

Specification for

Coal tar creosote for wood preservation

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Committees responsible for this British Standard

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British Telecommunications plc
 British Wood Preserving and Damp-proofing Association
 Chemical Industries Association
 Creosote Council
 Department of the Environment (Building Research Establishment)
 Timber Research and Development Association
 Timber Trade Federation
 Wood Panel Industries Federation

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Association of Consulting Scientists
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Foreword

This revision of BS 144 has been prepared under the direction of Technical Committee B/515, Wood preservation.

This British Standard was first published in 1921, with revisions in 1954, 1973 and 1990. The 1990 revision brought all types of creosote under one standard (low viscosity creosote not requiring heat for application had previously been covered by BS 3051). The 1990 revision specified three types of creosote to cover all applications; it also incorporated the provisions of BS 913, which was withdrawn. This revision reflects the subsequent advances in technology and particular attention has been paid to environmental issues.

Four types of creosote are specified in this revision, together with methods for determining conformity to the specifications. Methods of application previously specified in BS 144 : Part 2 : 1990 are now given in an informative annex.

Materials conforming to this specification and used as a wood preservative require Government approval under the Control of Pesticides Regulations 1986 (SI 1510) before they can be sold, supplied, stored, advertised or used in the United Kingdom.

This revision supersedes BS 144 : Part 1 : 1990 and BS 144 : Part 2 : 1990, which are withdrawn.

CAUTION. Attention is drawn to the Health and Safety at Work etc. Act 1974, and the need for ensuring that the methods specified in this British Standard are carried out with suitable precautions.

The procedures described in this British Standard are intended to be carried out by appropriately qualified and experienced persons or other suitably trained and/or supervised personnel. Normal safety precautions should be taken throughout performing the methods.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 20, an inside back cover and a back cover.

Specification

1 Scope

This British Standard specifies requirements for four types of coal tar creosote for wood preservation.

Type 1 creosote is intended for treatment of timber by pressure impregnation.

Type 2 creosote is also intended for pressure impregnation, but has a more closely defined distillation range and a more restricted residue content than creosote of type 1. Type 2 creosote is especially suitable for treatment of poles for overhead power and telecommunication lines, and for structural timbers where bleeding in service could present a problem.

Type 3 creosote is intended for treatment of timber by immersion and brushing.

Type 4 creosote is intended for treatment of timber by pressure impregnation at temperatures $> 100\text{ }^{\circ}\text{C}$. This creosote excludes the lower boiling fractions allowable in the other three creosote types and is especially suitable where volatility leading to odour is a problem.

NOTE. Creosote of types 1, 2 and 3 may be used for the hot-and-cold open tank process.

2 References

2.1 Normative references

This British Standard incorporates, by dated or undated reference, provisions from other publications. These normative references are made at the appropriate places in the text and the cited publications are listed on page 20. For dated references, only the edition cited applies; any subsequent amendments to or revisions of the cited publication apply to this British Standard only when incorporated in the reference by amendment or revision. For undated references, the latest edition of the cited publication applies, together with any amendments.

2.2 Informative references

This British Standard refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this Standard are listed on the inside back cover, but reference should be made to the latest editions.

3 Composition

All types of creosote shall consist of blends of distillates of coal tar and shall be free from petroleum oils or oils not derived from coal tar.

4 General requirements

4.1 When tested in accordance with the methods listed in table 1, the creosote, when manufactured, shall be in accordance with the limiting requirements given in that table. Samples for the assessment of conformity to these requirements shall be taken in accordance with BS EN 1014-1.

4.2 Requirements for creosote in use within treatment plants shall be as in table 1 except for:

| | |
|----------------------------------|--------------|
| water content (max.): | 3.0 % (V/V); |
| insoluble matter content (max.): | 0.6 % (m/m). |

5 Methods of treatment of timber

Guidance on methods of treatment of timber for use with these preparations is given in annex I.

| Table 1. Requirements for creosote types 1, 2, 3 and 4 | | | | | | | | | |
|--|--------|------|--------|------|--------|------|--------|------|---------------|
| Property | Type 1 | | Type 2 | | Type 3 | | Type 4 | | Test method |
| Liquidity Temperature (°C) at which the product is completely liquid, after: | | | | | | | | | Annex A |
| 2 h | 32 | | 32 | | — | | 50 | | |
| 4 h | — | | — | | 0 | | | | |
| Density (kg/m ³) at: | Min. | Max. | Min. | Max. | Min. | Max. | Min. | Max. | Annex B |
| 55 °C | — | — | — | — | — | — | 1003 | 1144 | |
| 38 °C | 1003 | 1108 | 1003 | 1108 | — | — | — | — | |
| 20 °C | — | — | — | — | 910 | 1120 | — | — | |
| Distillation Recovery of dehydrated creosote (% (m/m)) at: | | | | | | | | | Annex C |
| 205 °C | — | 6 | — | 5 | — | 15 | — | — | |
| 230 °C | — | 40 | 5 | 30 | — | 40 | — | — | |
| 270 °C | — | — | — | — | 30 | — | — | — | |
| 315 °C | — | 78 | 40 | 78 | 40 | 90 | — | 18 | |
| 355 °C | 60 | — | 73 | 90 | 65 | — | 65 | 95 | |
| Extractable phenols content (ml/100 g of dehydrated creosote) in the distillate up to 315 °C (as obtained in annex D): | Min. | Max. | Min. | Max. | Min. | Max. | Min. | Max. | Annex D |
| Types 1, 2 and 4, density range 1003 kg/m ³ to 1045 kg/m ³ | 5 | 20 | 5 | 20 | — | — | 5 | 18 | |
| Types 1, 2 and 4, density range 1046 kg/m ³ to 1144 kg/m ³ | 0 | 20 | 0 | 20 | — | — | 0 | 18 | |
| Type 3 | — | — | — | — | 1 | 20 | — | — | |
| Flash point (°C, Pensky-Martens closed tester) | 61 | — | 61 | — | 61 | — | 61 | — | BS EN 22719 |
| Viscosity (mm ² /s), kinematic at 40 °C | — | — | 4 | 20 | — | — | — | — | Annex E |
| Water content (% (V/V)) | — | 1.5 | — | 1.5 | — | 1.5 | — | 1.5 | Annex F |
| Insoluble matter content (% (m/m)) | — | 0.4 | — | 0.4 | — | 0.4 | — | 0.4 | Annex G |
| Naphthalene content (% (m/m)) | — | — | — | 15 | — | — | — | — | Annex H |
| Benzo[a]pyrene content (mg/kg) | — | 500 | — | 500 | — | 50 | — | 500 | DD ENV 1014-3 |
| Water extractable phenols content (% (m/m)) | — | 3 | — | 3 | — | 3 | — | 3 | BS EN 1014-4 |

Annexes

Annex A (normative)

Method for the determination of liquidity

A.1 Principle

The sample is maintained at the specified temperature (50 °C, 32 °C or 0 °C) for 2 h or 4 h and then examined for the presence of separated solid matter.

A.2 Apparatus

A.2.1 Conical flask, capacity 100 ml.

A.2.2 Thermometer, partial immersion thermometer of range -0.5 °C to 55 °C , graduated at each 0.1 °C and accurate to $\pm 0.2\text{ °C}$.

NOTE. A thermometer conforming to BS 593 is suitable.

A.2.3 Constant temperature baths. Depending on the type of creosote being tested, baths able to maintain temperatures of $(50 \pm 0.1)\text{ °C}$, $(32 \pm 0.1)\text{ °C}$ or $(0 \pm 0.1)\text{ °C}$.

A.3 Procedure

Pour about 50 ml of the laboratory sample (see BS EN 1014-1) into the conical flask. Fit the thermometer by means of a cork into the neck of the flask, with the bulb of the thermometer immersed in the creosote. Place the flask in the appropriate constant temperature bath.

Ensure that the surface of the creosote is below that of the water in the bath. Swirl the flask until the creosote reaches the appropriate temperature. Leave the flask in the bath for 2 h or 4 h as appropriate, then withdraw the flask and examine the surface of the creosote for solid matter. Rotate the flask slowly, holding it horizontally, and examine the sides for solid matter.

When the sample is free from solid matter, place the flask in the second bath, maintained at $(32 \pm 0.1)\text{ °C}$ for types 1 and 2 creosote, $(0 \pm 0.1)\text{ °C}$ for type 3, and $(50 \pm 0.1)\text{ °C}$ for type 4. Re-examine the sample for solids when the contents have reached the bath temperature, and again after having maintained the sample at the bath temperature for 2 h for types 1, 2 and 4, or 4 h for type 3.

A.4 Acceptance criteria

The sample shall be deemed to pass the liquidity test if it remains completely liquid for the specified period at the specified temperature.

