
International Standard



5256

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Steel pipes and fittings for buried or submerged pipelines — External and internal coating by bitumen or coal tar derived materials

Tubes et accessoires en acier utilisés pour canalisations enterrées ou immergées — Revêtements externe et interne au moyen de matériaux hydrocarbonés

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Foreword

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Steel pipes and fittings for buried or submerged pipelines — External and internal coating by bitumen or coal tar derived materials

1 Scope

This International Standard specifies the bitumen and coal tar derived coatings suitable for protecting the external and internal surfaces of steel pipes and fittings, either individually or after assembly, and used in buried pipelines or conduits, or underground galleries, or submerged, and also the conditions for the application of such protective coatings.

In the case of submerged pipes, special provisions shall be taken with regard to laying or immersion conditions (depth, fresh water, salt water).

These specifications cover use for the transport of fluids the temperatures of which are within the following limits :

- a) $-10\text{ }^{\circ}\text{C}$, $+80\text{ }^{\circ}\text{C}$ ¹⁾ if only an external coating is applied;
- b) $0\text{ }^{\circ}\text{C}$, $+40\text{ }^{\circ}\text{C}$ if an internal coating is applied (only or additionally).

In a case where different temperatures are required, the bitumen or coal tar derived materials to be used shall present characteristics to be defined by agreement between the parties concerned.

The internal protective coatings of pipes intended for the transport of potable water and food products shall satisfy the public hygiene criteria of the country of use.

2 Field of application

2.1 Types of pipes to be coated

The types of pipes to which this International Standard is applicable include both welded and seamless pipes of non-alloy steels used for the conveyance of fluids. In particular this International Standard is applicable to pipes covered by standards mentioned in clause 3.

2.2 Types of fittings to be coated

The types of fittings to which this International Standard is applicable are mainly bends, tees, reducers and collars.

3 References

ISO 559, *Welded or seamless steel tubes for water, sewage and gas.*

ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.*

ISO 630, *Structural steels.*

ISO 719, *Glass — Hydrolytic resistance of glass grains at 98 °C — Method of test and classification.*

ISO 2546, *Seamless plain end tubes made from unalloyed steel and without quality requirements.*

ISO 2547, *Welded plain end tubes made from unalloyed steel and without quality requirements.*

ISO 2604/2, *Steel products for pressure purposes — Quality requirements — Part 2 : Wrought seamless tubes.*

ISO 2604/3, *Steel products for pressure purposes — Quality requirements — Part 3 : Electric resistance and induction-welded tubes.*

ISO 2604/6, *Steel products for pressure purposes — Quality requirements — Part 6 : Submerged arc longitudinally or spirally welded steel tubes.*

ISO 3183, *Oil and natural gas industries — Steel line pipe.*

ISO 3419, *Non-alloy and alloy steel butt-welding fittings.*

ISO 4602, *Textile glass — Woven fabrics — Determination of number of yarns per unit length of warp and weft.*

ISO 8501/1, *Preparation of steel substrates before application of paints and related products — Visual assessment of rust grades and of preparation grades — Part 1: Rust grades and preparation grades of uncoated steel substrates and of steel substrates after overall removal of previous coatings.*²⁾

1) The basic materials which are most commonly used for making up the protective coating are suitable for temperatures of use between the limits -10 to $+60\text{ }^{\circ}\text{C}$. Studies are being undertaken to define the characteristics of the materials to be used at temperatures between 60 and $80\text{ }^{\circ}\text{C}$.

2) At present at the stage of draft.

4 Description of coatings

4.1 External coating

The external coating consists of

- a) a primer or bonding coat based on petroleum bitumen or synthetic resin, of the types defined in 5.2.1 and table 1;
- b) one or more protective coats based on petroleum bitumen or coal tar derived materials, with or without filler, of the types defined in 5.2.2 and tables 2 and 3 built up to form the thickness required for the class of protection (I to IV) defined in 7.1 and table 6;
- c) one or more reinforcements of glass fibre or woven glass cloth, embedded in each protective layer.

The various components of any coating shall be compatible, particularly chemically. The class of thickness of the coating shall be taken from table 6, and shall satisfy the requirements of 7.1.2.

External coatings usually also have solar protection, commonly lime, to prevent excessive heating of the coating by solar radiation.

In certain special cases (for example, nature of backfill, environmental temperature or working temperature between + 60 and 80 °C), additional mechanical protection can be applied by agreement between the parties concerned.

When the pipeline is to be cathodically protected, mechanical protection of any sort shall not form an insulating barrier to the protective current.

4.2 Internal coating

The internal coating consists of one or more layers of petroleum bitumen or coal tar derived materials, filled or unfilled, with or without a primer coat, as defined in 5.2.1, 5.2.2 and 5.2.3 and specified in tables 1, 2 and 3; it is applied in accordance with the required classes of protection (A to D), defined in 7.2 and table 7.

5 Raw materials

5.1 Definitions

5.1.1 bitumen and coal tar derived materials : (The use of the terms bitumen and coal tar derived material is limited to materials defined in 5.1.2 and 5.1.3 and which may be reinforced with a filler defined in 5.1.5.)

5.1.2 petroleum bitumen : A mixture of high molecular mass hydrocarbons derived from petroleum by oxidation of suitable selected bases to a varying extent, possibly by adding fillers, in order to produce a material conforming to one of the grades Pa, Pb, Pc or Pd of table 3.

5.1.3 coal tar derived material : A mixture of high molecular mass hydrocarbons, obtained by distillation of high

temperature coal tar, processed and containing fillers so that one of the grades Ha or Hb is obtained (see table 3).

5.1.4 primer : A material applied as an undercoat directly to the metal, either molten or in solution, in order to assist the bonding of a subsequent coating of bitumen or coal tar derived material. There are two types of primers : bitumen or coal tar primers and synthetic primers.

5.1.4.1 bitumen or coal tar primer : A primer whose base is a bitumen or coal tar derived material (see 5.1.1).

5.1.4.2 synthetic primer : A primer containing solvent and whose base consists of resins and synthetic plasticizers.

5.1.5 filler : An inert powder which can be incorporated in a bitumen or coal tar derived material in order to improve one or more of its useful properties without changing its quality.

5.1.6 reinforcement : An inert material in the form of a narrow strip of open structure, intended to be saturated within the coating of bitumen or coal tar derived material in order to improve its mechanical performance.

5.1.6.1 non-woven glass fibre fabric : A reinforcement (see 5.1.6) consisting of a continuous sheet of randomly arranged glass fibres held together by a suitable binder and of open structure, and which may be reinforced longitudinally with glass fibre threads. This reinforcement may be impregnated additionally with a suitable bitumen or coal tar derived material.

5.1.6.2 woven glass fibre fabric : A reinforcement (see 5.1.6) consisting of a regular woven pattern of glass threads. This reinforcement may be impregnated uniformly with a suitable bitumen or coal tar derived material.

5.1.6.3 composite glass fibre fabric : A reinforcement (see 5.1.6) consisting of one layer of glass fibre matting and one layer of woven glass, combined with the aid of a suitable bitumen or coal tar derived material binder.

5.1.7 mechanical protection : An inert material which may be in the form of a strip, intended to guard the coating against external mechanical forces.

NOTE — There are materials for mechanical protection other than the asbestos felt defined in 5.1.7.1. When they are used, their description and properties shall be the subject of individual specifications and their use shall be the subject of an agreement between the parties.

5.1.7.1 asbestos felt : A mechanical protection (see 5.1.7) in the form of a narrow strip, consisting mainly of asbestos fibres and impregnated by a suitable bitumen or coal tar derived material; this protection is reinforced longitudinally by glass threads.

5.2 Specifications

5.2.1 Primers

The primer shall be selected in conjunction with the bitumen or coal tar derived coating material with which it shall be compatible.

The primer selected shall meet the requirements of table 1 where the following abbreviations are used :

- p^{Pa} is the bitumen based primer for coating material Pa¹⁾;
- p^{Pbc} is the bitumen based primer for coating materials Pb and Pc¹⁾;
- p^{Hab} is the coal tar based primer for coating materials Ha and Hb¹⁾;
- ps is the synthetic primer.

5.2.2 Bitumen or coal tar derived material

The bitumen or coal tar derived material is selected according to the maximum service temperature as set out in table 2 and/or local conditions (particularly of climate, storage, handling and exposure).

Whichever grade of bitumen or coal tar coating is selected it shall meet the corresponding requirements in table 3.

Table 1

Characteristic	Unit	Specification				Methods of tests ¹⁾
		p ^{Pa}	p ^{Pbc}	p ^{Hab}	ps	
Ash content (mass) on dry extract	%	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5	A → M B
Softening point Ring and ball on bitumen base	°C	≥ 80	≥ 105			A → D
Cold bend	mm	See table 3 ²⁾				C → F
Adhesion and compatibility	—					C → J
Flow	mm					C → H

- 1) See description in the annex.
- 2) Tests carried out on primer-coating material system.

Table 2

Type of coating material		Pa or Ha	Pb or Pc or Hb	Pd
Maximum service temperature	External coating	35 °C	60 °C	—
	Internal coating	25 °C	40 °C	25 °C

Table 3

Characteristic	Unit	Grade of coating material						Methods of tests ¹⁾
		Pa	Pb	Pc	Pd	Ha	Hb	
Softening point (ring and ball)	°C	≥ 95	≥ 110	≥ 120	≥ 80	≥ 100	≥ 105	C → D
Penetration (25 °C; 100 g; 5 s)	10 ⁻¹ mm	< 25	< 20	< 20	< 30	10 to 20	5 to 12	C → E
Cold bending	mm	20	15	10	20	15	10	C → F
Indentation	mm	≤ 17	≤ 10	≤ 8		≤ 10	≤ 5	C → G
Flow (70 °C; 45°; 20 h)	mm	≤ 6	≤ 2	≤ 2	≤ 6 ²⁾	≤ 5	≤ 3	C → H
Adhesion and compatibility (40 °C; 5 d)	—	shall pass test						C → J
Change on heating								K → C → D → C → E
— difference in softening point	°C	< 10	< 10	< 10	< 10	< 10	< 10	
— difference in penetration	%	< 40	< 40	< 40	< 40	< 40	< 40	
Water absorption	g/m ²	≤ 1,5	≤ 1,5	≤ 1,5	≤ 1,5	≤ 1,5	≤ 1,5	C → L
Ash content (mass)	%	≤ 40	≤ 40	≤ 55	≤ 2	25 to 35	25 to 35	C → M

- 1) See description in the annex.
- 2) Thickness 2 mm.

1) See table 3.

5.2.3 Filler

The filler shall be non-hygroscopic and unreactive with the other constituents of the protection, and resistant to attack by the medium to which it will normally be exposed. It shall be stable at the maximum application temperature of the coating material. Powdered slate and talc are typical examples of suitable fillers.

5.2.3.1 Particle size

The particle size of the filler shall meet the following requirements (see method N described in the annex).

- a) > 500 µm : 0 %
- b) > 90 µm : < 10 %

5.2.4 Reinforcements

The type and number of reinforcements to be used are fixed by the class of coating chosen in relation to the performance required of the protection (see 7.1.2).

5.2.4.1 Appearance

The reinforcements shall have, on visual examination, the following appearance.

- a) Glass fibre matting

Uniform appearance, optional reinforcement threads evenly spaced over the width of the matting, freedom from visible

faults such as holes, slits, folds, thin areas, spots where the binder has not hardened, leafing, frayed or uneven edges, presence of foreign bodies (oily matter, mud, etc.).

- b) Woven glass

Regular woven pattern of glass threads, freedom from visible faults such as holes, slits, frayed edges, presence of foreign bodies (oily matter, mud, etc.).

- c) Composite

Uniform appearance, freedom from visible faults such as holes, slits, frayed edges, badly impregnated areas, presence of foreign bodies (oily matter, mud, etc.).

5.2.4.2 Characteristics

The glass used shall be of hydrolytic class III as a maximum, as tested to ISO 719.

