

**Specification for
coal-tar-based
hot-applied coating
materials for protecting
iron and steel,
including a suitable
primer**

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Committees responsible for this British Standard

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Foreword

This British Standard has been prepared by ISE/16/-/10. It supersedes BS 4164:1987, which is withdrawn. This new edition of BS 4164 incorporates technical changes only. It does not reflect a full review or revision of this standard, which will be undertaken in due course.

Protection by coal-tar-based materials is the most extensively used method of protecting buried and submerged steel pipes. Materials used in coating have to be properly chosen for performance and compatibility; these and many other requirements that apply are specified in ISO 5256 published by the International Organization for Standardization (ISO). It is evident that materials and practices vary widely in the countries represented on the ISO committee, and a common standard has to allow for the well established practices of the various countries. In this British Standard the aim has been to produce a document that is compatible with the international standard but that also prescribes closer limits for many properties, which experience in the UK has shown to be advantageous.

The use of hot-applied coal-tar-based coating materials is mentioned in a number of British Standards; BS 534 refers to steel pipes, fittings and specials, while BS 4508-1, BS 5834-2 and BS 5834-3 also specify these coating materials for specialized iron and steel articles. Hot-applied bitumen-based coating materials for protecting iron and steel are specified in BS 4147.

WARNING. The coatings specified in this British Standard are not to be used where they will come into contact with water for human consumption.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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 21 and a back cover.

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1 Scope

This British Standard specifies requirements for hot-applied coal-tar-based materials, reinforced with inert non-fibrous fillers, which are used to protect iron and steel against corrosion. Requirements for a primer are included because the performance of hot-applied coating materials depends on its use.

Different grades of material are used for different substrates, conditions of application and service. This standard indicates factors that need to be taken into account in selecting the grade for a particular use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

BS 410-1:2000, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth* (ISO 3310-1:2000).

BS 593, *Specification for laboratory thermometers*.

BS 1796-1:1989, *Test sieving — Method using test sieves of woven wire cloth and perforated metal plate* (ISO 2591-1:1988).

BS 7079-A1:1989, *Preparation of steel substrates before application of paints and related products — Visual assessment of surface cleanliness — Specification for rust grades and preparation grades of uncoated steel substrates and of steel substrates after overall removal of previous coatings* (ISO 8501-1:1988).

BS EN 1426:2000, *Methods of tests for petroleum and its products — Bitumen and bituminous binders — Determination of needle penetration* (BS 2000-49:2000).

BS EN 1427, *Methods of test for petroleum and its products — Bitumen and bituminous binders — Determination of softening point — Ring and ball method* (BS 2000-58).

BS EN ISO 2431, *Paints and varnishes — Determination of flow time by use of flow cups* (BS 3900-A6).

BS EN ISO 13736, *Petroleum products and other liquids — Determination of flash point — Abel closed cup method* (BS 2000-170, ISO 13736).

3 Terms and definitions

For the purposes of this British Standard, the following terms and definitions apply.

3.1

coal-tar-based material

material derived from crude coal-tar, produced only by the high-temperature carbonization of coal in coke ovens

3.2

hot-applied

process of application for materials of such a consistency at ambient temperature that heating is required before application

3.3

inert filler

finely divided mineral powder which is not substantially hygroscopic, not electrically conducting and does not react with other ingredients of the coating material or with the environment in which it is used

3.4

modified coal-tar

material manufactured by the hot dispersion of powdered coal in coal-tar or in suitable coal-tar oils

3.5

primer

material applied as a thin film to metal in order to ensure maximum adhesion of the subsequent protective coating

4 Synthetic primer for cold application

NOTE A synthetic primer for cold application is used with coating materials. This is quick drying and application of the subsequent coating is possible within a short time. The primer should be applied in accordance with the manufacturer's instructions.

4.1 Composition

The synthetic primer for cold application shall consist of chlorinated rubber and plasticizer and colouring matter, together with solvents needed to give a consistency suitable for application by spray, brush or other appropriate method, for example roller or sponge.

4.2 Characteristics

The characteristics of the synthetic primer shall be in accordance with Table 1 when tested in accordance with the given methods of test.

In addition, the primer should provide a bond between the metal and the coating material so that the coating material shall be in accordance with Table 2 when tested in accordance with the given methods of test.

Table 1 — Characteristics of synthetic primer for cold application

Characteristic	Limits	Method of test
Flow time (4 mm flow cup) at 23 °C	35 s to 60 s	BS EN ISO 2431
Flash point (Abel closed cup), minimum	23 °C	BS EN ISO 13736
Volatile matter (105 °C to 110 °C), maximum	75 % loss by mass	Annex A

5 Modified coating materials with inert non-fibrous fillers

NOTE The protective system can be ineffectual if the coating materials are not correctly applied, or if they are applied to an unsatisfactory surface. Guidance on the application of coating materials to iron and steel pipes is given in BS 7873. Guidance on the use of coating materials is given in Annex H.

5.1 Composition

The coating materials shall consist of a uniform mixture of modified coal-tar and inert non-fibrous filler.

The fineness of the inert non-fibrous filler shall be as follows when tested in accordance with BS 1796-1:1989, 7.3:

- passing 90 µm test sieve in accordance with the relevant requirements of BS 410-1:2000 — not less than 93 %;
- passing 250 µm test sieve in accordance with the relevant requirements of BS 410-1:2000 — not less than 99 %.

Table 2 — Tests for coating materials on primed metal

Test	Grade 105/15	Grade 105/8	Grade 120/5	Method of test
Sag, maximum, mm 70 °C, 24 h 80 °C, 24 h	1.5 —	1.5 —	— 1.5	Annex B
Low temperature cracking and disbonding –30 °C –25 °C –20 °C	None — —	— None —	— — None	Annex C
Bend at 0 °C First crack, minimum, mm Initial After heating Disbonded area, maximum, mm ² Initial After heating	20 15 2000 3000	15 10 3000 5000	— — — —	Annex D
Impact Disbonded area, maximum, mm ² 0 °C ^a 25 °C	15000 —	— 10000	— —	Annex E
Peel, initial and delayed, maximum, mm 30 °C 40 °C 50 °C 60 °C 70 °C	3.0 3.0 3.0 — —	— 3.0 3.0 3.0 —	— — — 3.0 3.0	Annex F
Cathodic disbonding in 28 days maximum, mm	5	5	5	Annex G
^a See 5.3.2.				

