conical ground glass joints'.

‡ B.S. 1752, 'Laboratory sintered or fritted filters'.