At the time of unrolling at ambient temperature, the successive layers of the reinforcement shall not stick to one another.

All reinforcements, whether or not impregnated, shall be compatible with the bitumen or coal tar derived material with which they are used and shall have a texture and binder content such that complete impregnation with the coating material is obtained during normal application. In addition the binder for the fibres shall be such as to resist the action of micro-organisms. The compatibility and ability to impregnate will be confirmed at the point of reinforcement application at the same time as the preliminary test on applying the coating.

The reinforcements shall also satisfy the requirements of table 4.

Table 4

Characteristic	Unit	Specifications			Methods of tests ¹⁾	
		Non-woven glass fibre fabric	Woven glass fibre fabric	Woven glass for composite	Reinforcement not impregnated	Reinforcement impregnated
Number of threads per 100 mm in each direction	—		> 30	> 65	R	Pa ²⁾ → Q
Mass per unit area after calcination	g/m ²	> 40	> 110	> 40	S	Pa → R
Loss of mass on ignition on mass of glass	%	< 20	< 3	< 3		
Tensile strength — longitudinally (R ₁₀) — transverse (R ₁₀)	N/50 mm N/50 mm	≥ 100 ≥ 25	≥ 300 ≥ 300	≥ 250 ≥ 250	T	Pb → S
Resistance to water	—	R ₁₁ ≥ 2/3 R ₁₀			U	Pb → T
Stability at application temperature	—	R ₁₂ ≥ 2/3 R ₁₀			U	

1) See description in the annex.

2) If necessary.

5.2.5 Mechanical protection

5.2.5.1 Asbestos felt

5.2.5.1.1 Appearance

The asbestos felt shall have, on visual examination, a uniform appearance, possibly with reinforcement threads evenly spaced over the width, small perforations as usually found evenly distributed over the whole surface, freedom from visible faults such as holes, slits, breaks, badly impregnated areas, delamination, uneven or frayed edges.

5.2.5.1.2 Characteristics

At the time of unrolling at ambient temperature, the successive layers of asbestos felt shall not stick to each other.

The bitumen or coal tar derived material used for impregnation shall be compatible with the bitumen or coal tar coating material.

The asbestos felt shall meet the requirements of table 5.

5.2.5.2 Other protection

There are other materials for mechanical protection. When they are used, their description and properties shall be the subject of individual specifications and their use shall be the subject of an agreement between the parties.

6 Application of coating

6.1 General

The external coating is applied :

- a) either in the factory or other workplace on each pipe or fitting in accordance with the conditions laid down, in particular, in 10.5;
- b) on the site after assembly of the pipeline,
 - on the whole line if the pipes have been delivered uncoated,

- on the joints if the pipes and fittings have been factory-coated.

To avoid condensation on the metal surface on the tube, the coating shall never be applied in the open air in rainy or foggy weather or when the temperature is less than the minimum specified by the product manufacturer.

The internal coating is applied in the factory or other workplace on each pipe or fitting in accordance with the conditions laid down in particular in 10.4 and 10.6. The ends of the pipes may be left uncoated for a fixed length, by agreement between the parties.

6.2 Preparation of the metal surface

For both external and internal protective coatings, the surface to be coated shall, at the time of application of the coating, be dry and free from all contaminants (such as previous coatings, paint, loose dirt, grease, oil, salt, etc.) which could be harmful to the surface preparation or to the adhesion of the coating to the steel.

6.2.1 General case

Complete removal of millscale, heat treatment scale and other adherent oxides shall be effected by blast-cleaning or by chemical cleaning; however, if the parties so agree, they may define the quality to be achieved and surface preparation may be by wire brushing.

6.2.1.1 Blast-cleaning

Before blast-cleaning, the surface to be treated shall be dry.

Blast-cleaning shall be carried out to achieve a quality of surface preparation of at least Sa 2, as in ISO 8501/1.

Immediately before the application of the coating, the surface shall be free from all trace of abrasive and dust.

6.2.1.2 Chemical cleaning

Chemical etching shall effect complete removal of oxides from the metal surface without damaging it. It shall be followed by thorough rinsing and optionally by passivating the metal. After

Table 5

Characteristic	Unit	Requirements	Methods of tests ¹⁾
Mass per unit area	g/m ²	≥ 586 ²⁾	V
Content of impregnation material calculated on unimpregnated felt (<i>m/m</i>)	%	22 to 40	V → W
Ash content on de-impregnated felt	%	≥ 73	W → Y
Asbestos content on de-impregnated felt	%	≥ 85	W → Z
Tensile strength, longitudinal	N/25 mm	≥ 110	AA
Pliability	—	shall pass test	AB

1) See description in the annex.

2) By agreement between the parties concerned a lower mass per unit area can be accepted subject always to the other requirements specified above being satisfied.

treatment the surface shall be free from acid residues. The treatment baths shall be maintained at effective concentrations.

6.2.2 Particular case — Surface covered by thin millscale

When the metal surface has a thin, continuous and adherent millscale of type A, according to ISO 8501/1, the coating may, by agreement between the parties, be applied over this scale with no other treatment than is required to remove contaminants.

6.3 Application of external coating — Joint protection and repairs

When hot-applied materials are being used, the recommendations of the manufacturer of the product with respect to maximum and minimum temperatures of application and times permitted for melting at elevated temperatures shall always be observed. The melting equipment shall be periodically cleaned to remove any deposits which may have formed. Necessary steps to ensure the homogeneity of the molten material shall be taken.

6.3.1 Application of the primer

The primer shall be applied to a clean dry surface prepared as described in 6.2. At the time of application, particularly when the weather is damp and cold, it may be necessary to pre-heat the metal; this operation shall not be prejudicial to the cleanliness of the surface or to the conditions required for the application of the primer.

The primer film, applied at the thickness specified by the primer manufacturer, shall be uniform and continuous.

6.3.1.1 Hot application

The primer shall be applied to a dry surface by dipping in a bath containing hot bitumen.

The duration of immersion in the bath shall be sufficient for the metal to reach the temperature of the bath.

This procedure is applicable only to petroleum bitumen, used in the factory or workplace.

6.3.1.2 Cold application of materials in solution

The application shall be made, for example, by brushing, mopping or spraying. The temperature of the metal at the time of application shall conform to the recommendations of the manufacturer of the primer.

6.3.1.3 Requirements for the primer coating before application of the protective coating

6.3.1.3.1 Dryness

The protective coating shall be applied over a cold-applied primer which has dried sufficiently or to an adequately cooled hot-applied primer, such that the protection will meet, in particular, the adhesion requirement in 7.1.3.

If the maximum time between priming and coating permitted by the manufacturer is exceeded, the primer coating shall be either removed and replaced or over-coated according to the manufacturer's instructions.

6.3.1.3.2 State of the primer coat

The protective layer shall be applied over a primer coat which is continuous, clean and free from moisture.

6.3.2 Application of the reinforced protective layer

Any method of application is permitted. The final coating shall meet the requirements of 7.1.

In the case of a factory-applied multi-layer coating, measures shall be taken to ensure adhesion between the layers. In the case of over-coating, the old coat shall be treated as necessary to ensure adhesion and the homogeneity of the entire coating.

6.3.3 Application of solar protection

Solar protection, if required, shall be applied to the final protective coating by a method appropriate to the product used. It shall be continuous and provide sufficient cover to form an effective barrier to solar radiation.

6.3.4 Application of mechanical protection

The application of mechanical protection by felt, woven or other suitable reinforcement wrap, if required, shall be made while the coating is still hot, before it sets (and before application of the optional solar protection). This mechanical protection shall be applied in such a manner as to minimise folds and voids, and so as to adhere to the underlying coating.

Asbestos felt, if used, is not part of the coating system and shall be regarded as mechanical protection.

Other forms of mechanical protection may be applied at the time of laying the pipe in the trench, following a procedure appropriate to each case.

6.3.5 Application conditions for protection at joints by means of bitumen and coal tar derived materials

The coating materials used shall be compatible with any coating previously applied.

Application conditions for primer and reinforced protective layer shall be those specified in 6.3.1 and 6.3.2.

The protection applied on the site to the joint shall overlap the protection applied in the factory for a sufficient length, so that in the vicinity of joints and fittings the requirements specified in 7.1.2, 7.1.3 and 7.1.4 are met. Solar protection and contaminants which could affect adhesion between coats shall be removed.

When the protective coating is applied by casting in a mould or a similar technique, the reinforcement(s) may be omitted or modified by agreement between the parties concerned.

6.3.6 Repair of coating by means of bitumen or coal tar derived materials

Repair coating materials used shall be compatible with the coatings to be repaired.

The technique to be used in each case shall be agreed between the parties.

The repaired coating shall meet the requirements specified in 6.3.2, 7.1.2, 7.1.3 and 7.1.4.

6.3.7 Assembly joint protection and repair using other products

The use of products other than the hot applied bitumen or coal tar derived materials, which are covered by this International Standard, is permitted for the protection of assembly joints and coating repairs. They shall however be agreed by the parties concerned. They shall be adherent to and compatible with the coating in contact.

6.4 Application of the internal coating – Joint protection and repairs

When hot-applied materials are being used, the recommendations of the manufacturer of the product with respect to maximum and minimum temperatures of application and times permitted for holding at elevated temperatures shall always be observed. The melting equipment shall be periodically cleaned to remove any deposits which may have formed. Necessary steps to ensure the homogeneity of the molten material shall be taken.

6.4.1 Application of primer or coating of classes A and B¹⁾

The application shall be made hot or cold as specified in 6.3.1.

6.4.2 Application of coating of classes C and D¹⁾

The application shall be made by rotating the pipe and introducing the coating material in the molten state. The coating shall always meet the requirements specified in 7.2.

6.4.3 Application conditions for protection of joints

The coating materials used shall be compatible with the coatings previously applied. Application conditions for the primer are as specified in 6.3.1.

Coating material in the molten state may be applied by any suitable means such as casting against a backing-up ring, trowelling, swabbing or by application using localized heating of pre-cast strips of lining material to the primed surface. In all cases it is essential to ensure non-porosity and good adhesion between the applied coating material, pipe surface and lining of pipes. When manual processes are used, the joint material shall be finished smooth and flush with the pipe linings, for example by smoothing with a heated flexible blade.

By agreement between the parties, protection at certain types of joint may be effected by the application of a thick, cold-applied bitumen or coal tar derived coating.

1) See requirements in table 7.

6.4.4 Repair of linings by means of bitumen or coal tar derived materials

Coating materials used shall be compatible with the previously applied coating.

Application conditions for the primer shall be as specified in 6.3.1. Damaged and non-adherent lining shall be removed before effecting a repair.

After cleaning and priming exposed surfaces, the lining shall be built up, pore-free, to the full thickness by trowelling or swabbing molten coating material, followed by smoothing to the original contour of the bore. Careful warming of the pipe metal and edges of the existing lining may be necessary to achieve satisfactory adhesion.

7 Characteristics of the applied coatings

7.1 External coating

7.1.1 Appearance and constitution (test according to 8.1.1)

On visual examination the coating shall be of uniform appearance and free from any faults detrimental to its performance. The length of uncoated parts shall satisfy the order requirements [see 10.4 d)].

The type and specified number of reinforcements, and the thickness of the coating shall conform to the class as defined in table 6 chosen by the purchaser.

The reinforcement(s) shall be completely impregnated with the protective material or a material compatible with it. The position of the reinforcement(s) shall be such that it (they) nowhere come(s) either less than 1 mm from the surface of the metal or outside the surface of the coating. Each reinforcement shall be free from folds and voids.

All reinforcements shall overlap. In the case of class I coatings, which contain only one reinforcement, the overlapping of successive turns shall be at least 15 mm. For the other classes the number of reinforcements in any cross-section of the protection shall be at least that specified (see 7.1.2).