5.2 Characteristics

The characteristics of the coating materials shall be in accordance with the appropriate grade given in Table 3 when tested in accordance with the given methods of test.

NOTE Guidance on the application and service temperatures of coating materials is given in Annex M.

5.3 Tests

5.3.1 General

The coating materials, in conjunction with the appropriate primer, shall be in accordance with the appropriate grade given in Table 3 when tested in accordance with the given method of test.

5.3.2 Impact tests

If the test specimen fails the impact test (see Table 2) at 0 °C when tested in accordance with Annex E, two further test specimens shall be prepared from the same sample as the failed test specimen and tested at 0 °C. The material shall be deemed to have passed the impact test provided both the additional test specimens pass the test.

Table 3 — Characteristics of coating materials

Characteristic	Grade 105/15	Grade 105/8	Grade 120/5	Method of test
Filler content by ignition, % by mass	25 to 35	25 to 35	25 to 35	Annex I
Density at 25 °C, g/cm ³	1.4 to 1.6	1.4 to 1.6	1.4 to 1.6	Annex J
Softening point (ring and ball), °C	105 to 116	105 to 116	120 to 130 ^a	BS EN 1427
Penetration (total moving mass), 10 ⁻¹ mm				
25 °C, 100 g	10 to 20	5 to 12	1 to 9	Annex K
45 °C, 50 g	15 to 55	8 to 30	3 to 16	
Flow time, s				
230 °C	9 to 16	9 to 16	—	Annex L
240 °C	—	—	9 to 24	

^a The softening point range for this grade may be exceeded by agreement between the manufacturer and the purchaser.

6 Packaging

Primers shall be packed in airtight containers.

The coating material shall be supplied in one of the following forms:

- a) as a solid — packed in easily strippable containers or in steel drums;
- b) as a hot liquid — in bulk tankers.

The manufacturer shall provide instructions for storing and handling of their material.

7 Marking

Containers of coal-tar-based hot-applied coating materials shall be legibly and permanently marked with the following information:

- a) the number and date of this British Standard, i.e. BS 4164:2002¹⁾;
- b) the grade number of the material (e.g. grade 120/5),
- c) the manufacturer's identity;
- d) the manufacturer's batch number.

8 Sampling

NOTE When samples of coating material or primer are required for testing, the purchaser and the supplier should agree on the number of packages to be sampled and the procedure to be adopted. The samples so taken should be identified by the supplier and one-half retained by the purchaser for the purpose of making such tests as they require.

During the cooling period after manufacture, some settling of the filler in the coating materials can occur. In order to ensure that they are representative, test samples shall be made up of equal amounts taken from the top, middle and bottom of the container.

A sample of primer shall be obtained by drawing not less than 1 L either at the filling stage or from one or more previously unopened containers. A suitable clean, dry, airtight container shall be filled with the sample so as to leave an ullage of approximately 5 %. Each sample container shall be sealed and marked with full details and the date of sampling.

¹⁾ Marking BS 4164:2002 on or in relation to a product represents a manufacturer's declaration of conformity, i.e. a claim by or on behalf of the manufacturer that the product meets the requirements of the standard. The accuracy of the claim is solely the claimant's responsibility. Such a declaration is not to be confused with third-party certification of conformity.

Annex A (normative) Determination of volatile matter

A.1 Principle

The primer is heated and the volatile matter of the primer is calculated as a percentage by mass of the primer.

A.2 Apparatus

A.2.1 *Tared flat-bottomed circular dish*, approximately 75 mm in diameter.

A.2.2 *Oven*.

A.2.3 *Desiccator*.

A.3 Procedure

Weigh to the nearest milligram between 1.0 g and 2.0 g of the primer into the tared flat-bottomed circular dish (**A.2.1**). Heat the dish and its contents in the oven (**A.2.2**) at the temperature given in Table 1 for 3 h. Allow the dish to cool to room temperature in the desiccator (**A.2.3**) and reweigh to the nearest milligram.

A.4 Calculation

Calculate the volatile matter, V , as a percentage by mass of the primer using the following formula:

$$V = 100 \frac{M_1 - M_2}{M_1}$$

where

M_1 is the mass of sample before heating;

M_2 is the mass of sample after heating.

Annex B (normative) Sag test

B.1 Principle

A primed and coated test plate is heated and the maximum sag of the coating is measured. This test assesses the integrity of the coating material at high temperatures.

B.2 Apparatus

B.2.1 *Oven*.

B.2.2 *Rule*, graduated in millimetres.

B.3 Preparation of test specimens

Prepare one test plate, measuring at least 300 mm × 300 mm × 3 mm, in accordance with Annex N, prime it in accordance with Annex P and coat it with coating material prepared in accordance with Annex Q. Leave a 15 mm uncoated border around the four edges of the plate and draw three lines parallel with one edge at 75 mm intervals across the surface of the coating material and continued on the uncoated surface of the plate to the edges. Draw the lines in such a way that the prepared surface is not damaged.

B.4 Procedure

Store the plate vertically in an oven (B.2.1). Maintain the oven at the temperature and for the period given for the appropriate grades in Table 2. At the end of this period remove the plate and allow it to cool to room temperature. Measure the maximum sag of each line on the plate with the rule (B.2.2).

NOTE In cases of disagreement, the manufacturer and the purchaser should each repeat the test on two fresh specimens.

B.5 Reporting

Report the sag of the coating material as the average of the maximum sags of the three lines.

Annex C (normative)

Low temperature test for cracking and disbonding

C.1 Principle

A primed and coated test plate is subjected to low temperatures and if it does not crack or disbond the integrity of the coating material is maintained.