The sample shall be deemed to fail the liquidity test if any solid matter is observed, either after cooling to the specified temperature or at any time up to the end of the specified period at that temperature.

Annex B (normative)

Method for the determination of density

B.1 Principle

Density is determined by means of a density hydrometer.

B.2 Apparatus

B.2.1 Hydrometer, conforming to series L50 of BS 718, calibrated for determination of density at 20 °C in g/ml, for use in liquids of low surface tension, and constructed of soda-lime glass.

NOTE. Hydrometers calibrated in kg/m^3 are also available in series L50 of BS 718, and these may be used instead. If such a hydrometer is used, it should be read to the nearest kg/m^3 (see B.3); the corrections described in B.4 should be multiplied by 1000, and the factor 1000 from the equation in B.4.2 omitted.

Examine the hydrometer before use to see that it is clean and dry and that there has been no displacement of the paper scale during use.

NOTE. Any displacement of the paper scale can be detected by reference to the means provided for this purpose: e.g., a horizontal line may be etched on the stem of the hydrometer and the corresponding datum marked on the paper scale. If the scale has been displaced, recertification of the hydrometer is necessary.

B.2.2 Hydrometer vessel, free from local irregularities producing distortion and several millimetres greater in diameter than the hydrometer bulb.

NOTE. A 1 l measuring cylinder conforming to BS 604 is suitable.

B.2.3 Partial immersion thermometer, of range -0.5 °C to 60 °C , graduated in 0.1 °C and accurate to $\pm 0.2\text{ °C}$.

NOTE. A thermometer conforming to BS 593 is suitable.

B.3 Procedure

Warm the laboratory sample (see BS EN 1014-1) to approximately 38 °C for types 1 and 2, 20 °C for type 3 and 55 °C for type 4 creosote. Fill the clean hydrometer vessel with the liquefied laboratory sample to a depth sufficient to allow the hydrometer to float.

NOTE. Pour the sample down the side of the vessel, to avoid formation of air bubbles.

Gently stir the sample, avoiding formation of air bubbles. Hold the hydrometer by the top of the stem, insert it carefully into the sample, and release it when approximately in the position of equilibrium, i.e. when it rises or falls only to a small degree.

Lightly press the top of the hydrometer stem, to immerse it further by a few millimetres. Release the hydrometer. After a few oscillations, when the hydrometer is steady, note the reading.

Observe the meniscus. If the stem is clean the meniscus shape will remain unchanged during the hydrometer oscillations. If the meniscus shape changes, clean the hydrometer and repeat the procedure.

Record the hydrometer reading to the nearest 0.001 g/ml and the temperature of the creosote to the nearest 0.1 °C. If the bottom of the meniscus is not visible, take the reading at the level where the meniscus merges into the stem of the hydrometer.

B.4 Calculation**B.4.1 Correction of hydrometer reading**

Calculate the corrected hydrometer reading, R_t , as follows:

$$R_t = R + C + 0.0007$$

where

- R is the hydrometer reading;
- C is the certification correction;
- 0.0007 is the meniscus height correction (to be used for opaque creosotes only).

NOTE. The value of R_t is the corrected hydrometer reading for the sample at t °C, the temperature of the creosote in the test.

B.4.2 Calculation of density

Calculate the density d , at the required temperature (55 °C, 38 °C or 20 °C), as follows:

$$d = 1000 (R_t m + a)$$

where

- d is the density (kg/m³);
- R_t is the corrected hydrometer reading (see **B.4.1**);
- m is the multiplication factor (see note);
- a is the addition factor (see note).

NOTE. m and a are obtained from table B.1 for types 1 and 2, from table B.2 for type 3, and table B.3 for type 4 creosote.

B.5 Expression of results

Express the density of the sample, to the nearest kg/m³, at the specified temperature.

Annex C (normative)**Method for the determination of distillation range****C.1 Principle**

A sample of creosote is dehydrated and distilled, and the fractions collected at specified temperatures.

C.2 Apparatus

C.2.1 Distillation apparatus, described in BS 658.

Assemble in accordance with BS 658, except as stated in a) to d).

- a) *Distillation flasks*:
 - 1) capacity 250 ml, untared;
 - 2) capacity 150 ml, tared, held in the vertical position by means of a clamp at the extreme upper end of the neck.
- b) *Condensers*, types 1 and 2.
- c) *Draught screen*, type 2, from which the shelf has been removed.
- d) *Crow receivers*, capacity 50 ml, tared.

C.2.2 Thermometer, with a range of -2 °C to 400 °C, as described in BS 593.

C.2.3 Separating funnels, capacity 50 ml, as described in BS 2021.

C.2.4 Fortin's barometer.

C.3 Corrections**C.3.1 General**

Apply the corrections described in **C.3.2** and **C.3.3** to the specified distillation temperatures before commencing the distillation.

C.3.2 Barometer readings

C.3.2.1 Read the barometer to obtain the atmospheric pressure in mbar or mmHg, and record the ambient temperature. Continue as described in either **C.3.2.2** for mbar or **C.3.2.3** for mmHg.

NOTE. If atmospheric pressure is measured in other units, the following conversion factors can be used:

$$1 \text{ mbar} = 100 \text{ N/m}^2 = 100 \text{ Pa};$$

$$1 \text{ mmHg} = 133.322 \text{ N/m}^2.$$

C.3.2.2 Correct the barometer readings for temperature as described in BS 658, and if the corrected reading differs from 1013 mbar, apply corrections to the specified distillation temperature by adding the value given in table C.1 for each millibar above 1013 mbar, or subtracting for each millibar below 1013 mbar.

Table B.1 Multiplication (*m*) and addition (*a*) factors for conversion of corrected hydrometer readings at *t* °C to density at 38 °C (creosote types 1 and 2)

| <i>t</i> °C | 0.0 | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 | 0.6 | 0.7 | 0.8 | 0.9 |
|-------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| 34 | 1.00318 – 0.00667 | 1.00309 – 0.00650 | 1.00299 – 0.00634 | 1.00290 – 0.00617 | 1.00281 – 0.00600 | 1.00271 – 0.00584 | 1.00262 – 0.00567 | 1.00253 – 0.00550 | 1.00243 – 0.00533 | 1.00234 – 0.00517 |
| 35 | 1.00225 – 0.00500 | 1.00215 – 0.00483 | 1.00206 – 0.00466 | 1.00197 – 0.00450 | 1.00188 – 0.00433 | 1.00178 – 0.00416 | 1.00169 – 0.00400 | 1.00160 – 0.00383 | 1.00150 – 0.00366 | 1.00141 – 0.00350 |
| 36 | 1.00132 – 0.00333 | 1.00122 – 0.00316 | 1.00113 – 0.00300 | 1.00104 – 0.00283 | 1.00094 – 0.00266 | 1.00085 – 0.00250 | 1.00076 – 0.00233 | 1.00067 – 0.00216 | 1.00057 – 0.00200 | 1.00048 – 0.00183 |
| 37 | 1.00039 – 0.00166 | 1.00030 – 0.00150 | 1.00020 – 0.00133 | 1.00011 – 0.00116 | 1.00002 – 0.00100 | 0.99992 – 0.00083 | 0.99983 – 0.00067 | 0.99974 – 0.00050 | 0.99964 – 0.00033 | 0.99955 – 0.00017 |
| 38 | 0.99946 0.00000 | 0.99937 0.00017 | 0.99928 0.00033 | 0.99918 0.00050 | 0.99909 0.00066 | 0.99900 0.00083 | 0.99890 0.00100 | 0.99881 0.00116 | 0.99872 0.00133 | 0.99863 0.00149 |
| 39 | 0.99853 0.00166 | 0.99844 0.00183 | 0.99835 0.00199 | 0.99826 0.00216 | 0.99816 0.00232 | 0.99807 0.00249 | 0.99798 0.00266 | 0.99789 0.00282 | 0.99780 0.00299 | 0.99770 0.00315 |
| 40 | 0.99761 0.00332 | 0.99752 0.00348 | 0.99743 0.00365 | 0.99733 0.00381 | 0.99724 0.00398 | 0.99715 0.00414 | 0.99706 0.00431 | 0.99697 0.00448 | 0.99687 0.00464 | 0.99678 0.00481 |
| 41 | 0.99669 0.00497 | 0.99660 0.00513 | 0.99650 0.00530 | 0.99641 0.00547 | 0.99632 0.00563 | 0.99623 0.00580 | 0.99614 0.00596 | 0.99604 0.00613 | 0.99595 0.00629 | 0.99586 0.00646 |

NOTE. The upper figure is the multiplication factor.