7.1.2 Thickness (test according to 8.1.2)

The minimum thickness, excluding mechanical protection, as measured at any point on the coating, shall be that specified in table 6 for the class selected by the purchaser except along the

Table 6

Class ¹⁾	Number of reinforcements	Minimum thickness of coating mm
I	1	3
II	2	3
III	≥ 2	4
IV	≥ 2	6

1) It should be noted that the numbering of the classes in the table is not to be interpreted as an order of quality.

line of the weld which shall be the subject of previous agreement between the parties concerned.

For all classes the first reinforcement shall be glass fibre matting.

For classes II and III subsequent reinforcement(s) may be glass fibre matting or woven glass. The final reinforcement may alternatively be a composite of glass fibre matting and woven glass.

For class IV any intermediate reinforcements may be glass fibre or woven glass. The final reinforcement shall be woven glass or a composite of glass fibre matting and woven glass.

7.1.3 Adhesion (test according to 8.1.3)

The coating shall adhere adequately to the pipe.

The adhesion is considered to be satisfactory when there is no clear separation between removable parts of the coating and primer, or primer and metal.

Any separation shall be less than 10 mm from the edge of the section.

7.1.4 Freedom from holidays (test according to 8.1.4)

The coating shall be free from defects which can be detected by applied electric potential using an electrode giving a spark length, set at the time of test to a minimum length of 10 mm, or twice the minimum specified thickness of the coating, whichever is the greater, corresponding approximately to 10 kV.

Testing shall preferably be carried out before application of any mechanical protection. However, if the coating carries a mechanical protection applied at the same time as the coating, the spark length shall be set to at least three times the minimum specified thickness of coating.

7.2 Internal coating

7.2.1 Appearance (test according to 8.2.1)

The coating shall be of uniform appearance and free from any faults detrimental to its performance.

7.2.2 Thickness (test according to 8.2.2)

The minimum thickness at any point of the coating shall be that specified in table 7 for the class selected by the purchaser.

Table 7

Class	Minimum thickness of coating µm
A ¹⁾	50
B	400
C	1 500
D	3 000

1) Class A, at a thickness of approximately 50 µm, shall not be considered a protective coating. It is a temporary coating only for transport, storage, and possibly for improved appearance.

7.2.3 Adhesion (test according to 8.2.3)

The coating shall adhere adequately to the pipe.

Internal coatings of class A shall not be submitted to this test. Pending agreement on a test method for adhesion for class B coatings, the adhesion test applicable to this class shall be agreed between the supplier and purchaser.

7.2.4 Continuity (test according to 8.2.4)

Internal coatings of classes C and D shall be free from defects which can be detected by applied electric potential using an electrode giving a spark length set at the time of test to at least twice the minimum specified thickness of the coating.

Coatings of classes A and B shall not be submitted to this test.

8 Test methods for inspection of coated pipes and fittings

8.1 External coating

8.1.1 Appearance and constitution

The general appearance of the coating is judged visually. In order to check its constitution and particularly the number of reinforcements and their position, examine a cross-section detached from the metal with a cutting tool.

8.1.2 Thickness

8.1.2.1 Principle

The thickness is determined

- either by direct measurement with a penetration gauge (destructive test);
- or by magnetic measurement (non-destructive test) to an accuracy of ± 10 %.

In case of dispute, only the results of a direct measurement shall be accepted.

8.1.2.2 Procedure

Carry out the non-destructive test with a magnetic or electromagnetic instrument, calibrate the instrument on the coated metal and in the range of thickness to be measured. Recalibrate the instrument frequently to ensure its satisfactory performance and precision.

When the measurement is made with a gauge, it shall be inserted perpendicular to the coating and its point brought into contact with the metal. Make good the coating damage immediately after each test. Submit all repairs to the electrical test for continuity.

Carry out the measurements at a minimum distance from the end of the coating to be agreed between the parties, but which shall in no case be less than 200 mm.

8.1.3 Adhesion

8.1.3.1 Principle

The test consists of peeling a strip cut from the coating.

8.1.3.2 Equipment

A cutting tool with a thin sharp blade.

8.1.3.3 Procedure

Carry out the test on a coating which has reached ambient temperature (35 °C max.) :

- a) either 48 hours after application and at not less than 10 °C; the coating shall adhere to the metal,
- b) or outside the conditions set in a).

See 9.3.1 e) for interpretation of the results of tests in a) and b).

Cut out, with the aid of a tool, a 50 mm square taking care to cut the coating down to the metal, avoiding in so doing any shock which may give rise to premature detachment of the remaining coating.

Insert the blade into the coating to a distance of about 10 mm (in the case of internal coatings) or into the middle of the layer between the first reinforcement and the metal (in the case of external coatings) and lift smoothly.

8.1.4 Electrical test for freedom from holidays

8.1.4.1 Principle

The test consists of looking for possible faults in the coating by means of a high tension scanning electrode.

8.1.4.2 Equipment

8.1.4.2.1 A variable voltage fault detector (holiday detector).

8.1.4.2.2 A scanning electrode in the form of a metallic brush or a joined spiral spring or conductive rubber.

8.1.4.3 Procedure

Carry out this test only on reinforcements free from surface moisture. Connect the apparatus to the selected electrode and also to the metal wall of the pipe fitting, or pipeline, or to a low resistance earth. Check at the time of test on a pipe of the type to be tested that the spark length is not less than the minimum value specified; see 7.1.4 and 7.2.4.

This calibration is to be verified at intervals not exceeding 4 h.

Place the electrode in contact with the surface to be tested. Operate the electrode with a continuous movement at the rate recommended by the maker of the equipment. In the absence of such recommendation the rate shall be approximately 0,2 m/s.

The coating is considered defective at all points which give rise to a spark and an acoustic or optical signal.

Mark all such defective areas and repair.

8.2 Internal coating

8.2.1 General appearance

The general appearance of the coating is judged visually.

8.2.2 Thickness

The thickness shall be determined by the non-destructive test, which is carried out with a magnetic or electromagnetic instrument. The instruments shall be calibrated on the coated metal and in the range of thickness to be measured; they shall be recalibrated frequently to ensure satisfactory performance and precision.

8.2.3 Adhesion

The method described in 8.1.3 shall also be used for internal coatings of classes C and D.

For classes A and B, see 7.2.3.

8.2.4 Electrical test for continuity

The method described in 8.1.4 shall be applied to classes C and D coatings.

For classes A and B, see 7.2.4.

9 Inspection of coated pipes and fittings

9.1 General

Coating inspection shall be carried out by the responsible department at the factory. It shall include three types of operations :

- a) continuous inspection of the various parameters applicable, i.e. :
 - surface preparation (see 6.2),
 - conditions of application (see 6.3 and 6.4),
 - overlapping of reinforcement (see 7.1.1);
- b) systematic inspection of each pipe or fitting, i.e. :
 - visual inspection of appearance (see 7.1.1 and 7.2.1),
 - inspection of continuity by electrical testing (see 7.1.4 and 7.2.4),
 - inspection of the uncoated lengths at the ends (see 7.1.1);

c) non-systematic inspection of samples taken according to the rules of 9.2, i.e. :

- visual inspection of constitution (see 7.1.1),
- adhesion inspection (see 7.1.3 and 7.2.3),
- thickness inspection (see 7.1.2 and 7.2.2).

The sequence of operations is arranged so as to avoid later tests being carried out unnecessarily.

The tests shall be performed by the manufacturer. A representative of the purchaser may attend the various operations.

9.2 Definition of batches and rules for sampling

9.2.1 Definition of a production unit

By a production unit is meant a whole set of coated pipes or fittings with the following characteristics :

- same type of product (bitumen or coal-tar derived coating and reinforcement);
- same outside diameter of pipe or type of fitting;
- same class of coating;
- same thickness of coating;
- same production series;
- same coating unit;
- a fixed number of pipes or fittings.

9.2.2 Rules for sampling for non-systematic inspection

The pipes or fittings on which the planned non-systematic tests [see 9.1 c)] are performed shall be selected at the beginning, middle and end of the production unit. The test pieces shall be taken from the middle or ends (at a minimum 1000 mm distance from ends) of pipes, or from the middle or ends (at a minimum 200 mm distance) of fittings, and marked. Similarly the pipes or fittings from which test pieces are taken shall be marked for ready identification.

The frequency of testing and number of pipes or fittings in the production unit are as specified in the product standard or by agreement between the parties concerned.

9.3 Results of inspection tests and interpretation

9.3.1 Requirements

The inspection shall be considered satisfactory if the results conform to the corresponding test requirements :

- a) appearance and constitution (see 7.1.1 or 7.2.1);
- b) lengths of uncoated ends (see 7.1.1);
- c) continuity (see 7.1.4 or 7.2.4);

d) thickness (see 7.1.2 or 7.2.2);

e) adhesion (see 7.1.3 or 7.2.3).

1) If the adhesion test has been carried out under the conditions of 8.1.3.3 a), any poor result leads to rejection subject to the provisions of 9.3.2.

2) If the adhesion has been carried out under the conditions of 8.1.3.3 b), and the results are satisfactory, the coating is accepted as having satisfactory adhesion. Poor results obtained in such conditions are not taken as final but necessitate repetition of the test after a longer time and/or at a higher temperature, until the results are either satisfactory or are obtained under the conditions of 8.1.3.3 a).

In case of dispute the decision shall be taken on the basis of the test carried out under the conditions defined in 8.1.3.3 a).

Account is not taken of tests where unsatisfactory results are due not to the quality of the protection but to :

- faulty taking of the test piece, or
- defective setting up or abnormal operation of the test equipment.

In such cases the test shall be repeated.

9.3.2 Interpretation of results

9.3.2.1 If all tests or inspections are considered satisfactory, the production unit is considered to conform to the requirements. The coating of those pipes or fittings used for destructive testing is then repaired (see 6.3.6 and 6.3.7). These are then inspected again using non-destructive tests for continuity and, if required, for appearance and thickness, the specifications adopted being those of the corresponding repair materials.

9.3.2.2 If, however, the results of one or more tests are found poor or insufficient, two cases arise :

a) tests or inspections according to 9.1 a) or 9.1 b) : in such cases the pipes or fittings of which the coating acknowledged defective shall be taken back by the manufacturer for appropriate improvement;

b) tests or inspections according to 9.1 c) : unless otherwise agreed, additional tests shall be carried out at the rate of two tests for each unsatisfactory test of the same type — on, if possible, the pipe or fitting the coating of which was applied immediately before or after that of the defective pipe or fitting.

If all these additional tests are satisfactory, the production unit shall be considered to conform to the prescribed conditions (refer to 9.3.2.1 for the reconditioning of the pipes or fittings after inspection).

If at least one additional test still does not give satisfactory results, the manufacturer may carry out the corresponding test on each pipe or fitting of a given type in the production unit concerned. He shall then present the results obtained indicating all pipes or fittings giving satisfactory results. Pipes and fittings with a defective coating shall be rejected.

10 Ordering or laying contract wording

In the order for pipes or fittings to be coated in the factory or workplace, in the pipeline laying contract or in the specifications provided by the purchaser, all or part of the following information shall be given as appropriate.

10.1 Characteristics of the pipes (welded or seamless, end shape) or fittings to be coated, that is :

- a) steel quality and diameter;
- b) wall thickness and total length of pipes;
- c) quality and diameter of fittings;
- d) nature of fluid to be transported.

10.2 For surfaces

- a) preparation of metal surfaces (shot or grit blasting, mechanical or manual wire brushing, etching, etc.), and the minimum care required;
- b) methods of preparation of already coated surfaces, to be protected by manually-applied or mechanical coating on the laying site.

10.3 Required coating type (external, internal or both); i.e.

- a) place;
- b) method of coating application (in works, at workplaces, on the site; for each pipe or fitting, for the whole pipeline; using a machine or manually);
- c) temperatures of operation of the pipeline (normal and maximum).

10.4 For all coatings :

- a) nature and system of application of the primer or bonding coat (cold or hot application);
- b) nature of bonding agent (petroleum bitumen, coal tar, mineral filler, main characteristics of the product);
- c) temperatures of melting and application of the binder (maximum and minimum);
- d) lengths of uncoated areas at pipe or fitting ends; surface condition (bare, with a temporary primer or protection layer, nature and type of the latter);
- e) the types of storage and transport of coated pipes or fittings.