C.2 Apparatus

C.2.1 Flaw detector, fitted with a soft metallic bristled straight brush approximately 75 mm long and 6 mm wide with bristles 6 mm long, adjusted to provide a voltage of 10 kV so arranged that the maximum short circuit current is not greater than 8 mA.

C.2.2 Refrigerator.

C.3 Preparation of test specimens

For the low temperature test use the test plate that has been used in the sag test described in Annex B. Check the coating material with the flaw detector (C.2.1) before refrigeration.

C.4 Procedure

Place the plate in the refrigerator (C.2.2) at room temperature. Reduce the temperature of the refrigerator to the test temperature given in Table 2 in not less than 30 min and maintain that temperature for 6 h.

At the end of this period remove the plate and allow it to reach room temperature.

C.5 Examination

C.5.1 Cracking

Examine the plate for evidence of cracking by means of the flaw detector (C.2.1). If the flaw detector is not equipped with voltage indication, set it to give a 12 mm spark in air to a steel plate. Pass the flaw detector over the coated specimen at approximately 6 mm distance from the coated steel surface. Ignore edge effects and flaws detected before refrigeration.

C.5.2 Disbonding

C.5.2.1 Examine the plate for evidence of complete loss of bond between the panel and the coating material by inserting a knife blade under an edge of the coating material and levering gently. In cases of complete disbonding, the complete unbroken layer of coating material becomes detached.

C.5.2.2 Examine the plate for evidence of partial loss of bond beneath the unbroken coating material by allowing the end of a wooden pencil to fall freely from 50 mm on to the coated side of the plate. Pierce hollow-sounding areas with a knife to determine the area of detachment.

Annex D (normative)

Bend tests

D.1 Principle

A primed and coated test plate is subjected to a deflecting load to induce bending. If the coating does not crack before a minimum deflection nor disbond more than a specified area the integrity of the coating material is maintained.

D.2 Apparatus

D.2.1 *Water/ice bath or refrigerator.*

D.3 Initial test

D.3.1 *Preparation of specimens*

Prepare four test plates, measuring 250 mm × 100 mm × 1.5 mm, in accordance with Annex N, prime them in accordance with Annex P and coat them with coating material prepared in accordance with Annex Q.

D.3.2 *Procedure*

D.3.2.1 Store the plates at 0 ± 1 °C in the water/ice bath or refrigerator (**D.2.1**) for a minimum of 6 h. Then remove them and test them immediately.

D.3.2.2 Test the plates for deflection by supporting them on 3 mm radius knife edges which are spaced on 240 mm centres. Apply the deflecting load centrally across the plate by means of a 12 mm radius mandrel at the rate of 1 mm/s (to produce tension in the coating) until cracking occurs, as indicated by an electrical flaw detector operated continuously as described in **C.5.1**. Continue the deflection to a maximum distance of 38.0 mm and note the deflection producing the first crack.

Remove the specimen from the machine for examination. Remove all disbonded coating from the plate and measure the area of metal exposed on the four plates.

D.3.3 *Reporting*

Report the following average results:

- a) the deflection producing the first crack (in millimetres);
- b) the disbonded area (in square millimetres).

D.4 “After heating” test

D.4.1 *Preparation of specimens*

Prepare four test plates, measuring 250 mm × 100 mm × 1.5 mm, in accordance with Annex N, prime them in accordance with Annex P and coat them with coating material, which has been maintained at the application temperature recommended by the manufacturer for 2 h, in accordance with **Q.3**.

D.4.2 *Procedure*

Follow the procedure described in **D.3.2**.

D.4.3 *Reporting*

Report the following average results:

- a) the deflection producing the first crack (in millimetres);
- b) the disbonded area (in square millimetres).

Annex E (normative)

Impact test

E.1 Principle

A primed and coated test plate is subjected to an impact load. If the coating does not disbond more than a specified area the integrity of the coating material is maintained.

E.2 Apparatus

E.2.1 Refrigerator.

E.2.2 Water bath.

E.2.3 Lint-free paper towel.

E.2.4 Block of wood.

E.2.5 Steel ball, weighing 630 g to 650 g.

E.2.6 Rule, graduated in millimetres.

E.3 Preparation of test specimen

E.3.1 General

Prepare a test plate, measuring approximately 300 mm × 300 mm × 12.5 mm, in accordance with Annex N, prime it in accordance with Annex P and coat it with coating material prepared in accordance with Annex Q.

E.3.2 For test at 0 °C

Allow the plate to reach room temperature and then maintain it at a temperature of 0 ± 1 °C in the refrigerator (**E.2.1**) for at least 6 h before testing. Remove the plate quickly from the refrigerator, dry it with a lint-free paper towel (**E.2.3**) if necessary and subject it to the impact test.

E.3.3 For test at 25 °C

Allow the plate to reach room temperature and then maintain it at a temperature of 25 ± 1 °C in the water bath (**E.2.2**) for at least 30 min before testing. Remove the plate quickly from the water bath, dry it with a lint-free paper towel (**E.2.3**), and subject it to the impact test.

E.4 Procedure

E.4.1 For test at 0 °C

Support the plate on a flat horizontal surface of the block of wood (**E.2.4**), with the coated face uppermost. Drop a 630 g to 650 g steel ball (**E.2.5**) with a well polished spherical surface from a height of 2.45 m above the surface of the plate, so as to strike the coating material at the centre of the plate.

E.4.2 For test at 25 °C

Proceed in accordance with **E.4.1** but drop the steel ball so as to strike the coating material at a point approximately 100 mm along the diagonal from one corner of the plate. Repeat the test in each of the other three quarters.

E.5 Assessment and recording

Examine the plate for evidence of disbonding in accordance with **C.5.2**. Remove all disbonded coating, make a trace of the area disbonded and measure its area in square millimetres using the rule (**E.2.6**). Record the area of disbonded coating for impact tests at 0 °C and the average of the four areas of disbonded coating for impact tests at 25 °C.

Annex F (normative)

Peel tests

F.1 Principle

A primed and coated test plate is cut and the coating peeled away from the surface of the plate. If the coating does not break beyond a maximum length of peel, the integrity of the coating material is maintained.