Table B.2 Multiplication (*m*) and addition (*a*) factors for conversion of corrected hydrometer readings at *t* °C to density at 20 °C (creosote type 3)

| <i>t</i> °C | 0.0 | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 | 0.6 | 0.7 | 0.8 | 0.9 |
|-------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| 15 | 1.00466 – 0.00835 | 1.00456 – 0.00818 | 1.00447 – 0.00801 | 1.00438 – 0.00785 | 1.00428 – 0.00768 | 1.00419 – 0.00751 | 1.00410 – 0.00734 | 1.00400 – 0.00718 | 1.00391 – 0.00701 | 1.00382 – 0.00684 |
| 16 | 1.00372 – 0.00667 | 1.00363 – 0.00651 | 1.00354 – 0.00634 | 1.00344 – 0.00617 | 1.00335 – 0.00600 | 1.00326 – 0.00584 | 1.00316 – 0.00567 | 1.00307 – 0.00550 | 1.00298 – 0.00533 | 1.00288 – 0.00517 |
| 17 | 1.00279 – 0.00500 | 1.00270 – 0.00483 | 1.00260 – 0.00467 | 1.00251 – 0.00450 | 1.00242 – 0.00433 | 1.00232 – 0.00416 | 1.00223 – 0.00400 | 1.00214 – 0.00383 | 1.00204 – 0.00366 | 1.00195 – 0.00350 |
| 18 | 1.00186 – 0.00333 | 1.00176 – 0.00316 | 1.00167 – 0.00300 | 1.00158 – 0.00283 | 1.00149 – 0.00266 | 1.00139 – 0.00250 | 1.00130 – 0.00233 | 1.00121 – 0.00216 | 1.00111 – 0.00200 | 1.00102 – 0.00183 |
| 19 | 1.00093 – 0.00166 | 1.00084 – 0.00150 | 1.00074 – 0.00133 | 1.00065 – 0.00117 | 1.00056 – 0.00100 | 1.00046 – 0.00083 | 1.00037 – 0.00067 | 1.00028 – 0.00050 | 1.00019 – 0.00033 | 1.00009 – 0.00017 |
| 20 | 1.00000 0.00000 | 0.99991 0.00017 | 0.99982 0.00033 | 0.99972 0.00050 | 0.99963 0.00067 | 0.99954 0.00083 | 0.99944 0.00100 | 0.99935 0.00117 | 0.99926 0.00133 | 0.99917 0.00150 |
| 21 | 0.99907 0.00166 | 0.99898 0.00183 | 0.99889 0.00200 | 0.99880 0.00216 | 0.99870 0.00233 | 0.99861 0.00250 | 0.99852 0.00266 | 0.99843 0.00283 | 0.99833 0.00300 | 0.99824 0.00316 |
| 22 | 0.99815 0.00333 | 0.99806 0.00350 | 0.99797 0.00366 | 0.99787 0.00383 | 0.99778 0.00400 | 0.99769 0.00416 | 0.99760 0.00433 | 0.99750 0.00450 | 0.99741 0.00467 | 0.99732 0.00483 |
| 23 | 0.99723 0.00500 | 0.99713 0.00517 | 0.99704 0.00533 | 0.99695 0.00550 | 0.99686 0.00567 | 0.99677 0.00584 | 0.99667 0.00600 | 0.99658 0.00617 | 0.99649 0.00634 | 0.99640 0.00651 |
| 24 | 0.99631 0.00667 | 0.99621 0.00684 | 0.99612 0.00701 | 0.99603 0.00718 | 0.99594 0.00734 | 0.99585 0.00751 | 0.99575 0.00768 | 0.99566 0.00785 | 0.99557 0.00801 | 0.99548 0.00818 |
| 25 | 0.99539 0.00835 | 0.99529 0.00852 | 0.99520 0.00868 | 0.99511 0.00885 | 0.99502 0.00902 | 0.99493 0.00919 | 0.99484 0.00935 | 0.99474 0.00952 | 0.99465 0.00969 | 0.99456 0.00986 |

NOTE. The upper figure is the multiplication factor.

Table B.3 Multiplication (*m*) and addition (*a*) factors for conversion of corrected hydrometer readings at *t* °C to density at 55 °C (creosote type 4)

| <i>t</i> °C | 0.0 | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 | 0.6 | 0.7 | 0.8 | 0.9 |
|-------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| 51 | 1.00315 – 0.00667 | 1.00306 – 0.00650 | 1.00297 – 0.00633 | 1.00288 – 0.00616 | 1.00279 – 0.00599 | 1.00270 – 0.00582 | 1.00261 – 0.00565 | 1.00252 – 0.00548 | 1.00243 – 0.00531 | 1.00234 – 0.00514 |
| 52 | 1.00222 – 0.00500 | 1.00213 – 0.00483 | 1.00204 – 0.00466 | 1.00195 – 0.00449 | 1.00186 – 0.00432 | 1.00177 – 0.00415 | 1.00168 – 0.00398 | 1.00159 – 0.00381 | 1.00150 – 0.00364 | 1.00141 – 0.00347 |
| 53 | 1.00129 – 0.00333 | 1.00120 – 0.00316 | 1.00111 – 0.00299 | 1.00102 – 0.00282 | 1.00093 – 0.00265 | 1.00084 – 0.00248 | 1.00075 – 0.00231 | 1.00066 – 0.00214 | 1.00057 – 0.00197 | 1.00048 – 0.00180 |
| 54 | 1.00036 – 0.00166 | 1.00027 – 0.00149 | 1.00018 – 0.00132 | 1.00009 – 0.00115 | 1.00000 – 0.00098 | 0.99991 – 0.00081 | 0.99982 – 0.00064 | 0.99973 – 0.00047 | 0.99964 – 0.00030 | 0.99955 – 0.00013 |
| 55 | 0.99943 0.00000 | 0.99934 0.00017 | 0.99925 0.00034 | 0.99916 0.00051 | 0.99907 0.00068 | 0.99898 0.00085 | 0.99889 0.00102 | 0.99880 0.00119 | 0.99871 0.00136 | 0.99862 0.00153 |
| 56 | 0.99850 0.00166 | 0.99841 0.00183 | 0.99832 0.00200 | 0.99823 0.00217 | 0.99814 0.00234 | 0.99805 0.00251 | 0.99796 0.00268 | 0.99787 0.00285 | 0.99778 0.00302 | 0.99769 0.00319 |
| 57 | 0.99757 0.00332 | 0.99748 0.00349 | 0.99739 0.00366 | 0.99730 0.00383 | 0.99721 0.00400 | 0.99712 0.00417 | 0.99703 0.00434 | 0.99694 0.00451 | 0.99685 0.00468 | 0.99676 0.00485 |
| 58 | 0.99664 0.00497 | 0.99655 0.00514 | 0.99646 0.00531 | 0.99637 0.00548 | 0.99628 0.00565 | 0.99619 0.00582 | 0.99610 0.00599 | 0.99601 0.00616 | 0.99592 0.00633 | 0.99583 0.00650 |
| 59 | 0.99571 0.00663 | 0.99562 0.00680 | 0.99553 0.00697 | 0.99544 0.00714 | 0.99535 0.00731 | 0.99526 0.00748 | 0.99517 0.00765 | 0.99508 0.00782 | 0.99499 0.00799 | 0.99490 0.00816 |

NOTE. The upper figure is the multiplication factor.

C.3.2.3 Correct the barometer readings for temperature as described in BS 658, and if the reading differs from 760 mmHg, apply corrections to the specified distillation temperature by adding the value given in table 4 for each millimetre of mercury above 760 mmHg, or subtracting for each millimetre of mercury below 760 mmHg.

| Specified temperature °C | Corrections °C | |
|-----------------------------|-------------------|---------------------------|
| | Per millibar | Per millimetre of mercury |
| 205 | 0.076 | 0.057 |
| 230 | 0.080 | 0.060 |
| 270 | 0.087 | 0.065 |
| 315 | 0.095 | 0.071 |
| 355 | 0.100 | 0.075 |

C.3.3 Thermometers

If necessary, make the appropriate adjustments indicated by the thermometer test certificate at any of the specified distillation temperatures (see table 1).

C.4 Preparation of test portion of dehydrated creosote

Transfer about 120 g of the sample (see BS EN 1014-1) into the 250 ml untared distillation flask, to which has been added fragments of porous inert material. Using the type 1 condenser, distil the sample and collect the distillate in a separating funnel (C.2.3), stopping the distillation when water ceases to distil. Allow the contents of the separating funnel to settle, run the lower water layer off, and return the oil layer to the distillation flask when the flask has cooled to about 40 °C. Mix the contents of the flask thoroughly, ensuring that the oil is homogeneous.

C.5 Procedure

Weigh, to an accuracy of 0.1 g, approximately 100 g of the dehydrated creosote (see C.4) directly into the tared 150 ml distillation flask. Record this mass as M_c . Add fragments of porous inert material and assemble the distillation apparatus, with the side arm of the flask extending at least 25 mm beyond the cork in the upper end of the type 2 condenser.