10.5 For external coatings :

- a) grade;
- b) minimum permitted thickness over seams or other prominent parts;
- c) number and type of reinforcements as in 5.1.6.1, 5.1.6.2 and 5.1.6.3 and minimum overlapping;
- d) nature and types of mechanical and solar protective coatings, if any;
- e) mean thickness and for bent or ballasted pipes maximum thickness;
- f) additional instructions for coating and repairs.

10.6 For internal coatings :

- a) grade;
- b) for potable water pipelines, hygiene criteria of the country of use.

10.7 Marking of coating (indication of class and type, colours, positioning, dimensions, etc.).

10.8 Coating inspection :

- a) composition of production units (number of pipes or fittings or total length of joints or pipeline);
- b) required inspection types and frequency per unit of production;
- c) number of samples and measurements (non-systematic testing) per unit;
- d) number of pipes, fittings, joints or length of pipeline represented by each sample and measurement;
- e) details of test result interpretation, of additional tests and of repaired coating inspection;
- f) type of acceptance certificate required.

11 Documents to be provided

The conformity of the delivery shall be guaranteed by the following documents.

11.1 Acceptance by the purchaser

The acceptance certificate signed by the purchaser's appointed inspecting authority contains the results of all tests and inspections specified carried out in his presence, and certifies that these were carried out on the manufacturer's premises on the samples taken from the products in the delivery.

11.2 Delivery by the works

The delivery certificate signed by the quality control officer's "Inspection by manufacturer" contains the results of all tests and inspections specified, and certifies that these were carried out by the manufacturer on the samples taken from the products in the delivery.

12 Marking

The marking on the coated pipe or fitting shall be applied by a suitable method such as painting, stencilling or printing which provides clearly legible, indelible identification using durable materials compatible with the protective coating.

The markings required shall be agreed between the parties concerned.

13 Handling, transport and storage

13.1 Handling

The coated pipes shall be handled so as to cause damage neither to the bevels nor the coatings. The direct use of steel or

hemp slings or other material the shape or nature of which could damage the pipe coating is prohibited.

13.2 Transport to coating works storage site

During transport of the pipes to the coating works storage site all appropriate precautions shall be taken to avoid damage to the pipes and coating.

13.3 Coating works storage site

Storage shall be controlled so that the coating suffers no damage. In particular, stacks of coated pipes shall be protected by an opaque covering if storage for over one year is intended.

13.4 Loading of pipes prior to delivery

All appropriate precautions to avoid damage to the pipes and coating during loading and transport shall be taken at the coating works.

In general this works is wholly responsible for the delivery of sound coated pipes while they are in his hands. In case of accidental damage to the coating, the making good and inspection are at coating works expense.

Bibliography

- [1] ASTM E 1, *Standard specification for ASTM thermometers.*

Annex

Test methods for raw materials

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Method A: Primer — Petroleum bitumen base — Separation of solvent from solution

A.1 Principle

The test consists of distilling the solvent under vacuum to avoid thermal degradation of the bitumen.

A.2 Apparatus

A.2.1 Sieve, 125 μm , in accordance with ISO 565-1972.

A.2.2 Distillation unit, composed of

A.2.2.1 500 ml round bottomed 3 necked flask.

A.2.2.2 Distillation column.

A.2.2.3 Refrigerant and vacuum receiver.

A.2.3 Heating apparatus (hot oil bath).

A.2.4 Thermometer, as specified in ASTM E 1/80, type 2 C^[1].

A.3 Procedure

A.3.1 If necessary, pass approximately 300 g of sample solution through the sieve, to remove solid impurities.

A.3.2 Stir the sample carefully and transfer to the distillation flask (A.2.2.1).

A.3.3 Heat the flask and distil the contents at a rate of 50 to 70 drops per minute, gradually increasing the temperature of the bath (A.2.3) to a maximum of 180 °C.

A.3.4 Subsequently reduce the pressure to 6 to 8 kPa (45 to 60 mmHg).

A.3.5 After completing the removal of solvents, heat the flask containing the residue to a temperature at which the bitumen can be poured.

A.3.6 Then fill the various pieces of apparatus to be used for further tests.

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Method B : Synthetic or coal tar based primer — Ash content

B.1 Principle

The test consists of determining the ash content of a sample of synthetic or coal tar based primer after calcination under specified heating conditions.

B.2 Apparatus

B.2.1 Flat bottomed dish, of porcelain or fused silica of approximately 100 mm diameter.

B.2.2 Drying oven, ventilated and controlled between 105 and 110 °C for synthetic primer or at 165 ± 1 °C for coal tar based primer.

B.2.3 Muffle furnace controlled at 700 ± 50 °C.

B.2.4 Desiccator (vacuum type for synthetic primer).

B.2.5 Balance, accurate to 0,001 g.

B.3 Method

B.3.1 Carry out the test in duplicate.

B.3.2 Weigh as quickly as possible into the previously weighed (m_1) dish (B.2.1), approximately 10 grams of synthetic or coal tar based primer.

B.3.3 Heat the dish in the oven (B.2.2) for 3 h at the temperature specified in B.2.2.

B.3.4 Allow the dish to cool in the desiccator (B.2.4) and weigh to the nearest 0,001 g (m_2).

B.3.5 Calcine the sample contained in the dish gently in the muffle furnace (B.2.3) to constant mass.

B.3.6 Then place the dish containing the ash carefully in the desiccator, allow it to cool and take all necessary precautions when opening the desiccator to avoid any loss of ash. Weigh (m_3).

B.4 Calculation of results

B.4.1 Calculate the ash, expressed as a percentage by mass, from the following formula :

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

where

m_1 is the mass of dish, in grams;

m_2 is the mass of the dish and dried primer solids, in grams;

m_3 is the mass of dish and ash, in grams.

B.4.2 Take the arithmetic mean of the two results.

Method C: Bitumen or coal tar derived materials — Preparation of sample

C.1 Principle

The test consists of heating a sample of bitumen or coal tar derived material to mix it and to remove impurities.

C.2 Apparatus

C.2.1 Heating apparatus : air or sand bath or oven.

C.2.2 Melting pot with the following approximate dimensions :

- diameter of base : 65 mm
- diameter of rim : 95 mm
- height : 65 mm

C.2.3 Thermometer, as specified in ASTM E 1, type 2C⁽¹⁾.

C.3 Procedure

C.3.1 Put 200 ± 25 g of sample in the melting pot (C.2.2) and heat it in the air or sand bath or oven (C.2.1) to a temperature at which the viscosity allows normal application and foaming ceases. The total heating time shall not exceed 30 min.

C.3.2 During the heating, stir the contents with the thermometer, in order to obtain a well-mixed sample. To remove persistent air bubbles, the sample may, if necessary, be placed in an oven held at the temperature indicated above. However, heat for as short a time as possible and for not longer than 1 h.

C.3.3 Remove any skin that has formed from the surface.

C.3.4 Fill the apparatus to be used for the tests immediately.

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Method D: Bitumen or coal tar derived material — Softening point — Ring and ball

D.1 Definition

The **ring and ball softening point** is the temperature at which a disc of the material, contained in a ring, undergoes a standard deformation caused by the weight of a ball under standardized test conditions.

D.2 Apparatus

D.2.1 Two brass rings with shoulders with the dimensions shown in figure 1.

D.2.2 Steel plate.

D.2.3 Two steel balls, of diameter 9,53 mm and mass $3,5 \pm 0,05$ g.

D.2.4 Ball centring guides such as those shown in figure 2.

D.2.5 Knife.

D.2.6 Glass beaker, having the dimensions shown in figure 3.

D.2.7 Ring support in metal having the dimensions shown in figure 4.

D.2.8 Thermometer, as specified in ASTM E 1, type 16C^[1].

D.2.9 Release agent, consisting of equal parts by mass of glycerol and mineral talc.

D.2.10 Glycerol of density 1260 kg/m³.

D.2.11 Pair of tongs.

D.3 Procedure

D.3.1 Prepare the sample according to method C.

D.3.2 Heat the two brass rings (D.2.1) to a temperature approximately equal to that of the sample, and place them on the steel plate (D.2.2), previously treated with the release agent (D.2.9).

D.3.3 Fill the rings with the hot bitumen or coal tar derived material sample.

D.3.4 Cool for at least 30 min to room temperature, but the overall test time after filling the rings shall not exceed 240 min.

D.3.5 Then level the bitumen or coal tar derived material in the rings by removing the excess with a warmed knife (D.2.5).

D.3.6 Place the rings in the frame (D.2.7) : put the thermometer (D.2.8) and ball centring guide (D.2.4) in position.

D.3.7 Fill the glass beaker (D.2.6) with glycerol at a temperature of 32 ± 1 °C to a height of minimum 102 mm and maximum 108 mm.

D.3.8 Hold the glycerol temperature at 32 ± 1 °C for 15 min.

D.3.9 Place the balls (D.2.3), previously heated to a temperature of 32 ± 1 °C, in the ball centring guides (D.2.4), using a pair of tongs (D.2.11).

D.3.10 Raise the temperature of the glycerol at a uniform rate of 5 °C/min for the first 3 min, and subsequently at a rate of $5 \pm 0,5$ °C per min. Avoid any draught, using screens if necessary.

D.3.11 Note the temperature at which the bitumen or coal tar derived material surrounding each steel ball touches the bottom plate (no correction is made for the emergent thermometer stem).

D.3.12 Carry out a repeat test if the temperatures obtained for the duplicated samples differ by more than 1 °C.

D.4 Reporting of results

Report the softening point as the arithmetic mean of the temperatures noted for the duplicate samples, rounded off to the nearest 0,5 °C.

D.4.1 Repeatability

The results of a duplicate test obtained by one person on the same apparatus are suspect if they differ by more than 1 °C.

D.4.2 Reproducibility

The result of an analysis, being the mean value of the duplicate temperatures obtained by one laboratory; the result shall not differ by more than 2 °C from that of another laboratory.

Dimensions in millimetres

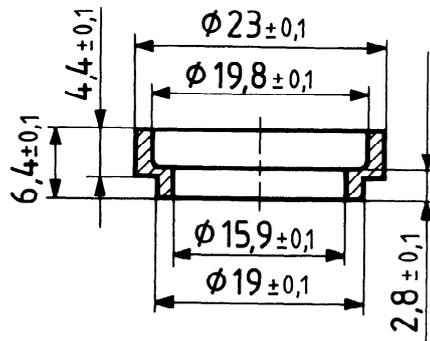


Figure 1 – Ring (see D.2.1)