F.2 Apparatus

F.2.1 *Oven.*

F.2.2 *Water bath.*

F.2.3 *Stiff scraper*, with sharpened square-ended blade of approximately 20 mm width.

F.3 Preparation of test specimens

Prepare two test plates, measuring approximately 300 mm × 300 mm × 12.5 mm, in accordance with Annex N, prime them in accordance with Annex P and coat them with coating material prepared in accordance with Annex Q. After applying the coating material, allow the plates to cool to room temperature.

F.4 Procedure

Use one test plate, without further treatment, for the initial peel test. Store the second test plate horizontally, with the coated side up, in the oven (**F.2.1**) at 70 ± 2 °C for 72 h. At the end of this period remove the plate, allow it to cool to room temperature and use it for the delayed peel test.

Carry out the peel test by immersing the plate in the water bath (**F.2.2**) for approximately 30 min at the lowest temperature given for the particular grade of coating material in Table 2. At the end of this period remove the plate from the bath and immediately test for peel as follows.

Lay the plate horizontal. Make two parallel cuts through the coating material approximately 20 mm apart and 100 mm long toward the centre of the plate.

Using the scraper (**F.2.3**), make a cut through the coating for the full width between the parallel cuts. With a gentle levering action separate an approximately 15 mm long strip of coating from the steel plate.

Carefully turn the blade of the scraper and the separated coating in an upward direction until they are vertical. Gripping the coating and blade between finger and thumb, pull upwards until the coating breaks.

Measure the amount of peeling from the point where the separation through leverage and cutting had ceased to the line of breakage of the coating.

Carry out at least two tests at each temperature.

NOTE In case of failures, more tests may be carried out, extending the parallel cuts if necessary and reporting the average results obtained.

Repeat this procedure, making new parallel cuts, separated by at least 15 mm from the cuts for the preceding test, for each of the successive temperatures given in Table 2.

Annex G (normative) Cathodic disbonding test

G.1 Principle

A primed and coated test plate is subjected to a voltage of -1.500 V to induce disbonding. If the coating does not disbond more than a specified extent the integrity of the coating material is maintained.

G.2 Apparatus

G.2.1 *Stabilized d.c. power unit*, having a controlled voltage output between 0 V and 10 V and a current capacity sufficient to supply 20 mA simultaneously to each test site in circuit.

G.2.2 *Digital voltmeter*, range 1.999 V ($3\frac{1}{2}$ digit), input impedance 10^3 M Ω , accuracy 0.1 % \pm digit at 23 ± 1 °C. Maximum offset input current not greater than 10 A to 11 A.

G.2.3 *Variable resistor*, range 5 k Ω \pm 10 %, 1 W for each test site.

G.2.4 *Fixed resistor*, 10 Ω \pm 1 %, 1 W for each test site.

G.2.5 *Fixed resistor*, 510 Ω \pm 2 %, 1 W for each test site.

G.2.6 *Flaw detector* (as described in C.5.1).

G.2.7 *Reference electrode*, saturated calomel type, constructed from glass or plastics with porous plug.

G.2.8 *Platinum wire*, of 0.8 mm diameter, one 75 mm length for each test site.

G.2.9 *Rigid plastics tube*, of 50 mm nominal bore, one 60 mm length for each test site.

G.2.10 *Elastomeric adhesive*, for fixing the plastics tube solution-containers to the test surface.

NOTE Suitable materials are two-part polysulphide rubber and silicone rubber.

G.2.11 *Twist drill*, of 6 mm diameter.

G.2.12 *Lint-free paper towel*.

G.3 Reagents

G.3.1 *Sodium chloride solution* (3 % m/V).

G.3.2 *Phenolphthalein acid/base indicator*.

G.4 Procedure

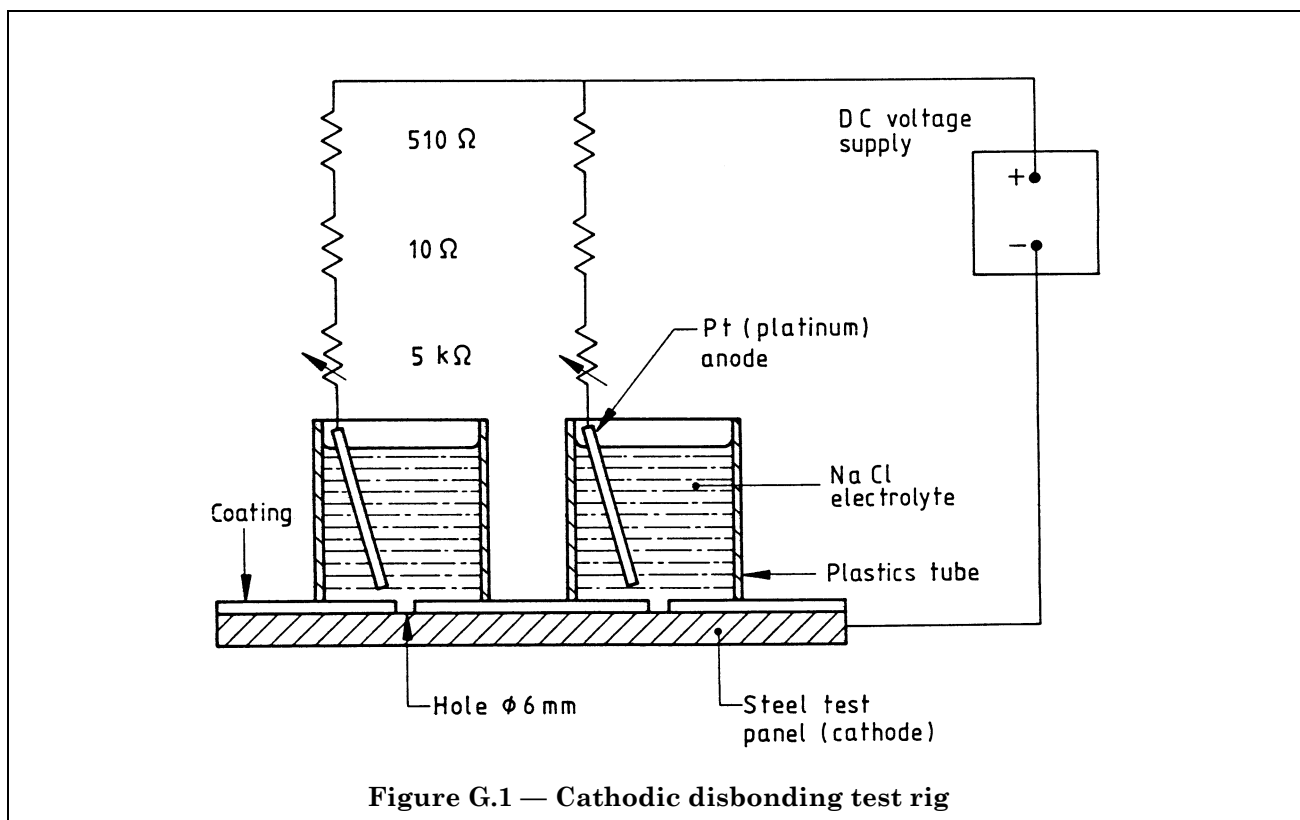
Prepare one test plate, measuring at least 200 mm \times 100 mm \times 15 mm, in accordance with Annex N, prime it in accordance with Annex P and coat it with coating material prepared in accordance with Annex Q.

