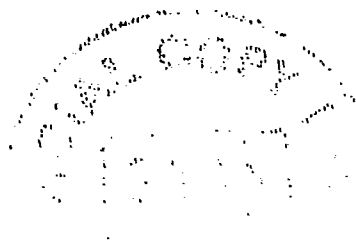


SPECIFICATION FOR  
CRESYLIC ACID  
OF SPECIFIED  
*ORTHO*-CRESOL CONTENT

**B.S. 517 : 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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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. Ir. changeable conical ground glass joints.
- R.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. 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 D63/9806

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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 Tests 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

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# BRITISH STANDARD SPECIFICATION FOR CRESYLIC ACID OF SPECIFIED *ORTHO*-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.

This present revision affects only the methods of test as given in the appendices; the specification clauses remain unchanged, except for slight re-editing, from the 1955 revision.

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 ( $R_r$ ) 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:

**$R_{D1}$ .** 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.

**$R_{D2}$ .** 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 having a specified *ortho*-cresol content of not less than 45 per cent nor more than 90 per cent (see Clause 4).

### DESCRIPTION

2. British Standard cresylic acid of specified *ortho*-cresol content shall be a clear, colourless to very pale yellow liquid or solid.

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 1. 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.

NOTE 2. The material may show a tendency to crystallize at normal laboratory temperatures. In this event, the sample should be gently warmed, just sufficiently to render it liquid, and maintained in the liquid state while the colour comparison is made.

### ORTHO-CRESOL

4. The material shall contain not less than 45 per cent nor more than 90 per cent of *ortho*-cresol when tested by the method described in Appendix B. A 5 per cent range (within these limits) shall be agreed between the purchaser and the vendor.

### WATER

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

### SPECIFIC GRAVITY‡

6. The specific gravity *S* 15.5/15.5°C of the material shall be not lower than 1.043 nor higher than 1.055 when determined by one of the methods described in Appendix D. In the event of dispute, Method 1 shall be used.

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

† The Tintometer Ltd., Salisbury.

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

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**DISTILLATION RANGE**

7. When 100 ml of the material is tested by the method described in Appendix E, not less than 90 ml shall distil between 190°C and 200°C (running-points).

**RESIDUE ON DISTILLATION**

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

**NEUTRAL OILS AND PYRIDINE BASES**

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

**HYDROGEN SULPHIDE**

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

**FREEDOM FROM ACIDS AND ALKALIS**

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

**SAMPLING AND SIZE OF SAMPLE**

12. 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'. When the material is in drums and received in the solid crystallized or partly crystallized state it shall be sampled in accordance with the instructions for 'liquefiable solids', in B.S. 616, an adaptation of which is given for the sake of convenience in Appendix J. 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 be 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.

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.

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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 water, as given in Table 1.

TABLE 1. COLOUR STANDARD SOLUTIONS

| S.T.P.T.C.<br>colour | grammes             | Reagents                                                   |
|----------------------|---------------------|------------------------------------------------------------|
| 1                    | 0.90<br>0.015       | Cobalt sulphate<br>Potassium dichromate                    |
| 2                    | 6.0<br>0.015        | Cobalt sulphate<br>Potassium dichromate                    |
| 3                    | 4.0<br>0.075<br>1.0 | Cobalt sulphate<br>Potassium dichromate<br>Copper sulphate |
| 4                    | 22.5<br>0.06        | Cobalt sulphate<br>Potassium dichromate                    |
| 5                    | 10.0<br>0.18<br>2.5 | Cobalt sulphate<br>Potassium dichromate<br>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 1 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 sample 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 *ORTHO*-CRESOL IN  
CRESYLIC ACID(Technically identical with S.T.P.T.C. Serial No. PC 17-62 and the relevant  
part of S.T.P.T.C. Serial No. PC 10-62.)

## B1. Outline of method.

The method is based on the fact that *o*-cresol forms an *o*-cresol/cineole complex when excess of cineole is added to cresylic acid.

It is primarily intended for samples containing 40 per cent or more of *o*-cresol.

The water content of the sample is first determined. The sample is then dehydrated and the *o*-cresol content is calculated from the water content of the sample and the crystallizing-point of a complex of the sample and cineole.

## B2. Reagents.

*a. Cineole.* It is essential that the crystallizing-point of the cineole shall be not lower than 1.2°C when determined in the apparatus shown in Fig. 2 with suitable adjustment of the bath temperature. The cineole should be kept quite dry and away from strong light, in amber-coloured bottles containing a little anhydrous calcium chloride. Check the crystallizing-point 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 is unsuitable for use in the test.

## B3. Apparatus.

*a. Distillation flask.* A 100 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. Thermometers.* As listed below and detailed in Appendix K.

For testing the cineole

S.T.P.T.C. schedule number†

T1c

For use in the crystallizing-point apparatus

T5c or T17c

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

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

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