Dimensions in millimetres

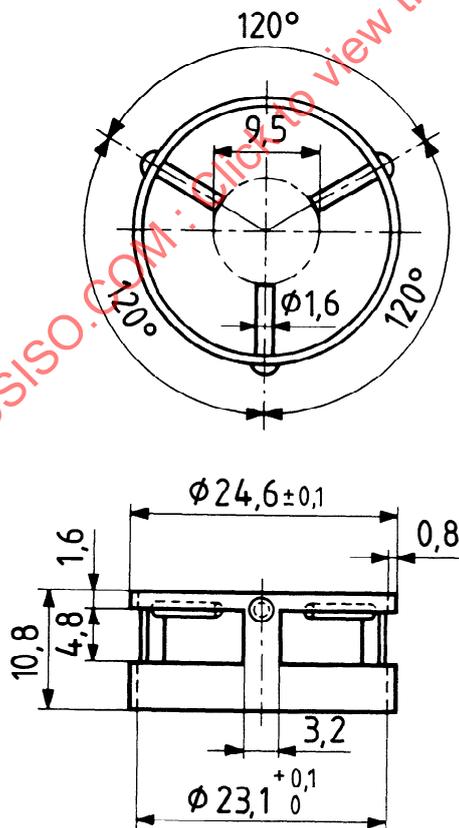
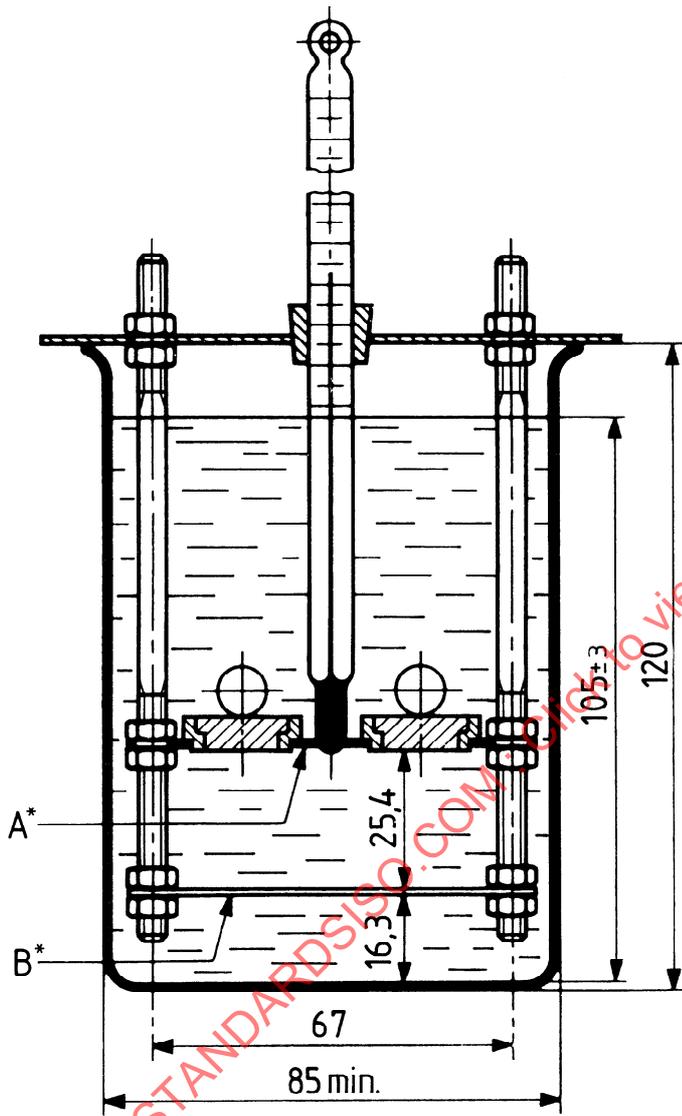


Figure 2 – Ball centring guide (see D.2.1)

Dimensions in millimetres



* Metal support, see figure 4.

Figure 3 — Ring and ball apparatus (see D.2.6)

Dimensions in millimetres

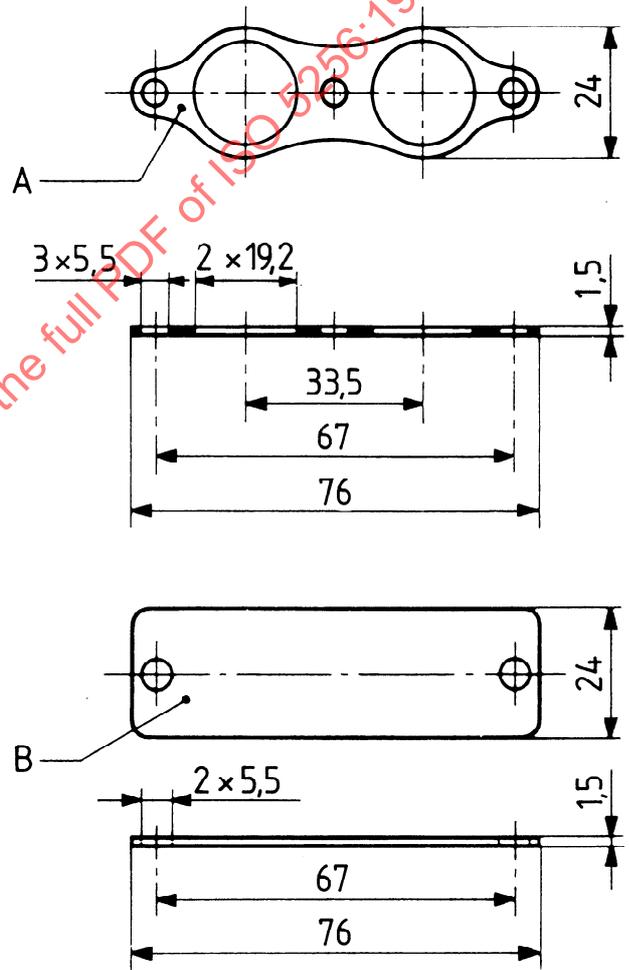


Figure 4 — Metal supports A and B (see D.2.7)

Method E: Bitumen or coal tar derived material — Penetration

E.1 Definition

penetration value : The depth, expressed in units of 0,1 mm, to which a standard needle placed vertically on the surface of the sample of bitumen or coal tar derived material, and loaded with a 100 g weight under the specified conditions of temperature (25 °C) and time (5 s) will enter.

E.2 Apparatus

E.2.1 Penetrometer : an instrument as shown in figure 5 which permits a needle holder to move vertically with negligible friction, and registers the displacement (penetration of the needle) in units of 1/10 mm.

The mass of the needle holder shall be $47,5 \pm 0,05$ g to which shall be added the mass of the needle $2,5 \pm 0,05$ g and a detachable weight of mass $50 \pm 0,05$ g to give the load required.

The plate supporting the metal dish shall be flat and horizontal.

E.2.2 Penetration needles : At least three highly polished and hardened steel needles are needed, with the dimensions and tolerances as indicated in figure 6. Each needle of mass $2,5 \pm 0,05$ g consists of a perfectly cylindrical rod with a point in the shape of a straight truncated cone, mounted co-axially in a cylindrical brass shank.

The edges of the flattened needle points shall be sharp and free from burrs. Each shank shall bear an identification number.

After each test the needles shall be cleaned with cotton wool and toluene, and stored in vaseline. Needles with traces of rust or damage shall not be used; regular inspection under a microscope is therefore required.

E.2.3 Mould of non-oxidizing metal, of cylindrical shape and having a flat bottom, of the following dimensions :

- internal diameter : 55 mm
- depth : 35 mm

When a coal tar derived material is being tested, the mould has no bottom; it is placed on a polished brass plate, preferably treated with a release agent.

E.2.4 Thermostat bath containing a minimum of 10 l of water and controlled at a temperature of $25 \pm 0,1$ °C and fitted with a perforated metal plate not less than 50 mm from the bottom.

The height of the bath shall be such that the mould (E.2.3) is immersed to a depth of at least 100 mm.

E.2.5 Dish with a flat bottom (inside and outside) having a minimum capacity of 350 ml. The height shall be such that the mould can be completely immersed in it. Provision shall be made to prevent any movement of the mould during a test.

E.2.6 Timing device with a minimum accuracy of 0,1 s.

E.2.7 Thermometer as specified in ASTM E 1, type 90C⁽¹⁾.

E.3 Procedure

E.3.1 Prepare the sample according to method C.

E.3.2 Fill the mould (E.2.3) with molten material carefully, so that the sample contains no air bubbles, to a depth at least 10 mm greater than the expected penetration depth.

E.3.3 Then cool at room temperature for 60 to 90 min, taking care that no dust or other impurities fall into the sample.

E.3.4 Place the mould and the flat-bottomed dish (E.2.5) on the perforated plate (at least 100 mm under the surface) in the thermostat bath (E.2.4) and allow 60 min for the sample to reach the test temperature of 25 °C. In the case of a coal tar derived material the mould is inverted.

E.3.5 Then transfer the mould under water to the flat-bottomed dish and place this on the base of the penetrometer (E.2.1).

E.3.6 Clean the needle (E.2.2) with toluene and cotton wool. Make sure that the needle and the needle holder are clean and dry, and fix the needle into the holder. Place the detachable weight of mass 50 g on the needle holder.

E.3.7 Then adjust the position of the needle so that the tip just touches the sample, i.e. so that the tip just makes contact with its image on the surface of the sample.

E.3.8 Bring the bar of the indicator mechanism into contact with the needle holder and note the position of the pointer. Then allow the needle to penetrate the bitumen for 5 s.

E.3.9 Again bring the bar of the indicator mechanism into contact with the needle holder and note the new position of the pointer.

E.3.10 Calculate the depth of penetration as the difference between the two readings in 1/10 mm.

E.3.11 Carry out at least three determinations in the same mould at points at least 10 mm from the wall of the mould and from each other.

E.3.12 Use at least three different needles, and replace the flat-bottomed dish with the mould in the thermostat bath while fitting a new needle. If any movement of the mould occurs during the test, ignore the determination.

E.4 Reporting of results

Calculate the penetration as the arithmetic mean of at least three determinations; the highest and lowest of these results shall not differ by more than two units.

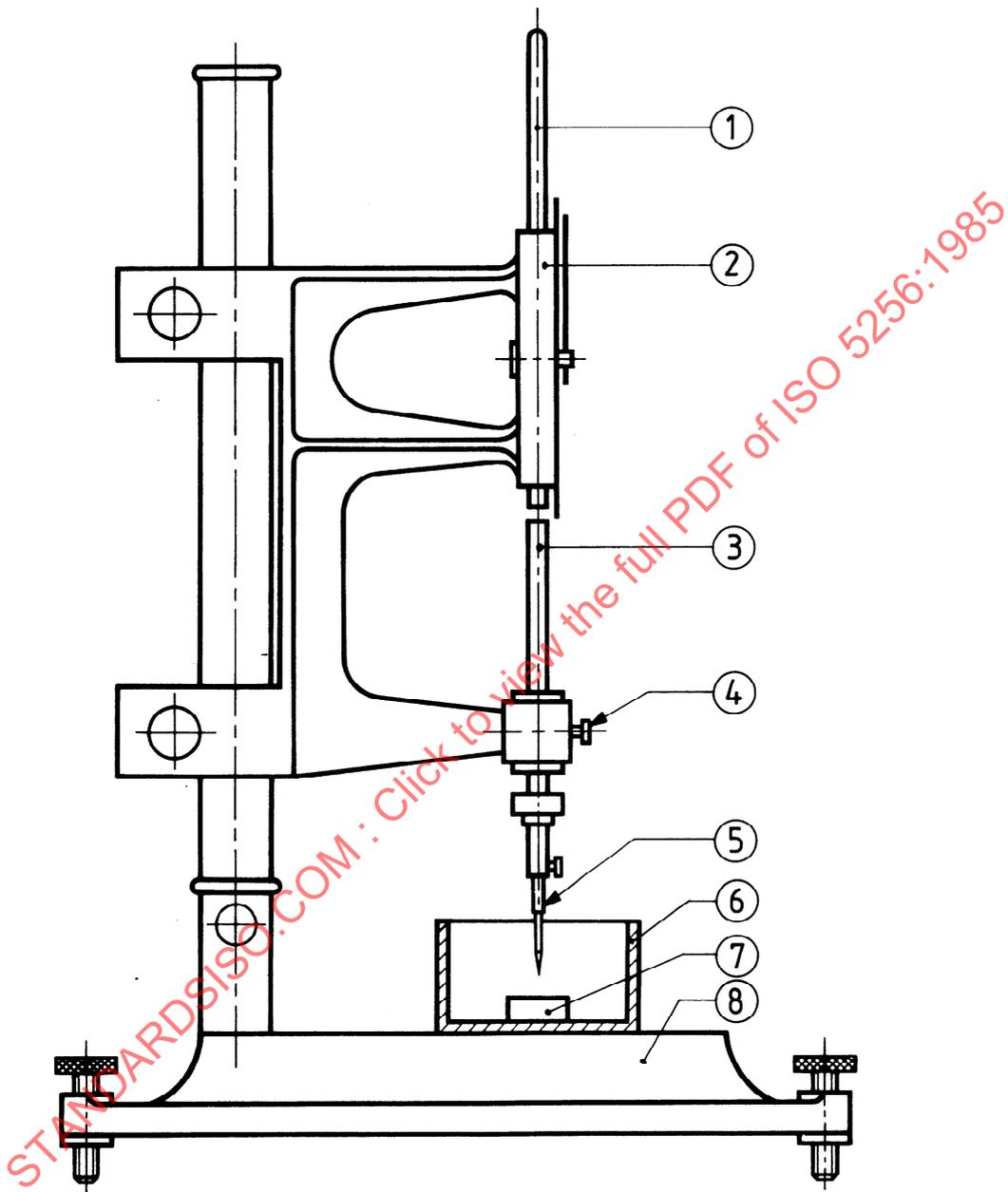
E.4.1 Repeatability

The results of a duplicate determination obtained by one person with the same apparatus are suspect if they differ by more than one unit.