To ensure freedom from accidental damage, test the prepared plate with the flaw detector (G.2.6) in accordance with C.5.1.

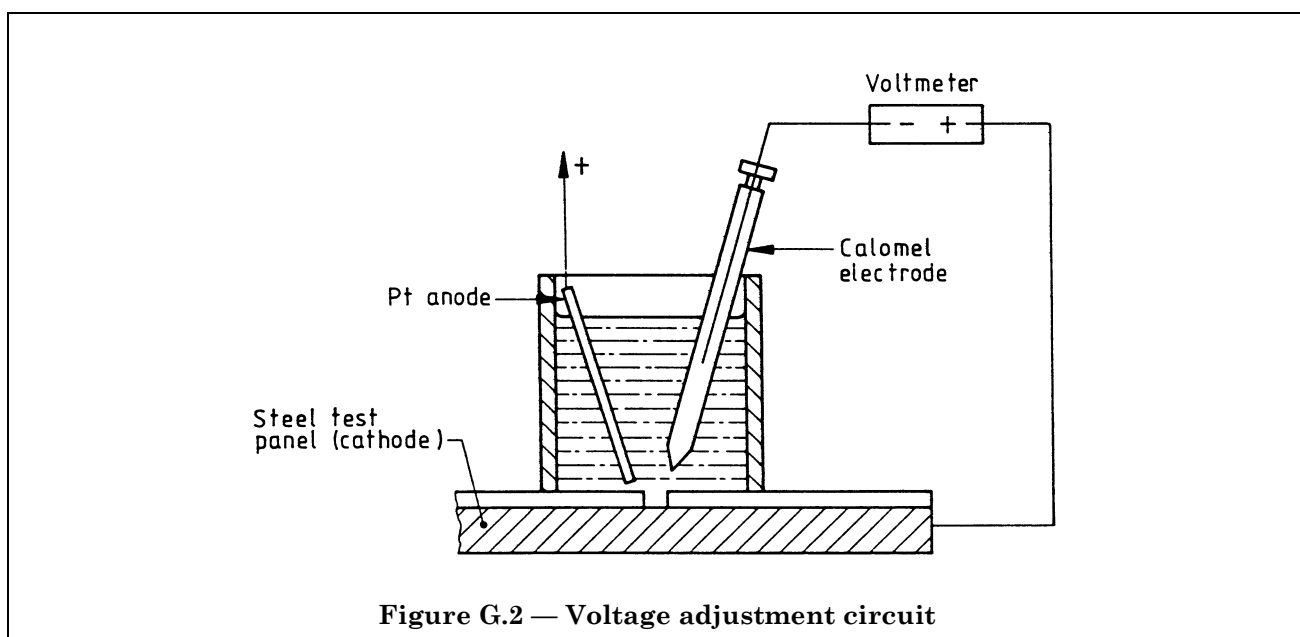
Affix two plastics tubes (G.2.9) perpendicular to the coated surface using a suitable elastomeric adhesive (G.2.10). Place them at a minimum distance of 33 mm from the panel ends and from each other with their centres on the centreline of the panel width. Leave overnight to allow the adhesive to cure fully.

Drill a 6 mm hole through the coating material (G.2.11) to the metal surface in the centre of each test site, as a pre-damage area.

Fill each plastics tube (G.2.9) to a depth of approximately 50 mm with sodium chloride solution (G.3.1) and connect the apparatus as shown in Figure G.1.



Connect the voltmeter (G.2.2) as shown in Figure G.2 and, with the porous tip of the reference electrode (G.2.7) placed within 10 mm of the hole in the coating material, adjust the variable resistor (G.2.3) until the voltmeter reads -1.500 V with respect to the calomel electrode.



At intervals of 24 h record the voltmeter reading and adjust the variable resistor to correct any drift from the -1.500 V setting.

Continue the test for 28 days, maintaining the temperature at 20 ± 5 °C. After this period assess disbonding at both test sites in accordance with **G.5**. Then assess the bonding in accordance with **G.5**.

G.5 Assessment

Remove the plastics tube from each test site and wipe along the surface of the coating using a lint-free paper towel (**G.2.12**) and cathode area material.

Make two parallel incisions through the coating and 12.5 mm apart across the panel so as to include the predamage area. The cuts should extend 50 mm on each side of the predamage area.

Using a square ended pallet knife insert it into the centre portion of the predamage area, between the parallel cuts, down to the metal. Using a gentle levering action, lift the strip of coating, if possible, with a slow peeling action and then grip the coating between the blade and thumb and continue the peeling action until the coating breaks.

Repeat the peeling test in the opposite direction and then repeat the procedure at an angle of 90° to the first test.