Apply heat, and distil at a rate of (5 ± 0.5) ml/min. If for any reason the distillation rate falls outside the specified limits at any time after the first 5 ml of distillate has been collected, discontinue the test and start again on another portion of the original sample.

NOTE. The specified distillation rate corresponds to approximately 90 drops/min (i.e. three drops in 2 s), but this figure should only be taken as a guide so that the rate in millimetres per minute may be kept under close observation.

If solids are deposited in the condenser during the distillation, warm the condenser so that such solids are collected in the fraction with which they distil.

Change the receiver at each corrected specified temperature (see tables 1 and 4), without stopping the distillation. Remove the heat when the thermometer indicates the highest corrected specified temperature. The final fraction includes the oil that drains from the condenser within 5 min after the heat has been removed.

Weigh each receiver containing distillate fraction. Note the mass of each fraction (f_1, f_2, f_3, f_4 , and f_5) and calculate the accumulative distillate as a percentage by mass of the dehydrated creosote (M_c).

Reserve the distillate fractions (f_1, f_2, f_3 , and f_4) for testing in accordance with annex D.

C.6 Calculation

Calculate the percentage by mass of each fraction as follows:

let f_1 be the mass of the fraction distilled up to 205 °C;

let f_2 be the mass of the fraction distilled between 205 °C and 230 °C;

let f_3 be the mass of the fraction distilled between 230 °C and 270 °C;

let f_4 be the mass of the fraction distilled between 270 °C and 315 °C;

let f_5 be the mass of the fraction distilled between 315 °C and 355 °C.

Calculate the cumulative distillate up to each of the specified temperatures as a percentage by mass of the dehydrated creosote (M_c) as required in table 1, using the generalized formula:

$$\text{cumulative percentage to a specific temperature} = \frac{100 \sum f_n}{M_c}$$

for $n = 1$ to 5, as appropriate.

C.7 Expression of results

Express the cumulative distillate as a percentage by mass of the dehydrated creosote (M_c) at the specified temperature.

Annex D (normative)

Method for the determination of extractable phenols

D.1 Principle

The phenols are extracted from the distillate up to 315 °C (obtained from the distillation described in annex C as f_1 , f_2 , f_3 , and f_4) with sodium hydroxide. The neutral oils and bases are removed by boiling. The phenols are liberated by hydrochloric acid and measured.

NOTE. The phenols, as recovered, contain water derived from the reagents used in the test. No correction is made for this water.

D.2 Reagents

NOTE. All reagents should be of recognized analytical grade and water conforming to grade 3 of BS EN ISO 3696 should be used throughout.

D.2.1 Hydrochloric acid (HCl), concentrated, 425 g/l.

D.2.2 Sodium chloride (NaCl), powdered solid.

D.2.3 Sodium chloride solution, saturated.

D.2.4 Sodium hydroxide solution (NaOH), 100 g/l.

D.2.5 Toluene [C₆H₅CH₃].

D.2.6 Methyl orange indicator solution, 4-[4-dimethylaminophenylazo]benzenesulfonic acid sodium salt, 1 g/l.

D.3 Apparatus

D.3.1 Phenols flask, capacity 200 ml or 150 ml (see notes 2 to 5 of **D.5**), with graduated neck conforming to BS 676.

D.3.2 Separating funnel, capacity 250 ml, stoppered, conforming to BS 2021.

D.3.3 Measuring cylinder, capacity 100 ml, conforming to BS 604.

D.3.4 Thermometer, of range - 10 °C to 110 °C, conforming to BS 593.

D.3.5 Glass wool.

D.4 Test portion

The test portion (see note) shall be obtained by combining distillate fractions $f_1 + f_2 + f_3 + f_4$, collected below 315 °C (see annex C).

NOTE. A known mass (approximately 100 g) of dehydrated creosote (M_C) will have been distilled to produce the combined fractions ($f_1 + f_2 + f_3 + f_4$) which constitute this test portion.

D.5 Procedure

If necessary, warm the test portion (see **D.4**) until completely liquid, and transfer it to the separating funnel (**D.3.2**). With 50 ml of sodium hydroxide solution (**D.2.4**) (see notes 1 and 2), rinse the receivers originally containing the test samples into the separating funnel. If the combined samples contain solids which have separated, warm the separating funnel just sufficiently to redissolve the solids.

Stopper the funnel, shake vigorously for 1 min to 2 min, and allow to stand. After separation (see note 1), pour the alkaline layer into a beaker. Repeat this operation with successive 25 ml portions of sodium hydroxide solution (**D.2.4**) until all the phenols have been removed from the oil layer (see notes 1 and 2).

After separation, add the alkaline layers to the first sodium hydroxide washing.

During the extraction procedure, ensure that the contents of the separating funnel are completely liquid. If necessary, immerse the funnel in warm water (40 °C to 70 °C).

Take the combined sodium hydroxide washings and boil for 20 min (using, if necessary, a fragment of inert material of approximately 2 mm³, to prevent bumping), and roughly maintain the initial volume by adding water. Cool the sodium hydroxide washings to laboratory temperature and, if clear, transfer them directly to the phenols flask (see note 4). If the solution contains suspended matter, filter it through glass wool moistened with sodium chloride solution (**D.2.3**) and collect in the 200 ml phenols flask (**D.3.1**) (see note 4). Wash the glass wool with a further 25 ml of sodium chloride solution and add to the filtered sodium hydroxide washings.

Add a few drops of methyl orange indicator solution (**D.2.6**) to the washings, and slowly add concentrated hydrochloric acid (**D.2.1**) until the methyl orange just indicates distinct acidity, mixing the two layers by swirling. While adding the hydrochloric acid, keep the contents of the flask cool by immersing it periodically in cold water.

Add just sufficient powdered sodium chloride (**D.2.2**) to saturate the aqueous layer and leave a few particles undissolved. Shake to ensure thorough mixing and then stand to allow separation, the phenols forming the upper liquid layer. Bring the phenols into the graduated portion of the flask by adding the saturated sodium chloride solution (**D.2.3**). After setting (preferably overnight), measure the volume of phenols (see note 5). Record the volume as V_p .

NOTE 1. Complete removal of the phenols can be verified by slightly acidifying the final washings with concentrated hydrochloric acid (**D.2.1**) and examining for separated phenols.

NOTE 2. It may be assumed that 25 ml of sodium hydroxide solution (**D.2.4**) is sufficient to remove about 5 ml of phenols.

NOTE 3. If necessary, the upper layer of the flask may be diluted with toluene (**D.2.5**) to secure a satisfactory separation.

NOTE 4. If it is expected that the sample for test contains only a small amount of phenols, it is preferable to use about half the specified volumes of sodium hydroxide solution for the successive washings and to collect the sodium hydroxide washings (after filtration through the glass wool if necessary) in the 150 ml phenols flask.

NOTE 5. For some creosotes, measurement of the liberated phenols is difficult because of their viscous nature. This may be overcome by adding a measured volume of toluene (**D.2.5**) to the phenols flask immediately before the final addition of saturated sodium chloride solution. From the observed volume of the separated upper layer in the phenols flask, subtract the volume of toluene added.

D.6 Expression of results

Express the volume of phenols (V_p), in millimetres, in 100 g of the original sample of dehydrated creosote (M_c), as follows:

$$\frac{V_p \times 100}{M_c}$$

D.7 Precision

D.7.1 Repeatability

Duplicate results for the volume of extractable phenols obtained by the same operator shall be considered suspect if they differ by more than 0.8 ml.

D.7.2 Reproducibility

Single results for the volume of extractable phenols obtained at separate laboratories shall be considered suspect if they differ by more than 2.9 ml.

Annex E (normative)

Method for the determination of kinematic viscosity

E.1 Principle

The kinematic viscosity of a sample of creosote at 40 °C is determined using a U-tube reverse flow viscometer.

E.2 Apparatus

E.2.1 U-tube reverse flow viscometers, type BS/IP/RF, conforming to BS 188 : 1977, size 2 (viscosity range 2 mm²/s to 10 mm²/s, nominal factor $C = 0.01$ mm²/s²) and size 3 (viscosity range 6 mm²/s to 30 mm²/s, nominal factor $C = 0.03$ mm²/s²), with certificate of calibration.

E.2.2 Test sieve, nominal aperture size 75 µm, conforming to BS 410.

E.2.3 Water bath, thermostatically controlled, able to adjust to (40 ± 0.1) °C.

NOTE. The temperature control should be such that the bath does not vary by more than 0.01 °C during the period of measurement, over the viscometer or between viscometers. The depth of the bath should be such that, when the viscometer and sample are in position, no part of the sample is less than 20 mm below the surface of the water, or less than 20 mm above the bottom of the bath.