E.4.2 Reproducibility

The results reported by two laboratories, each being the calculated mean of the results of duplicate determinations, are suspect if they differ by more than four units.

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- | | |
|------------------------------|---------------------------|
| ① Bar of indicator mechanism | ⑤ Penetration needle |
| ② Indicator mechanism | ⑥ Flat bottom dish |
| ③ Needle holder | ⑦ Mould containing sample |
| ④ Clamp | ⑧ Base plate |

Figure 5 — Penetrometer (see E.2.1)

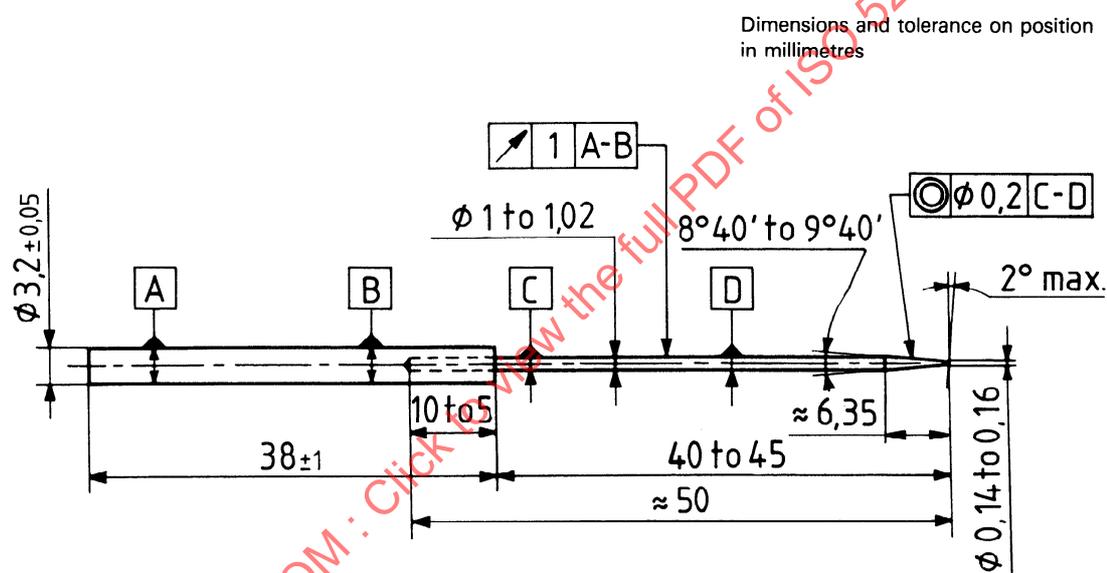


Figure 6 — Penetration needle (see E.2.2)

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Method F: Bitumen or coal tar derived material — Cold bending

F.1 Principle

The test consists of verifying the flexibility at low temperature of bitumen or coal tar derived material used as a coating on steel pipes and under conditions simulating the bending of coated pipes.

F.2 Apparatus

F.2.1 Flat test plates, of steel Fe 360 (to ISO 630) of dimensions 300 mm × 100 mm × 1,6 mm, blast-cleaned to SA 2,5 quality as defined in 6.2.1.1.

F.2.2 Mould, according to figures 7 a) and 7 b) consisting of a flat steel base plate having dimensions 300 mm × 100 mm × 10 mm, and a frame with coverplate. The dimensions of the frame and coverplate shall be such that a layer of the sample of $2 \pm 0,2$ mm can be applied to the test plate.

F.2.3 Bending apparatus, according to figure 8, capable of bending a test plate over a central mandrel of 25 mm diameter at a constant rate of 1 mm/s, with provision for measuring the deformation depth.

F.2.4 Holiday detector (see 8.1.4.2.1) supplying a potential such that the adjusted spark length amounts to three times the measured coating thickness.

F.2.5 Thickness gauge, magnetic or electromagnetic.

F.2.6 Water bath at 4,5 °C, filled with a mixture of water and melting ice.

F.2.7 Doctor blade [see figure 7 a)].

F.2.8 Primer, as used in the coating of the pipe.

F.2.9 Release agent, consisting of equal parts by mass of glycerol and mineral talc.

F.3 Procedure

F.3.1 Apply a thin film of primer (F.2.8) to the test plates (F.2.1) which have been suitably prepared to the thickness recommended by the supplier. Allow to dry for a maximum of 48 h at room temperature.

F.3.2 Treat the vertical edges of the coverplate with release agent (F.2.9). Assemble the mould (F.2.2) with the test plate inside.

F.3.3 Prepare the sample of bitumen or coal tar derived material according to method C.

F.3.4 Pour the molten test sample on to the plate coated with primer inside the mould so that the product rises to a level 1 to 2 mm above the top of the mould.

F.3.5 Remove the excess product whilst still hot, with the warm doctor blade (F.2.7).

F.3.6 Allow the mould to cool, and then remove the test plate from the mould.

F.3.7 Measure the thickness of the layer of coating to the nearest 0,1 mm and check for absence of holidays with the holiday detector (F.2.4).

Use for the bend test only test plates with a coating layer of $2,0 \pm 0,2$ mm thickness, free from holidays.

F.3.8 Immerse the test plates in the water bath (F.2.6) at 4,5 °C for 60 to 90 min.

F.3.9 Remove one of the test plates from the water bath, dry the surface, for example with filter paper, as thoroughly as possible, and immediately fit the plate into the bending apparatus (F.2.3), with the central mandrel against the uncoated surface.

F.3.10 Bend immediately at a constant speed of 1 mm/s until the deformation depth is that specified in table 3. (Less than 120 s shall elapse between the removal of the test plate from the water bath and the completion of bending.) Check immediately with the holiday detector whether cracking of the bitumen layer of coating has occurred.

F.3.11 Carry out a repeat test on a second test plate.

F.4 Reporting of results

Report the observations made on the two test plates :

— If no cracking has been detected, the coating material is considered satisfactory with respect to the cold bending test.

— If, however, cracks are produced on only one of the two plates the bending test shall be repeated on a third plate : according to the result obtained, absence or presence of cracks, the coating shall be considered satisfactory or otherwise.

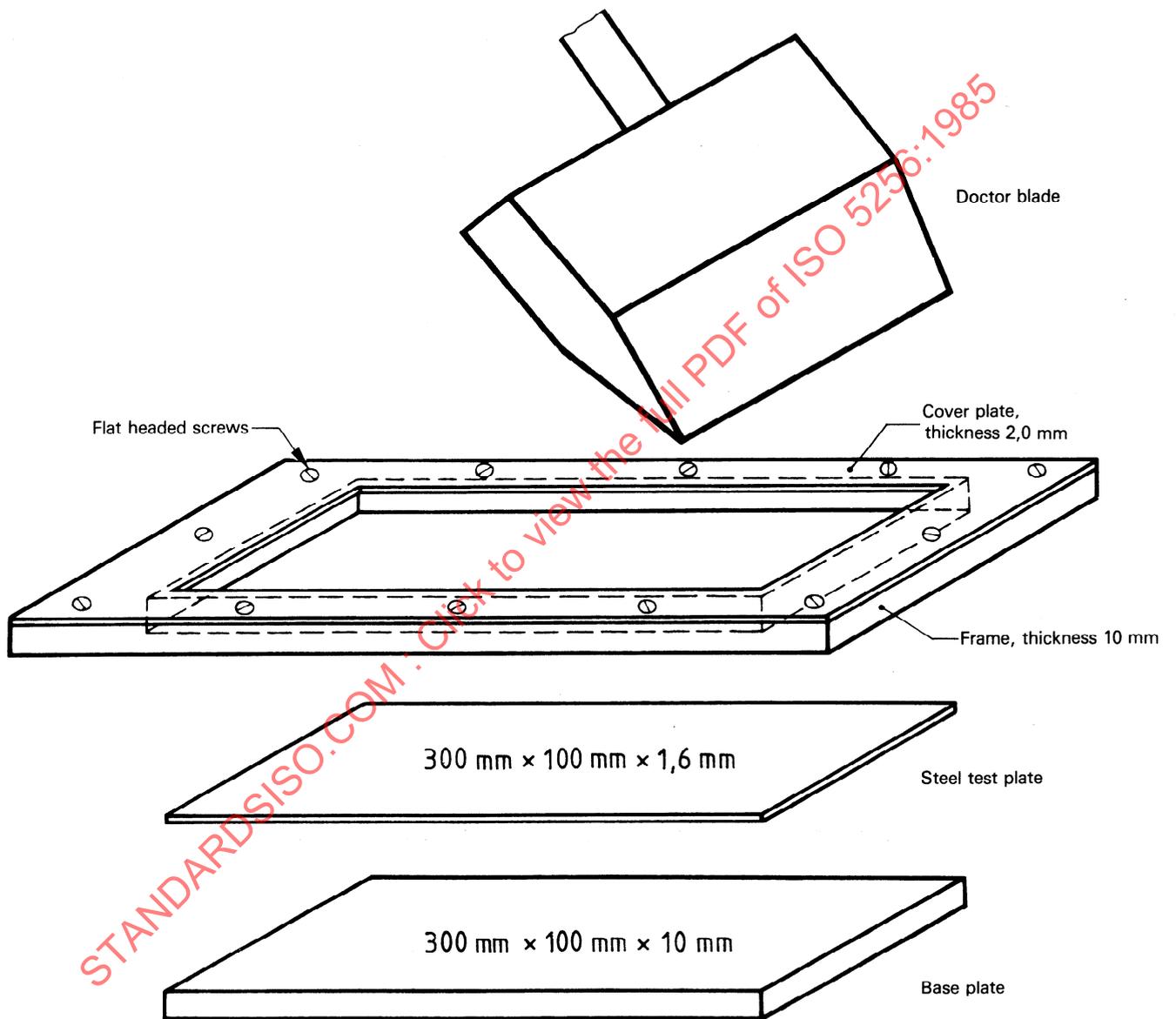


Figure 7 a) — Mould and doctor blade for bending test
[see also figure 7 b)]