Apply one spot of phenolphthalein acid/base indicator (**G.3.2**) to the exposed metal surface at the outside edge and allow it to flow towards the predamaged area. The purple boundary indicates the extent of disbonding.

G.6 Reporting

Report the extent of disbonding as the average distance in millimetres from the edges of the predamaged areas. Alternatively, if the coating is strongly adherent to the steel substrate, take the average distance at which the coating breaks as the extent of disbonding.

Annex H (informative)

Recommendations for the use of coating materials

Coating materials are heavy-duty products for application at a minimum thickness of 2.4 mm to provide long term protection underground and in submarine installations. They are applied to iron and steel used over a wide range of service temperatures. They are particularly suitable for flood-coating previously primed products. Agitation of coating materials in the molten state is necessary to prevent settling of the filler. When these materials are applied externally to pipes it is usual to incorporate one or more reinforcing layers of inert fabric.

All materials should be applied in accordance with the manufacturer's instructions. Care should be exercised to ensure there is no mixing of material from different sources or of different types. In particular, it should be recognized that the chemical and physical characteristics of bitumen-based coatings differ from those of coal-tar-based coatings and that the two kinds of coating should not be blended in protective coatings. It is also essential to clean out plant thoroughly when the use of coal-tar coating materials follows that of bitumen coating materials or vice versa.

Annex I (normative) Determination of filler content by ignition

I.1 Principle

The coating material is heated and the filler content by ignition of the coating material is calculated as a percentage by mass of the original sample of coating material.

I.2 Apparatus

I.2.1 *Tared pre-ignited silica crucible*, approximately 40 mm in diameter and 30 mm deep.

I.2.2 *Cold muffle furnace*.

I.2.3 *Desiccator*.

I.3 Procedure

Weigh to the nearest milligram between 1 g and 1.5 g of coating material into the crucible (**I.2.1**). Place the crucible in the cold muffle furnace (**I.2.2**) with good ventilation and increase the temperature to 700 °C to 750 °C over approximately 2 h. Maintain that temperature for not less than 1 h. Then remove the crucible from the muffle furnace and allow it to cool in the desiccator (**I.2.3**). Weigh the crucible and ash to the nearest milligram.

Report the filler content by ignition as the percentage by mass of the original sample of coating material.

I.4 Calculation

Calculate the filler content, F , as a percentage by mass of the coating material using the following formula:

$$F = 100 \frac{M_2}{M_1}$$

where

M_1 is the mass of sample before heating;

M_2 is the mass of sample after heating.

Annex J (normative) Determination of density at 25 °C

J.1 Principle

The coating material is melted and a moulded specimen made. The moulded specimen is then suspended in water and the density calculated from the weight of the specimen in and out of the water.

J.2 Apparatus

J.2.1 *Mould*, of polished brass, as shown in Figure J.1, assembled and placed on a polished brass plate.

J.2.2 *Melting pot*, of steel, of approximately 80 mm diameter and 100 mm height, with a loose fitting lid.

J.2.3 *Balance with hook*.

J.3 Procedure

Break 250 g to 300 g of coating material into pieces not greater than 15 mm in size. Heat them slowly in the melting pot (J.2.2), keeping the lid on when not stirring. Until the pieces have melted, stir continually to avoid local over-heating and excessive loss of vapour. Thereafter stir occasionally.

When the temperature of the fluid material has reached 60 °C to 70 °C above the expected softening point, pour the fluid material carefully into the mould (J.2.1), avoiding the inclusion of air bubbles, until the mould is almost full.

NOTE It is convenient to fill the rings for the softening point tests, the cylinder for the penetration test and the mould for the density test at the same time.

Allow the material and the mould to cool to ambient temperature and then part the mould.

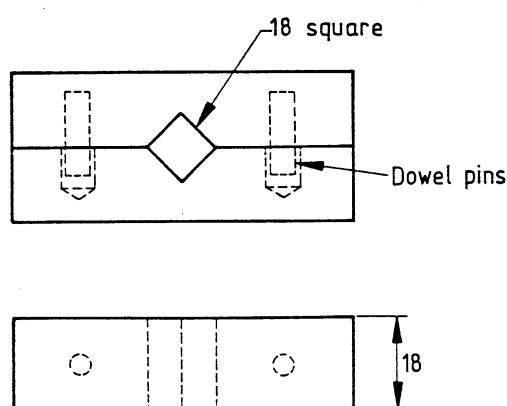


Figure J.1 — Mould for sample for density determination

Suspend the moulded specimen from the hook on the balance (J.2.3) using a suitable length of nylon thread, and weigh it to the nearest milligram.

Add a few drops of wetting agent to a small beaker of water at 25 °C. Place the beaker on a bridge across the balance pan. Suspend the moulded specimen from the hook on the balance so that it is fully immersed in the water and not touching any part of the beaker. Carefully remove all air bubbles adhering to the specimen and to the thread.

Weigh the specimen to the nearest milligram while it is suspended in the water.

J.4 Calculation

Calculate the density of the coating material at 25 °C, D , (in g/cm³) using the following formula:

$$D = \frac{M_a}{M_a - M_w}$$

where

M_a is the mass of sample in air;

M_w is the mass of sample in water at 25 °C.

Annex K (normative)

Determination of penetration

K.1 General

This method is based on the penetration test for bitumen specified in BS EN 1426:2000, but is modified for the following reason. Coal dispersion pitches have a tendency to form a hard thin skin while hot, and penetration values determined by the above method can show a wide variation, dependent on the preparation of the sample, which is not easy to control, and on the point chosen on the surface for the test. The behaviour and performance of these coal dispersion pitches is a function of the body of the material and not of any surface skin.