E.2.4 Thermometer, total immersion type. Details of suitable thermometers are given in BS 2000 :

Part 0 : Addendum 1.

E.2.5 Viscometer holder, to hold the viscometer firmly in the thermostatic bath, in the alignment described in the certificate of calibration.

E.2.6 Timing device, graduated in divisions of 0.2 s or less, accurate to 0.07 % over 15 min.

E.3 Preparation of test portion

Warm the creosote sample (see BS EN 1014-1) to 45 °C and filter a minimum of 20 ml through the test sieve (E.2.2).

E.4 Procedure

E.4.1 Filling the viscometer

NOTE. See figure E.1.

Vertically mount the thermometer (E.2.4) in the bath (E.2.3), so that the top of the mercury column is within 2 mm of the surface of the water. Mount the clean, dry viscometer (E.2.1) inside the viscometer holder (E.2.5) in the bath, in the alignment described in the certificate of calibration. Allow the viscometer to reach bath temperature. Stop tube L with a rubber bung fitted with a stopcock or similar device so that the air can be prevented from escaping. With the stopcock open, pour the creosote sample (E.3) into the filling tube N to a point just below the upper filling mark H, without wetting the glass above H. Allow the sample to flow through the capillary tube R, ensuring that the creosote column remains unbroken, until it has reached a position about 5 mm below the lower filling mark G. Stop the flow of creosote at this point by closing tube L.

Add more of the sample to the filling tube N to bring the upper surface of the creosote to just below the mark H. Keep the viscometer in the bath for at least 30 min, to allow the sample to reach bath temperature and for any air bubbles to rise to the surface. With the stopcock, carefully adjust the lower level of the creosote so that its ring of contact with the glass coincides with the bottom of mark G. Next add further sample to tube N, until the ring of contact of the creosote coincides with the bottom of mark H. Allow time for this additional amount of sample to reach bath temperature.

E.4.2 Measurement

Remove the rubber bung from tube L or open the stopcock to allow the creosote to flow under its own head. Measure the time for the uppermost ring of contact of the sample with the glass to rise from the bottom of mark E to the bottom of mark F. Record the thermometer readings before, during, and after the timed flow.

NOTE. As some of the sample may remain on the walls of the viscometer between the timing marks, repeat determinations of flow time may be made only after emptying, cleaning and drying the viscometer.

E.5 Expression of results

The kinematic viscosity of the creosote sample is calculated from the following equation:

$$\eta = Ct$$

where

η is the kinematic viscosity (mm²/s);

C is the viscometer constant;

t is the mean flow time (s).

E.6 Repeatability

Duplicate results for the kinematic viscosity, obtained by the same operator with the same viscometer, shall be considered suspect if the difference is greater than 0.35 % of the mean.

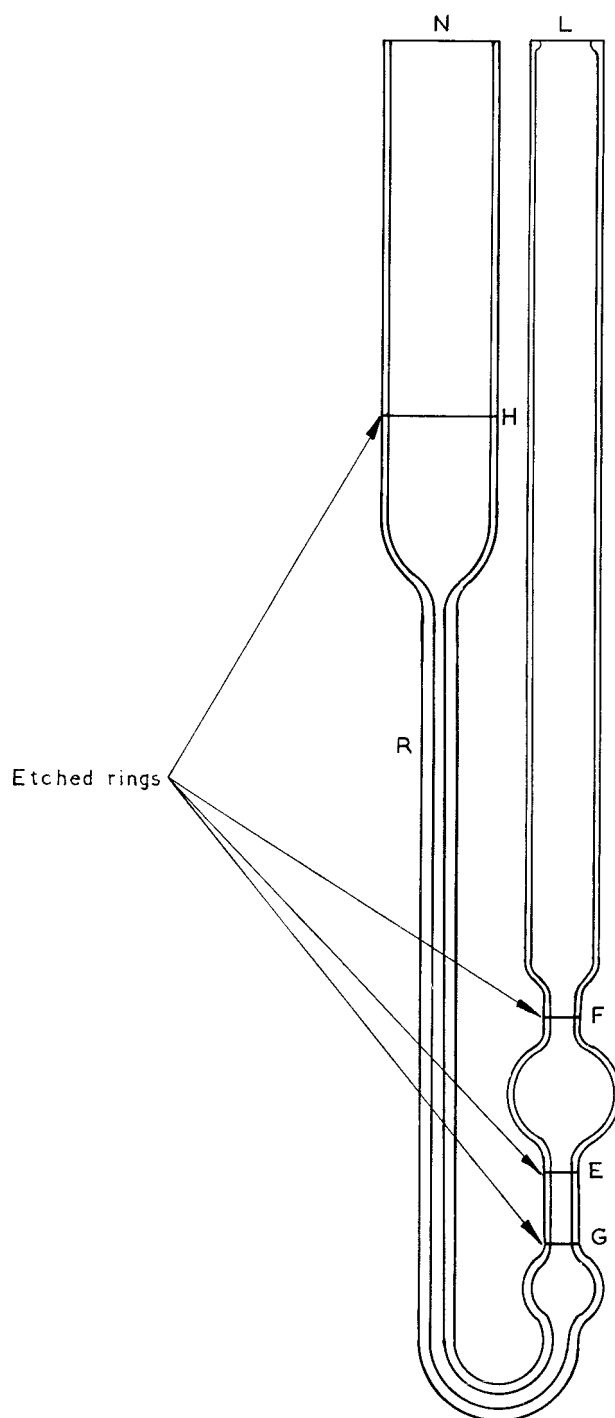


Figure E.1 BS/IP/RF U-tube reverse flow viscometer

Annex F (normative)**Method for the determination of water content****F.1 Principle**

The sample is azeotropically distilled with a suitable solvent, and the water collected and measured.

F.2 Reagent**F.2.1 Solvent**, either:

- a) toluene [C₆H₅CH₃]; or
- b) xylene [C₆H₄(CH₃)₂].

F.3 Apparatus

F.3.1 Measuring cylinder, capacity 100 ml, conforming to BS 604.

F.3.2 Dean and Stark condensing and collecting system, conforming to BS 756 : 1952, type 1, with a 2 ml receiver.

F.3.3 Round-bottomed flask, capacity 500 ml.

F.4 Procedure

Fill the measuring cylinder (**F.3.1**) to the 100 ml mark with thoroughly mixed sample (see BS EN 1014 : Part 1) at laboratory temperature (or at the lowest temperature of complete liquidity if above laboratory temperature), and transfer it to the flask (**F.3.3**). Wash the measuring cylinder with successive quantities of solvent (**F.2.1**), using 100 ml altogether, and add the washings to the flask. Add a fragment of porous inert material and connect the flask to the Dean and Stark condensing and collecting system (**F.3.2**).

Heat the flask so that the condensate falls from the end of the condenser at a rate of 2 drops/sec to 5 drops/sec. Continue the distillation until condensed water is no longer visible in any part of the apparatus (except at the bottom of the graduated tube), and the volume of collected water remains constant. Record the volume of water in the graduated tube.

If a ring of condensed water persists in the condenser tube, clean the condenser and repeat the determination.

F.5 Expression of results

The water content, as a percentage by volume, is equal to the volume, in millimetres, of water in the graduated tube.

Annex G (normative)**Method for the determination of matter insoluble in toluene****G.1 Principle**

A sample of creosote is mixed with toluene and the soluble matter filtered out. The remainder is washed, dried and weighed.

G.2 Reagent

G.2.1 Toluene [C₆H₅CH₃].

G.3 Apparatus

G.3.1 Beakers, capacity 250 ml, with glass covers.

G.3.2 Filter crucibles, capacity 30 ml, glass or porcelain, porosity grade P16, conforming to BS 1752 : 1983.

G.3.3 Measuring cylinder, capacity 100 ml, conforming to BS 604.

G.3.4 Water bath, able to heat to boiling.

G.3.5 Laboratory oven, (105 ± 5) °C.

G.3.6 Desiccator, with silica gel desiccant.

G.3.7 Analytical balance.

G.3.8 Stainless steel filter, mesh size 150 μm, conforming to BS 410.

G.4 Procedure

Dry a filter crucible (**G.3.2**) in the oven at 105 °C, cool in the desiccator and weigh to the nearest 0.1 mg. Repeat until successive weighings do not differ by more than 0.2 mg. Record the mass of the crucible (m_1).

Pour 25 g to 30 g of thoroughly mixed sample (see BS EN 1014-1) through the stainless steel filter (**G.3.8**) into a beaker (**G.3.1**), weigh to the nearest 0.1 g and record the mass of the sample (m_2). Add 100 ml of toluene (**G.2.1**) to the beaker, place a glass cover on the beaker, and heat on the boiling water bath.