Dimensions in millimetres

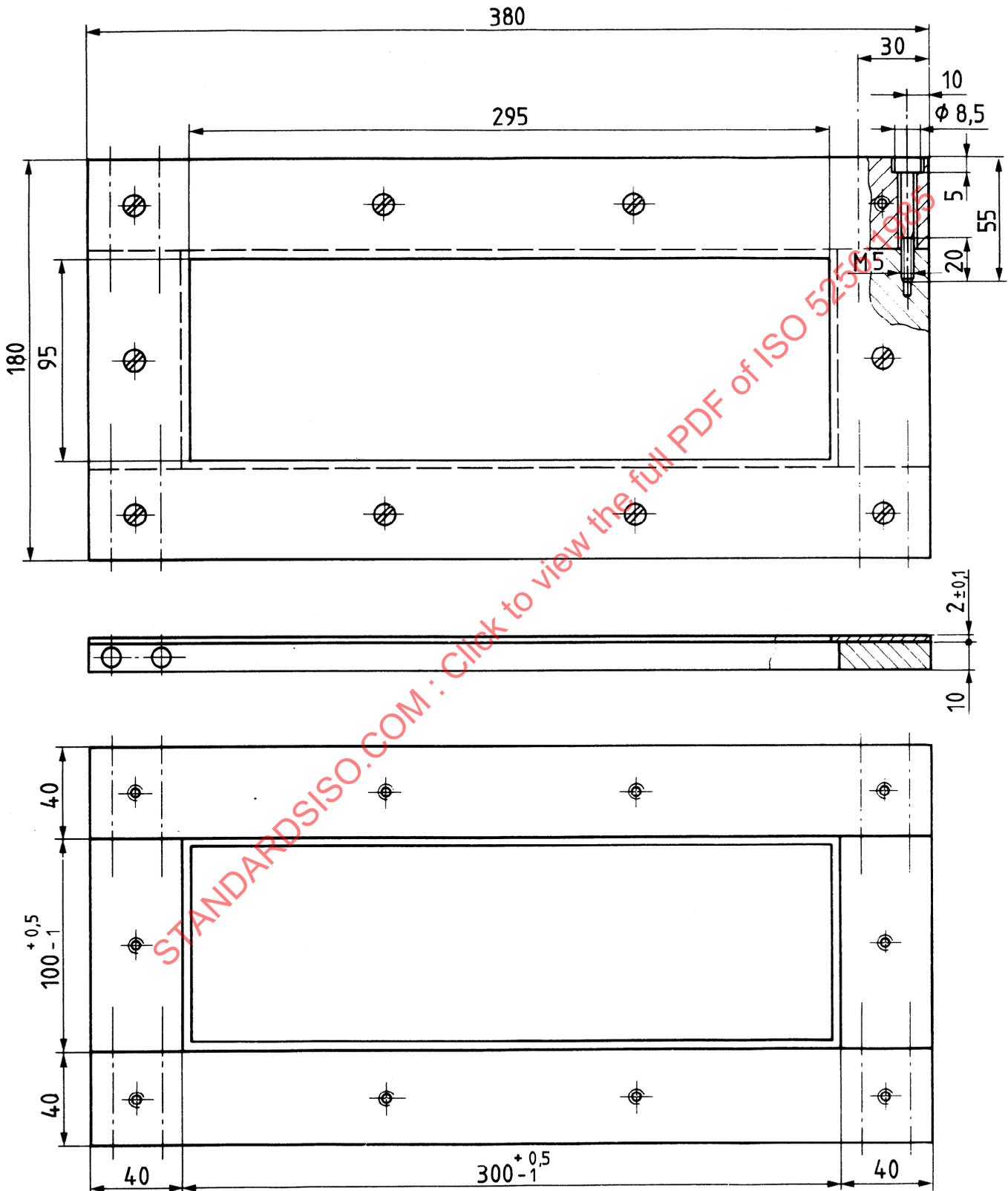


Figure 7 b) — Details of mould for bending test

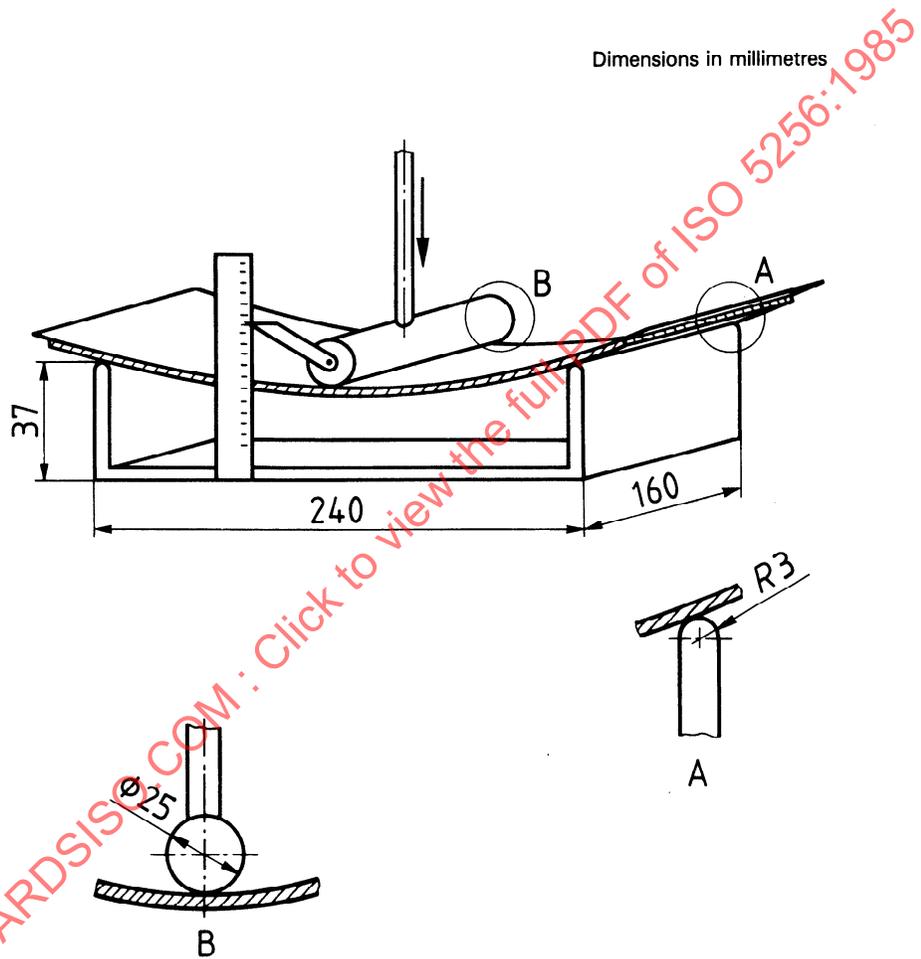


Figure 8 — Bending apparatus

Method G : Bitumen or coal tar derived material — Indentation

G.1 Definition

indentation : The penetration, expressed in millimetres, of a standard rod, placed vertically on the surface of a bitumen or coal derived material and loaded under specified conditions of temperature (25 °C), load (25 N) and time (24 h).

G.2 Apparatus

G.2.1 Indentor, a cylindrical metal bar, having a cross-section of 100 mm², loaded so that the total downward stress is 250 Pa (25 N/mm²).

G.2.2 Penetrometer (Vicat) with a scale calibrated in millimetres as shown in figure 9.

G.2.3 Non-oxidizing metal container for sample, of diameter 55 mm and depth 35 mm, as used in the determination of penetration (see method E).

G.2.4 Thermostat controlled water bath, at 25 ± 1 °C.

G.2.5 Ring and collar (see figure 9), to fit closely around the container (G.2.3).

G.2.6 Knife.

G.2.7 Release agent, consisting of equal parts by mass of glycerol and mineral talc.

G.3 Procedure

G.3.1 Apply release agent (G.2.7) to the ring (G.2.5) and fix it on the container (G.2.3).

G.3.2 Prepare the sample according to method C.

G.3.3 Fill the container with the material so that it rises above the rim.

G.3.4 Allow to cool and remove the ring. Trim off the excess material with a warm knife (G.2.6) so as to obtain a smooth surface.

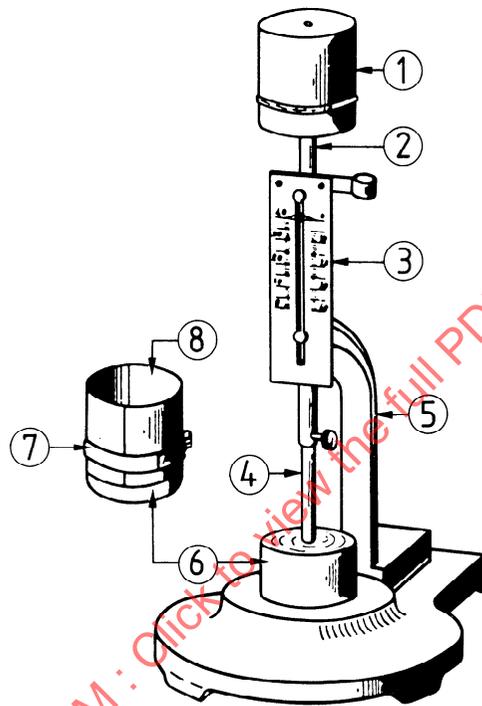
G.3.5 Put the container and the penetrometer (G.2.2) with the indentor (G.2.1) in the water bath (G.2.4) for one hour.

G.3.6 Then position the indentor on the surface of the bitumen or coal tar derived material, take the penetrometer reading (P_1) and apply the total mass of 25 N.

G.3.7 After 24 hours take the penetrometer reading again (P_2).

G.4 Reporting of results

Record the indentation test values as the difference between the two readings to the nearest 0,5 mm ($P_2 - P_1$).



- | | |
|---|-----------------------------|
| ① Weight | ⑤ Stand |
| ② and ④ Bar of cross-section 100 mm ²
(bar and weight have a combined mass of 2,5 kg) | ⑥ Sample container |
| ③ Graduated scale | ⑦ Metal collar for clamping |
| | ⑧ Ring for filling |

Figure 9 — Penetrometer

Method H: Bitumen or coal tar derived material — Flow

H.1 Principle

The test consists of measuring the displacement of the surface of a coating of a bitumen or coal tar derived material by its own weight under specified conditions of temperature (70 °C) and time (20 h).

H.2 Apparatus

H.2.1 Steel test plates, completely flat, of dimensions 200 mm × 100 mm × 3 mm, blast-cleaned to SA 2,5 quality as defined in 6.2.1.1.

H.2.2 Moulds, consisting of a flat steel base of dimensions 320 mm × 130 mm × 15 mm (or wider if several moulds are combined) and attached strips of suitable dimensions. These strips shall be fastened with screws to the sides of the test plate, except on one of the short sides where the strip shall be fastened so that a 30 mm length of the test plate is not covered with bitumen or coal tar derived material [see figures 10 a) and 10 b)]. By using strips of a suitable thickness a coating of the material 5 mm thick (2 mm only, in the case of coating material Pd) can be produced.

H.2.3 Doctor blade, 120 mm wide, for trimming the bitumen or coal tar derived material [see figure 7 a)].

H.2.4 Knife.

H.2.5 Flow measuring device [see figure 10 c)], consisting of a copper plate bent back twice at right angles in such a way that it can be pushed over the test plate including the 5 or 2 mm layer of bitumen or coal tar derived material.

This copper plate has 12 small holes positioned so that marks made on the bitumen or coal tar derived material layer through these holes are not closer than 10 mm to the edge of this layer.

H.2.6 Stand [in accordance with figure 10 d)] on which the plates can be inclined at an angle of 45°.

H.2.7 Oven, maintained at $70 \pm 0,5$ °C and fitted with a fan to ensure a uniform temperature.

H.2.8 Primer, as used for adhesion of the bitumen or coal tar derived material to the pipes.

H.2.9 Release agent, consisting of equal parts by mass of glycerol and mineral talc.

H.2.10 Marking ink, white.

H.2.11 Marking needle, for making the marks on the coating of bitumen or coal tar derived material.

H.3 Procedure

H.3.1 Apply a thin film of primer (H.2.8) to the test plates (H.2.1) at the thickness recommended by the supplier and allow to dry for 24 h at room temperature.

H.3.2 Assemble the mould (H.2.2) on the steel base plate and the strips pre-treated with release agent (H.2.9).

H.3.3 Pour prepared bitumen or coal tar derived material (according to method C) on to the plate in the mould so that it rises approximately 3 mm beyond the top edge of the mould.

H.3.4 Before cooling remove the excess material with the warm doctor blade (H.2.3).

H.3.5 Remove the plate from the mould, cut off the sides of the bitumen or coal tar derived material at an angle of 45° with the hot knife (H.2.4).

H.3.6 Slide the flow measuring device (H.2.5) over the plate and make 12 white marks (H.2.10) on the surface of the coating with the needle (H.2.11).

H.3.7 Remove the marked plate from the measuring device and store for 12 h in a horizontal position at room temperature.