K.2 Principle

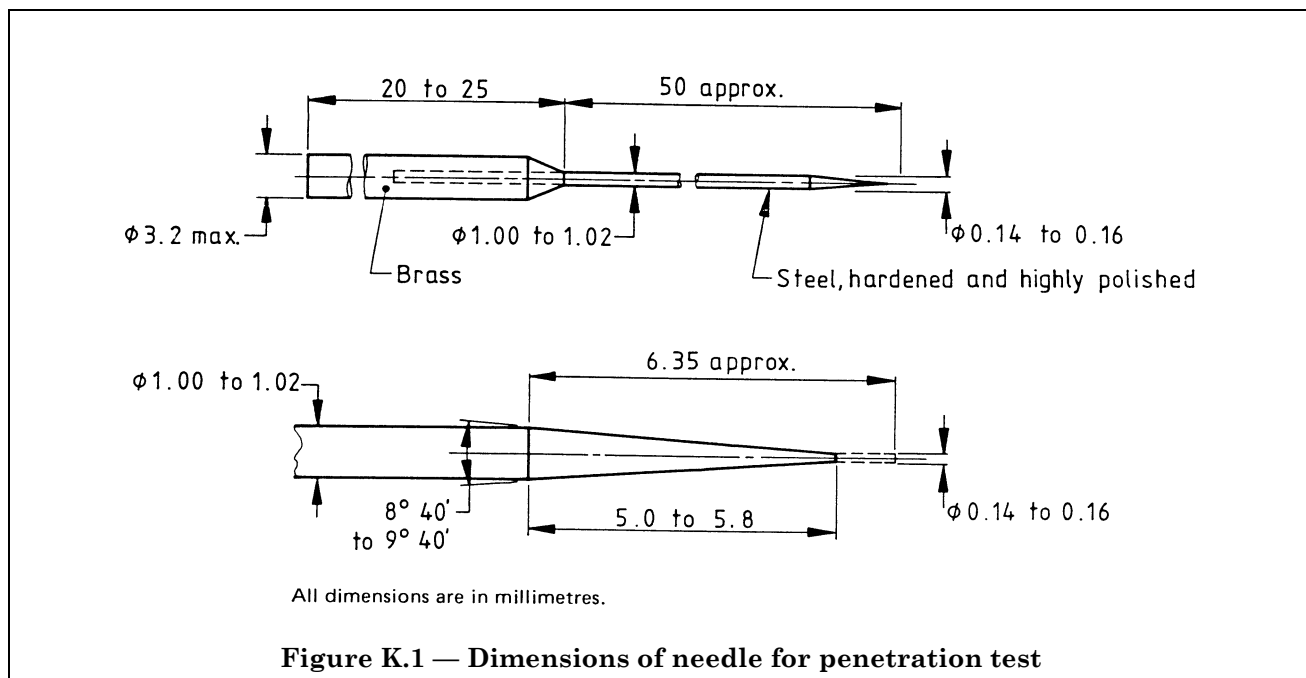
The coating material is melted and a poured into a sample cylinder. The depth of penetration of a needle into the sample is then measured for a specific time period.

K.3 Apparatus

K.3.1 Penetration needle, of the dimensions given in Figure K.1 and, if it is to be submitted to the National Physical Laboratory for certification, bearing an identification number on the shank.

K.3.2 Penetrometer, which permits the needle-holder to move in the guide without appreciable friction and which is capable of indicating the depth of penetration to the nearest 0.1 mm. The total moving mass (needle, needle-holder, and superimposed weight) is 50 ± 0.25 g or 100 ± 0.25 g in accordance with Table 3.

K.3.3 Constant temperature water-bath, having a capacity of not less than 10 L and regulated at the temperature of test ± 0.1 °C. The bath has a perforated shelf supported in a position not less than 50 mm from the bottom and not less than 100 mm below the top of the bath.



K.3.4 Transfer dish, for the sample container, i.e. a dish or tray of a capacity that ensures complete immersion of the container during the test. It is provided with means to ensure a firm bearing and prevent rocking of the container.

K.3.5 *Sample containers*, of steel or brass, open cylinders of approximately 3 mm wall thickness, 40 mm internal diameter and 40 mm depth. The two ends are machine-finished perfectly smooth and perpendicular to the axis. A suitable flat brass plate is provided.

NOTE The container can readily be prepared from a section of pipe.

K.3.6 *Bath thermometer*, in accordance with the appropriate requirements of BS EN 1426.

K.3.7 *Timing device*, of sufficient accuracy to meet the requirements of the repeatability of the method.

K.3.8 *Melting pot*, of steel, of approximately 3 mm wall thickness, 80 mm diameter and 100 mm height, with a loose fitting lid.

K.4 Procedure

Break 250 g to 300 g of coating material into pieces not greater than 15 mm in size. Heat them slowly in the melting pot (**K.3.8**), keeping the lid on when not stirring. Until the pieces have melted, stir continually to avoid local overheating and excessive loss of vapour. Thereafter stir occasionally.

The brass plate (see **K.3.5**) shall be well polished. Place the sample container (**K.3.5**) on the plate so that close contact is maintained between rim and plate. When the temperature of the coating material is approximately 60 °C to 70 °C above the expected softening point, give it a final stir and reject the first two or three grams. Fill the sample container about three-quarters full and allow it to cool to ambient temperature.

NOTE It is convenient to fill the cylinder for the penetration test, the rings for the softening point tests and the mould for the density test at the same time.

Place the container, inverted, in a transfer dish in the constant temperature water-bath (**K.3.3**), which is kept at the temperature given in Table 3 (**K.3.6**), for 1 h.

Clean the penetrometer needle (**K.3.1**) with a suitable solvent, dry it and insert it into the penetrometer (**K.3.2**). Load the needle-holder to bring the total moving mass to that given in Table 3. Place the transfer dish (**K.3.4**) containing the sample container and filled with water from the constant temperature water-bath on the table of the penetrometer. Slowly lower the needle until the tip just makes contact with its image on the surface of the sample. Position the needle at least 10 mm from the edge of the dish and 10 mm from any previous hole.

NOTE A suitably placed light is a help in obtaining precise adjustment of the needle to the sample surface.

Depending on the type of penetrometer used, either note the dial reading or return it to zero. Then quickly release the needle-holder. Leave it free and using the timing device (**K.3.7**) time for 5 ± 0.1 s and then lock it. Read the depth of penetration in tenths of a millimetre from the scale and record it.