Cautiously stir the contents of the beaker with the glass rod. When the sample has dissolved, cover the beaker and leave on the water bath for about 10 min to allow the greater part of the insoluble matter to settle.

Heat approximately 200 ml of toluene (**G.2.1**) on the water bath (80 °C to 100 °C) for washing purposes.

Decant the supernatant solution in the beaker through the filter crucible, using gentle suction to assist filtration. With the hot toluene, quantitatively transfer the insoluble matter to the crucible and rinse out the beaker. Wash the filter and its contents three or four times using the remainder of the hot toluene.

Dry the crucible in the oven at 105 °C, cool in the desiccator and weigh to the nearest 0.1 mg. Repeat until successive weighings do not differ by more than 0.2 mg. Record the final mass of the crucible (m_3).

G.5 Expression of results

Calculate the matter insoluble in toluene as a percentage by mass of the sample using the following equation:

$$\frac{(m_3 - m_1) \times 100}{m_2}$$

where

- m_1 is the mass of the crucible (g);
- m_2 is the mass of the test portion (g);
- m_3 is the mass of the crucible and residue after drying (g).

Annex H (normative)

Method for the determination of naphthalene content by gas chromatography

H.1 Principle

A toluene solution of the creosote sample is analysed by gas chromatography using a flame ionization detector, and the response compared with that of a standard naphthalene solution.

H.2 Reagents and materials

NOTE. All reagents should be checked for purity by passing a sample through the chromatograph under the conditions of the determination (see H.6). If a response is obtained on the chromatogram that is likely to cause significant errors in the determination on the test sample, then the reagent or material should be rejected.

H.2.1 Carrier gas, nitrogen (N₂), oxygen-free.

H.2.2 Detector gases, hydrogen (H₂) and compressed air, to be used as directed by the manufacturers of the gas chromatograph.

H.2.3 Toluene [C₆H₅CH₃], giving no peaks that coincide with those of naphthalene or tetralin when 1 μl (see note) is analysed by gas chromatography.

NOTE. Some sample splitters may require a different injection volume to 1 μl. In these cases, account should be taken using the procedure given in H.6.1.3.

H.2.4 Benzo[b]thiophene (thionaphthalene), of a purity such that, when used in the column performance solution (H.5), and the solution is analysed by gas chromatography, there are no additional peaks between the main, single peaks of naphthalene and benzo[b]thiophene.

H.2.5 Naphthalene [C₁₀H₈], minimum melting point 79.6 °C.

H.2.6 1,2,3,4-tetrahydronaphthalene (tetralin) [C₆H₄(CH₂)₃CH₂], showing no peaks that coincide with those of naphthalene by gas chromatography.

H.3 Apparatus

H.3.1 Gas chromatograph, fitted with a flame ionization detector and heated injection port with sample splitter, and the following:

- a) a column temperature range of 130 °C to 200 °C;
- b) the injection port temperature set at 200 °C to 250 °C;
- c) a silica capillary column, approximately 25 m in length and 0.2 mm ID, coated with methyl silicone gum (OV1);
- d) a potentiometric strip chart recorder with minimum chart width of 200 mm and maximum response time of 1 s.

NOTE. An electronic integrator may be used either in addition or as an alternative to the chart recorder.

H.3.2 Micro-pipette, syringe type, suitable for accurately injecting 1 μl portions into the gas chromatograph.

H.3.3 Micro-pipette, syringe type, able to accurately measure 100 μl.

H.3.4 Analytical balance.

H.3.5 One-mark volumetric flasks, capacity 10 ml, conforming to BS 1792.

H.4 Calibration solutions

Prepare duplicate calibration solutions of approximately the same composition by the following procedure.

Add approximately 0.1 g of naphthalene (H.2.5) to a tared 10 ml one-mark volumetric flask (H.3.5), stopper the flask, and weigh to the nearest 0.0002 g. Add carefully, from a syringe, 10 μl of 1,2,3,4-tetrahydronaphthalene (H.2.6), replace the stopper, and re-weigh. Dilute to the mark with toluene (H.2.3).

H.5 Column performance solution

Take approximately 0.01 g of naphthalene (H.2.5), weigh accurately and transfer quantitatively to a 10 ml one-mark volumetric flask (H.3.5). Take approximately 0.10 g of benzo[b]thiophene (H.2.4), weigh accurately and transfer quantitatively to the same 10 ml one-mark volumetric flask (H.3.5). Add toluene (H.2.3), first to dissolve, then to make up to the mark.

H.6 Procedure

NOTE. Throughout this procedure all solutions are analysed by injecting 1 μl samples into the gas chromatograph (see also the note to H.2.3).

H.6.1 Setting up the gas chromatograph

H.6.1.1 Oven conditions

For all analyses, maintain the column oven at 130 °C until the benzo[b]thiophene has been eluted. Then raise the oven temperature to 200 °C (either manually or by a temperature programmer) and maintain at this temperature until the rest of the sample has been eluted. Reset the oven to 130 °C, allowing sufficient time for the temperature to equilibrate before running the next sample.

H.6.1.2 Carrier gas

Adjust the carrier gas flow to give a retention time for naphthalene of 15 min to 20 min.

H.6.1.3 Detector signal

If using a strip chart recorder, adjust the detector signal to give a naphthalene peak height of 50 % to 99 % of full-scale deflection.

H.6.1.4 Column performance

NOTE. The criteria in a), b), and c) should be checked during the adjustments to the chromatograph described in **H.6.1.1**, **H.6.1.2** and **H.6.1.3**.

Satisfy the following before the column is used for analysis:

- that peaks to be measured are symmetrical, i.e. show no obvious tailing;
- that the number of theoretical plates measured on the naphthalene peak is not less than 5000. The theoretical plate number (n) is calculated from the equation:

$$n = 16 (\text{retention time/peak width})^2$$

where both retention time and peak width are measured in length terms directly from the recorder chart;

- that the separation between naphthalene and benzo[*b*]thiophene is such that the trough ratio is less than 0.2. The trough ratio is determined by dividing the height of the trough between two partially resolved peaks by the height of the smaller of the two peaks, both measurements being made from the baseline.

H.6.2 Calibration

Analyse in turn 1 μl of each of the duplicate calibration solutions by chromatography (**H.4**), applying the conditions described in **H.6.1** to obtain separate chromatograms.

Measure the heights of the naphthalene and 1,2,3,4-tetrahydronaphthalene peaks from the chromatograms (or take the equivalent values from an integrator). Calculate the calibration factor f_n for each solution, from the formula:

$$f_n = \frac{H_t}{H_n} \times \frac{m_n}{m_t}$$

where

H_t is the peak height (or integrator reading) of 1,2,3,4-tetrahydronaphthalene;

H_n is the peak height (or integrator reading) of naphthalene;

m_t is the mass of 1,2,3,4-tetrahydronaphthalene in 10 ml of solution (g);

m_n is the mass of naphthalene in 10 ml of solution (g).

Determine the mean of the two factors.

H.6.3 Analysis

If the approximate naphthalene content is not known, weigh 0.20 g of the test sample (see BS EN 1014-1) into a one-mark volumetric flask, dilute to 10 ml with toluene (**H.2.3**) and analyse 1 μl of the solution by chromatography under the same conditions as for the calibration (**H.6.2**).

NOTE. If the test sample requires warming to dissolve solid matter before analysis, care should be taken that volatile constituents are not lost.

Measure the peak height for naphthalene and compare this with the peak heights obtained from the calibration solutions. Calculate the approximate naphthalene content.

Weigh a test portion containing 0.05 g to 0.1 g of naphthalene into a tared 10 ml one-mark volumetric flask and dilute to the mark with toluene. Inject 1 μl of this solution into the chromatograph to obtain the first chromatogram. Weigh the flask and add 100 μl of 1,2,3,4-tetrahydronaphthalene as in the calibration. Shake and re-weigh the flask, and inject 1 μl of this solution into the chromatograph to obtain the second chromatogram.

H.6.4 Measurement of peak heights

Draw in the peak base for each relevant peak on the chromatogram, and measure the vertical distance between the peak base and the apex of the peak.

NOTE. The peak base is defined as the interpolation line drawn between the start and the finish of the peak, and represents the base line that the chromatogram would have followed if the sample component forming the peak had not been present.

Record the peak heights for naphthalene and 1,2,3,4-tetrahydronaphthalene in the second chromatogram, and for naphthalene and any coincident peak at the 1,2,3,4-tetrahydronaphthalene retention time in the first chromatogram.

H.7 Expression of results

The naphthalene content of the test sample, as a percentage by mass, is given by the following formula:

Naphthalene content =

$$\frac{(H_{n1} + H_{n2})}{2m_s} \times \frac{m_t}{(H_{t2} - H_{t1})} \times 100f_n$$

where

- f_n is the calibration factor;
- m_t is the mass of 1,2,3,4-tetrahydronaphthalene added (g);
- m_s is the mass of sample taken (g);
- H_{n1} is the height of the naphthalene peak (or integrator reading) in the first chromatogram (mm);
- H_{n2} is the height of the naphthalene peak (or integrator reading) in the second chromatogram (mm);
- H_{t1} is the height of the peak (or integrator reading) at the 1,2,3,4-tetrahydronaphthalene retention time in the first chromatogram (mm);
- H_{t2} is the height of the 1,2,3,4-tetrahydronaphthalene peak (or integrator reading) in the second chromatogram (mm).

If $H_{t1} = 0$, the formula simplifies to:

$$\text{Naphthalene content} = \frac{(H_{n1} + H_{n2})}{2m_s} \times \frac{m_t}{H_{t2}} \times 100f_n$$

Annex I (informative)

Guidance on the methods of treatment of timber

Introduction

This annex gives guidance on the methods that can be used for the treatment of timber with creosote.

The method to select depends on the end use of the treated timber and the penetration and retention of creosote required by the specifier.

NOTE. Guidance on the treatment of timber with preservatives and an introduction to the European Standards for wood preservation is given in BS 1282 and DD239 (in preparation).

I.1 Condition and preparation of timber for treatment

I.1.1 Moisture content

Unless otherwise specified, or when using the Boulton process (see I.2.6.5), the average moisture content of the timber should not exceed 28 % (m/m) on an oven dry basis.

The Boulton process can be used on green or unseasoned timber, to ensure a moisture content of 28 % (m/m) or lower before continuing with a full cell or empty cell process.

NOTE. If timber is frozen, the penetration power of preservative is limited, so treatment should not be carried out in this condition.

I.1.2 Incising

If incising the timber is specified, incisions should be made parallel to the general direction of the grain. Incisions (usually of up to 20 mm in length) should penetrate to a depth of approximately 20 mm, and should be spaced 25 mm apart across the timber, in rows extending across all faces of the timber. The distance between each row along the timber should be 60 mm, and incisions in adjacent rows should not be in direct line but staggered at intervals of approximately 6 mm (i.e. incisions in every fourth row are in direct line, see figure I.1).

NOTE 1. Sawn timbers rated as resistant to preservative (e.g. Douglas fir), of thickness 75 mm or more, and intended for exterior use, can benefit from incising before treatment.

NOTE 2. For definitions of resistant or extremely resistant timber, refer to BS EN 350-1 and BS EN 350-2.

I.2 Processing by pressure impregnation with creosote types 1, 2 or 4

I.2.1 General

Many different schedules for applying creosote under pressure have been devised; each requires the timber to be contained in a pressure cylinder and uses hot creosote with varying combinations of time, temperature, pressure and vacuum. Details of the most commonly used processes in the UK are given in I.2.6.2, I.2.6.3 and I.2.6.4. An associated process that may be used to condition green or unseasoned timber prior to pressure impregnation, the Boulton process, is described in I.2.6.5.

Full cell processes are normally used where high absorption of creosote is required or where impermeable species are being treated. After treatment, the timber cells are nearly full of creosote, which may give rise to exudation of free creosote on the surface of the timber when in service.

Empty cell processes are normally used when permeable species are being treated, and the high absorption resulting from a full cell process is not necessary. After treatment, the timber cells contain almost no creosote, minimizing exudation of creosote on the surface.

I.2.2 Stacking in the cylinder

The timber should be stacked in the cylinder so that the creosote can reach all surfaces. If necessary, the timber should be separated by sticks or laths.

NOTE 1. Consideration of stacking is particularly important when planed timber is to be treated.

NOTE 2. Only timbers of the same species and similar cross-sectional areas should be treated in the same charge, and differing timbers should be separated unless the most intense schedule can be applied without detriment to the more easily treated timbers.

I.2.3 Applied pressure and temperature

The applied pressure should be a gauge pressure not lower than 10 bar and not higher than 14 bar. During the pressure period, the minimum temperature for type 1 and type 2 creosote should be 65 °C and the maximum temperature should be 100 °C.

For type 4 creosote the minimum temperature should be 90 °C and the maximum 125 °C.

I.2.4 Pressure period

The pressure period should be measured from the time the specified maximum working pressure (I.2.3) is reached. Pressure should be maintained until the required penetration and retention has been achieved.

I.2.5 Penetration and retention

I.2.5.1 General

The method that should be used by specifiers to describe the quality of treatment required in preservative-treated timber is given in BS EN 351. This method requires the result of the treatment to be

expressed rather than the treatment process. The result is described in terms of the preservative penetration into the wood, and the retention of the wood preservative in the defined analytical zone.

NOTE 1. It is anticipated that penetration and retention requirements may be routinely achieved through a defined treatment process, provided that it has been established that the defined process is able to produce treated timber with the required penetration and retention values.

NOTE 2. Guidance on methods for sampling treated timber to determine penetration and retention values is presented in BS EN 351-2.

I.2.5.2 Penetration

Listed in BS EN 351-1 are nine penetration classes that can be used to specify penetration, starting from P1 (no penetration requirement) to P9 (full sapwood penetration and a minimum of 6 mm penetration of exposed heartwood).

I.2.5.3 Retention

The average retention for the charge, determined at the analytical zone (according to BS EN 351-1), should be not less than that specified. Retention values are based upon the critical value of the preservative for the hazard class concerned (see BS EN 599-1).

I.2.6 Treatment schedules

I.2.6.1 General

The timber should be treated with coal tar creosote of type 1, type 2 or type 4, using one of the procedures described in I.2.6.2 to I.2.6.4, subject to any additional requirements of the user.

The Boulton process (see I.2.6.5) is essentially a conditioning process, and is particularly applicable to treatment of unseasoned timber. Immediately following treatment by this process, timber can be further treated using one of the processes detailed in I.2.6.2 to I.2.6.4.

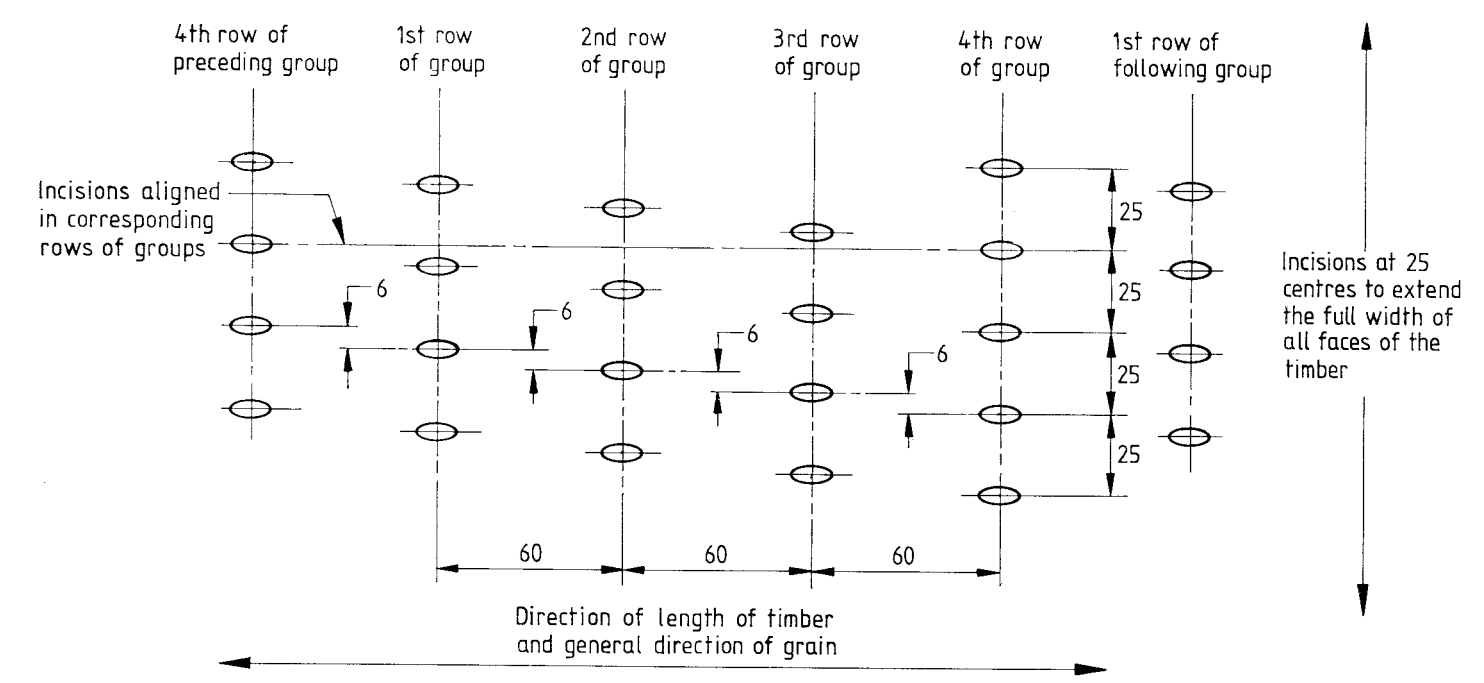
NOTE. The Boulton process is a very severe treatment process, and unless extreme caution is exercised, breakdown of the timber structure may occur. The process should only be used with prior agreement with the customer.