Repeat the procedure so as to obtain five readings within the limits of repeatability given in Table 3 of BS EN 1426:2000.

It is important to ensure that the sample container does not move or rock during the test. If it does move in any way, discard the reading.

NOTE When not in use the needle should be removed from the penetrometer and protected from corrosion and damage. Frequent examination of the needle profile should be made to ensure that it is in accordance with **K.3.1**.

K.5 Reporting

Report the mean value obtained as the penetration at the given temperature and loading.

Annex L (normative)

Determination of flow time

L.1 Principle

The coating material is melted and the viscosity determined by noting the time when the coating flow breaks during its descent from a cup.

L.2 Apparatus

L.2.1 *Zahn no. 4 viscometer.*

L.2.2 *Partial immersion Celsius thermometer, conforming to BS 593.*

L.2.3 *Stop-watch.*

L.3 Procedure

Maintain the coating material, melted in accordance with **Q.3**, at 230 ± 2 °C or 240 ± 2 °C (**L.2.2**), avoiding prolonged exposure in an open vessel. Immerse the cup of the viscometer (**L.2.1**) in the coating material and move it around for at least 5 s. Empty by holding the cup horizontally and refill. Repeat the operation.

Lift the cup above the liquid level quickly, keeping it vertical and free from draughts, and start the stop-watch (**L.2.3**) simultaneously. Stop the stop-watch when the stream of sample first breaks.

L.4 Reporting

Report the interval in seconds recorded by the stop-watch as the viscosity of the sample.

Annex M (informative)

Guidance on the application and service temperatures of coating materials

For application by flooding or other means, the temperature of the coating material should be such that the viscosity is controlled to give the thickness of coating required, and not so high as to cause excessive fuming. No grade of material should be heated above 265 °C nor applied above the maximum application temperature given in Table M.1.

In general, materials of higher softening point or lower penetration are intended for use under higher temperature conditions. Grade 105/15 is suitable at normal and lower than normal ambient temperatures in temperate climates.

Grade 105/8 is suitable at ambient temperatures in both temperate and hotter climates. Grade 120/5 is designed for use at elevated service temperatures up to 80 °C, or up to 115 °C in the case of off-shore pipelines when an additional concrete antibuoyancy coating material is used. Under these conditions a degree of hardening can occur early in use. The manufacturer of the product should be consulted as to its suitability under particular conditions.

Table M.1 — Application temperatures for coating materials

Grade of coating material	Maximum application temperature °C
105/15	250
105/8	250
120/5	260

Annex N (normative)

Preparation of steel surfaces for the tests described in Annex B to Annex G

Prepare the steel surfaces used in the tests described in Annex B to Annex G using the following method.

First free the surface of the steel plate from all oil and grease. Then abrasive blast clean the surface so that it conforms to Sa 2½ of BS 7079-A1:1989, and the peak to trough profile is $75 \pm 25 \mu\text{m}$.

NOTE 1 This quality level is roughly equivalent to the near-white standard of the Steel Structures Painting Council (USA) and the Sa 2½ standard of the Swedish standard SS 055900.

NOTE 2 It is important to ensure that blasted steel surfaces are free from traces of previous coating materials and do not subsequently become contaminated with oil or grease.

Annex P (normative)

Priming of steel surfaces for the tests described in Annex B to Annex G

P.1 Principle

A prepared steel surface is covered with a quick drying primer which is then allowed to dry.

P.2 Apparatus

P.2.1 *Clean flat bristle brush*, of 25 mm width.

P.3 Procedure

After preparing the steel surfaces in accordance with Annex N, prime them using the appropriate primer specified in Clause 4.

Apply the primer with the clean flat bristle brush (**P.2.1**) to the plates lying horizontally. Apply the primer at the rate of coverage recommended by the manufacturer, in such a manner that the surface is uniformly covered with an even film free from air bubbles.

Allow the primer to dry at an air temperature of not less than 15 °C, in a well ventilated atmosphere having a relative humidity not greater than 60 %.

When the film is hard dry, apply coating material to the plate in accordance with Annex Q, not less than 1 h after applying the primer.

Annex Q (normative)

Application of coating material for the tests described in Annex B to Annex G

Q.1 Principle

The coating material is melted and poured over a primed steel surface to cover the specimen with a given thickness of coating.

Q.2 Apparatus

Q.2.1 *Container and lid*, of uniform cross section and of diameter not less than 150 mm nor more than 300 mm.

Q.3 Procedure

Break 2 kg to 15 kg of the coating material, according to the number of tests required, into pieces not greater than 50 mm in size. Melt the pieces in the container (**Q.2.1**).

NOTE 1 Heating may be by any convenient method such as fluidized or liquid bath, electric hotplate or gas.

Heat the container so that the coating material reaches the application temperature recommended by the manufacturer within 2 h. If gas is used, interpose a steel plate of not less than 6 mm thickness between the container and the gas flame.

Stir the material frequently until it reaches the application temperature recommended by the manufacturer, replacing the lid between stirs. Immediately it has reached the application temperature, pour the coating material over the primed surface of each plate, held horizontally and at a temperature between 15 °C and 30 °C, in such a manner that fresh enamel constantly strikes the specimen surface.

Apply the coating material to a thickness of 1.5 mm to 2.5 mm to each test specimen.

NOTE 2 It is convenient to adjust to this thickness immediately after coating by sweeping off the surplus hot enamel with a warmed blade moved along guide rails set for the required thickness.

Q.4 “After heating” bend tests

For the “after heating” bend tests, maintain a depth of not less than 100 mm of enamel at the application temperature recommended by the manufacturer. If there is insufficient enamel left after coating the remainder of the plates, add further pieces of enamel and bring the contents of the container back to the application temperature. During the 2 h heating period, stir the enamel with a metal bar at intervals of 15 min. Then apply the coating material.

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ISO 5256:1985, *Steel pipes and fittings for buried or submerged pipelines — External and internal coating by bitumen or coal tar derived materials.*

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