I.2.6.2 Full cell (Bethel) process

In the Bethel process, timber is enclosed in a pressure cylinder, and a vacuum of at least -0.75 bar applied. Hot creosote is introduced into the cylinder until the cylinder is full, and the vacuum is then released.

Next, a pressure of sufficient magnitude (see I.2.3) is applied and maintained long enough to ensure that the requirements regarding penetration and retention (see I.2.5) are met.

The pressure is released and the cylinder emptied of creosote. Finally, a vacuum of not less than -0.75 bar is applied so that the timber is substantially free from surplus creosote before removal from the cylinder.



Dimensions in millimetres (approx.)

Figure I.1 Example spacing of incisions

1.2.6.3 Empty cell (Rueping) process

In the Rueping process, timber is enclosed in a pressure cylinder, and an air pressure greater than atmospheric pressure but not exceeding 4 bar applied. While the gauge pressure is maintained, hot creosote is introduced into the cylinder until the cylinder is full. The pressure is increased to a sufficient magnitude and maintained for long enough to ensure that the requirements regarding retention and penetration (see 1.2.5) are met.

The pressure is then released and the cylinder emptied of creosote. A final vacuum of not less than -0.75 bar is applied for a sufficient period to ensure that the air in the cylinder has expanded and forced out any free creosote from the cells of the timber, reducing the creosote retention.

NOTE. With most permeable timber, creosote retention can be raised by lowering the initial air pressure.

1.2.6.4 Empty cell (Lowry) process

The procedure for the Lowry process is the same as for the Rueping process (1.2.6.3), except that the air in the cylinder is maintained at atmospheric pressure until the cylinder is full.

NOTE. Creosote retention by the Lowry process is normally higher than with the Rueping process.

1.2.6.5 Boulton process (boiling under vacuum)

In the Boulton process, timber is enclosed in a pressure cylinder, and sufficient hot creosote introduced to immerse the timber but ensuring that an air space is left. A vacuum is applied to the cylinder and a sufficiently high temperature maintained so that the water in the timber boils under the vacuum.

The boiling water vapour is condensed and measured in a suitable receiver. When sufficient water has been extracted to achieve the required moisture content in the timber, the process is halted by releasing the vacuum and emptying the creosote from the cylinder.

NOTE. This treatment can be followed immediately by pressure impregnation using any of the processes detailed in 1.2.6.2 to 1.2.6.4.

1.3 Processing by hot-and-cold open tank with creosote types 1, 2 or 3**1.3.1 Treatment materials**

The treatment material should be coal tar creosote conforming to either type 1, type 2 or type 3.

1.3.2 General method

The hot-and-cold open tank process can be carried out in any convenient open tank or drum suitable for the size and quantity of wood to be treated. It is essential to provide some means of lifting the timber in and out of the tank, and to ensure that the timber is completely submerged in the creosote during treatment. The timber is immersed in the creosote and then heated safely to $(90 \pm 5)^\circ\text{C}$ (see caution), causing air to be extracted from the timber by expansion (the vapour pressure within the wood also increases, expelling further air). The temperature is maintained for at least 1 h, depending on the size and permeability of the timber, after which the creosote and timber are

allowed to cool. On cooling, the remaining air in the timber contracts and the vapour pressure decreases, creating a partial vacuum that draws the creosote into the wood.

Caution. Naked flames should not be used for heating. Smoking and other potential sources of ignition should be avoided, because the creosote may be heated above its flash point.

NOTE 1. Practically all of the creosote absorption takes place during the cooling period. For this reason, equivalent results are not obtained if the timber is merely immersed in the hot preservative for steeping.

NOTE 2. Permeable species often absorb much more creosote than necessary. Excess creosote can be removed by reheating the charge to 85°C to 95°C for about 1 h, then removing the timber while still hot. During reheating, up to 50 % of the retained creosote is expelled.

1.3.3 Variations

1.3.3.1 It is permissible to vary the hot-and-cold open tank method in a number of ways. For example, cooling can be achieved more quickly by transferring the timber to a tank of cold preservative, or the timber can be preheated for 1 h or 2 h by any convenient alternative means, such as with hot air, before transferring into cold creosote.

1.3.3.2 A convenient but less efficient variation of the hot-and-cold open tank method, used to treat small numbers of fence posts, is the butt-treatment process. The fence posts are placed upright in a drum with only their ends immersed in the creosote, therefore covering the part of the post which will be in or near the ground, and then heated and cooled as described before.

1.4 Processing by brushing and immersion**1.4.1 Treatment materials**

The treatment material should be coal tar creosote conforming to type 3.

1.4.2 Brushing

NOTE 1. Brushing is the easiest but generally the least effective method of applying wood preservatives.

NOTE 2. In situ brush treatment of outdoor timber is best carried out between late spring and early autumn, after a period of dry weather, when the surface of the timber is warm and dry. The timber is then in its most absorbent state, and the higher prevailing temperatures aid penetration. It is advisable to re-treat the timber periodically.

The creosote should be applied liberally and the timber allowed to absorb as much as possible, particularly into the sapwood and end grain. It is essential to cover the whole of the surface thoroughly and to ensure that the creosote penetrates into all the splits and surface checks. More than one coating should be applied and the timber allowed to dry between applications.

1.4.3 Immersion

The timber should be submerged in cold preservative for the period required by the appropriate commodity specification.

NOTE. The efficiency of this method largely depends on the species, sapwood content and period of immersion. Fairly deep penetration may be obtained with some species of timber if left in the preservative for several days or even weeks. Resistant timbers, however, will merely show surface penetration.

List of references

Normative references

BSI publications

BRITISH STANDARDS INSTITUTION, London

- BS EN 1014 : *Wood preservatives — Creosote and creosoted timber — Methods of sampling and analysis*
- BS EN 1014-1 : 1995 *Procedure for sampling creosote*
- DD ENV 1014-3 : 1996 *Determination of the benzo[a]pyrene content of creosote*
- BS EN 1014-4 : 1996 *Determination of the water extractable phenols content of creosote*
- BS EN 22719 : 1994 *Methods of test for petroleum and its products — Petroleum products and lubricants — Determination of flash point — Pensky-Martens closed cup method*
- BS EN ISO 3696 : 1995 *Water for analytical laboratory use — Specification and test methods*
- BS 188 : 1977 *Methods for determination of the viscosity of liquids*
- BS 410 : 1986 *Specification for test sieves*
- BS 593 : 1989 *Specification for laboratory thermometers*
- BS 604 : 1982 *Specification for graduated glass measuring cylinders*
- BS 658 : 1989 *Specification for apparatus for the determination of distillation range (including flasks and receivers)*
- BS 718 : 1991 *Specification for density hydrometers*
- BS 756 : 1952 *Specification for Dean and Stark apparatus*
- BS 1752 : 1983 *Specification for laboratory sintered or fritted filters including porosity grading*
- BS 1792 : 1982 *Specification for one-mark volumetric flasks*
- BS 2000 : *Methods of test for petroleum and its products*
- BS 2000 : Part 0 : *Standard reagents and thermometers*
- BS 2000 : Addendum 1 : 1987
- BS 2021 : 1980 *Specification for separating and dropping funnels for laboratory use*

Informative references

BSI publications

BRITISH STANDARDS INSTITUTION, London

- BS EN 350 : *Durability of wood and wood-based products — Natural durability of solid wood*
- BS EN 350-1 : 1994 *Guide to the principles of testing and classification of natural durability of wood*
- BS EN 350-2 : 1994 *Guide to natural durability and treatability of selected wood species of importance in Europe*
- BS EN 351 : *Durability of wood and wood-based products — Preservative-treated solid wood*
- BS EN 351-1 : 1996 *Classification of preservative penetration and retention*
- BS EN 351-2 : 1996 *Guidance on sampling for the analysis of preservative-treated wood*
- BS EN 599 : *Durability of wood and wood-based products — Performance of preventive wood preservatives as determined by biological tests*
- BS EN 599-1 : 1997 *Specification according to hazard class*
- BS 1282 : 1975 *Guide to the choice, use and application of wood preservatives*
- DD 239¹⁾ *Code of practice for preservation of timber*

¹⁾ In preparation.

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