H.3.8 Place the plate in the stand (H.2.6) inclined at an angle of 45° with the 30 mm edge not covered with coating material at the bottom and keep the stand in the oven (H.2.7) for 20 h at $70 \pm 0,5$ °C.

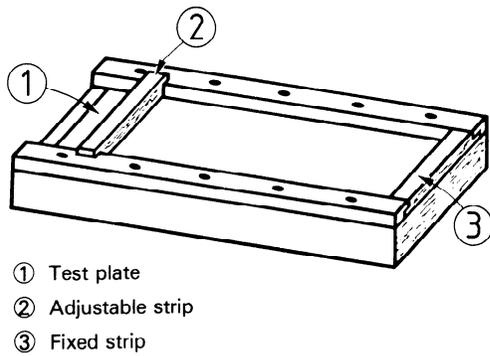
H.3.9 Remove the plate from the oven and allow to cool in a horizontal position.

H.3.10 Slide the flow measuring device over the plate and again make 12 white marks on the surface of the coating with the needle.

H.3.11 Measure the displacement of the marks.

H.4 Reporting of results

Report the flow as the mean value of displacement of the 12 white marks to the nearest 0,5 mm.



- ① Test plate
- ② Adjustable strip
- ③ Fixed strip

Figure 10 a) – Mould

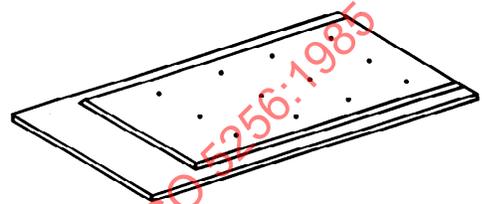
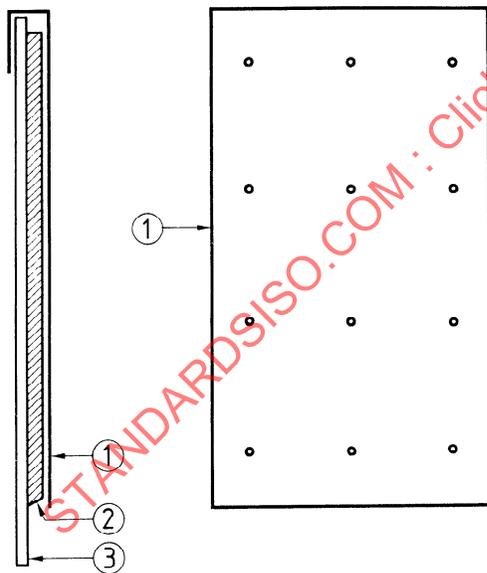
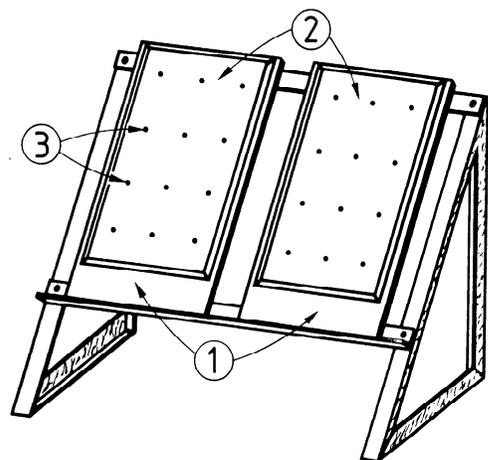


Figure 10 b) – Test plate covered with protective coating



- ① Copper plate (with 12 holes)
- ② Coating
- ③ Steel test plate

Figure 10 c) – Flow measuring apparatus



- ① Steel plate
- ② Coating
- ③ Reference points

Figure 10 d) – Stand

Method J : Bitumen or coal tar derived material — Adhesion and compatibility

J.1 Principle

The test consists of verifying on a laboratory scale the adhesion of a bitumen or coal tar derived material to the primer over which it is to be applied.

J.2 Apparatus

J.2.1 Steel test plates, of dimensions

200 mm × 100 mm × 3 mm, blast-cleaned to SA 2,5 quality according to the conditions specified in 6.2.1.1.

J.2.2 Thermostat water bath at $40 \pm 0,5$ °C.

J.2.3 Apparatus for clamping the test plates horizontally.

J.2.4 Jute strips of dimensions 300 mm × 100 mm having a mass of approximately 200 g/m².

J.2.5 Primer as used with the bitumen or coal tar derived material.

J.2.6 Bitumen or coal tar derived material as used for pipe protection.

J.3 Procedure

J.3.1 Apply the primer (J.2.5) to two steel test plates (J.2.1).

— If the primer is to be applied as a solution, coat the test plates once with a thin film to the thickness specified by the supplier.

— If the primer is to be applied by dipping, immerse the plates in the product at a temperature which will give a coating of maximum thickness 0,3 mm.

J.3.2 Do not apply the bitumen or coal tar derived material (J.2.6) while the primer film is still tacky.

J.3.3 Prepare the sample of bitumen or coal tar derived material according to method C.

J.3.4 Pour sufficient test material on to the primer film to form a coating 3 mm thick.

J.3.5 Lay on the still hot material a strip of jute (J.2.4), previously impregnated with this same material, so that it projects 50 mm beyond both short sides of the plate.

J.3.6 Then pour over the strip a further layer 3 mm thick of the bitumen or coal tar derived material.

J.3.7 Allow the plates to cool for 24 h and then hold them in the thermostat bath at 40 ± 5 °C for 5 d.

J.3.8 Immediately after cooling, clamp the plates horizontally with the apparatus (J.2.3), and pull the projecting strip of jute vertically upwards by hand, the plate remaining horizontal.

J.3.9 Observe whether with both plates the layer of bitumen or coal tar derived material under the jute adheres completely to the primer film.

J.4 Reporting of results

J.4.1 If the coating layer adheres on both test plates completely to the primer film, report that the adhesion of primer and bitumen passes the test.

J.4.2 If the coating layer does not adhere, report that the adhesion does not pass.

Method K: Bitumen or coal tar derived material — Change on heating

K.1 Principle

The test consists of heating the bitumen or coal tar derived material for 5 h to the maximum application temperature specified by the supplier of the product and then measuring the changes in certain characteristics.

K.2 Apparatus

K.2.1 Steel pot of diameter 200 mm and height 300 mm, fitted with a lid.

K.2.2 Mechanical stirrer.

K.2.3 Thermometer as specified in ASTM E 1, type 2C^[1].

K.2.4 Heating system adapted to the requirements of the test.

K.2.5 Apparatus described under method D, for ring and ball softening point.

K.2.6 Apparatus described under method E for penetration.

K.3 Procedure

K.3.1 Fill the pot (K.2.1) about three quarters full with lumps of the bitumen or coal tar derived material. Place the lid in position.

K.3.2 Raise the contents of the pot to the maximum application temperature specified by the supplier by means of a flame in contact with the bottom of the pot, but controlled so as to avoid localized overheating.

K.3.3 Keep this temperature constant for 5 h.

K.3.4 During the whole operation stir the molten mass with the stirrer rotating at 1 to 2 r/s.

K.3.5 On completing the heating, use the "aged" material to determine

- the ring and ball softening point by method D;
- the penetration by method E.

K.4 Reporting of results

Express in degrees Celsius and percentage respectively, the differences in the values of softening point and penetration thus determined and the initial values.

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Method L: Bitumen or coal tar derived material — Water absorption

L.1 Principle

The test consists of determining the increase in mass of bitumen or coal tar derived material by exposing to water at 40 °C for 5 h.

L.2 Apparatus

L.2.1 Round, stainless steel dishes, having an internal diameter of 100 mm (area 0,007 85 m²) and depth of 5 mm.

L.2.2 Thermostat bath, containing distilled water and controlled at 40 ± 1 °C.

L.2.3 Doctor blade, 120 mm wide, for trimming the bitumen or coal tar derived material [see figure 7 a)].

L.2.4 Balance, accurate to 0,001 g.

L.2.5 Pieces of soft filter paper and soft cloth.

L.3 Procedure

L.3.1 Pour the sample prepared by method C into three dishes (L.2.1) to above the rims. Allow to cool, but before the material has completely cooled remove the excess with the warmed doctor blade (L.2.3) so that a flat surface is left.

L.3.2 Hold the dishes at room temperature for at least 4 h and weigh each to 0,001 g (m_1).

L.3.3 Immerse the dishes for 5 h in the thermostat bath (L.2.2) at a temperature of 40 ± 1 °C.

L.3.4 Remove them from the bath.

L.3.5 Mop the surfaces with filter paper (L.2.5) and dry the dishes with a soft cloth (L.2.5).

L.3.6 Weigh as quickly as possible to an accuracy of 0,001 g (m_2).

L.3.7 Determine the increase in mass of each dish ($m_2 - m_1$); the increases for the three dishes shall not vary by more than 0,01 g.

L.4 Reporting of results

Calculate the arithmetic mean increase in mass of the three dishes, expressed in grams, and convert the result to g/m² taking the surface area of each dish as 0,007 85 m².

Method M: Bitumen or coal tar derived material — Ash content

M.1 Principle

The test consists of determining the filler content of a filled bitumen or coal tar derived material is measured by its ash content as determined by calcination of a sample under specified temperature conditions.

M.2 Apparatus

M.2.1 Balance, accurate to 0,001 g.

M.2.2 Crucibles, made of porcelain or fused silica, of diameter 65 mm.

M.2.3 Muffle furnace, which can be adjusted for temperatures of up to at least 800 °C.

M.2.4 Drying oven, controlled at a temperature of 150 ± 5 °C.

M.2.5 Desiccator.

M.2.6 Ammonium carbonate, saturated solution.

M.3 Procedure

M.3.1 Prepare the sample according to method C.

M.3.2 Carry out the test in duplicate.

M.3.3 Weigh into a previously weighed (m_1) crucible (M.2.2) approximately 3 g of the sample to the nearest 0,001 g (m_2).

M.3.4 Ignite the sample carefully and then heat at 700 ± 50 °C to constant mass.

M.3.5 Moisten the cooled ash with a few drops of ammonium carbonate solution (M.2.6) and dry in the oven (M.2.4) controlled at 150 ± 5 °C to constant mass.

M.3.6 Allow to cool in the desiccator (M.2.5) and weigh (m_3).

M.4 Calculation of results

M.4.1 Calculate the ash content, expressed as a percentage, from the following formula :

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

where

m_1 is the mass of the crucible, in grams;

m_2 is the mass of the crucible and sample, in grams;

m_3 is the mass of the crucible and ash, in grams.

M.4.2 Take the arithmetic mean of the two results obtained.

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Method N : Filler — Particle size

N.1 Principle

The test consists of determining by wet sieving the particle size of the filler to be incorporated in the bitumen or coal tar derived material.

N.2 Apparatus

N.2.1 Balance, accurate to 0,05 g.

N.2.2 Sieves with apertures 500 μm and 90 μm , as specified in ISO 565.

N.2.3 Glass beaker of capacity approximately 1 l.

N.2.4 Drying oven, maintained at 110 ± 5 °C.

N.2.5 Acetone, chemically pure.

N.3 Procedure

N.3.1 Dry at least 100 g of the sample of filler (free from lumps or foreign bodies) in the drying oven (N.2.4) to constant mass.

N.3.2 Weigh out 100 ± 5 g to the nearest 0,1 g (m_0).

N.3.3 Transfer the sample to the beaker (N.2.3) and moisten it with acetone (N.2.5).

N.3.4 Add sufficient water to cover the sample completely and stir vigorously to disperse the fine material.

N.3.5 Immediately pour the suspension of filler over the assembled sieves (N.2.2) the 500 μm sieve being placed on top.

N.3.6 Wash the filler residue out of the beaker into the sieves with water.

N.3.7 Wash the sieves with water until the water passing through contains no more filler.

N.3.8 Dry the residues left on the sieves in the drying oven to constant mass.

N.3.9 Weigh the residues to the nearest 0,1 g (m_1 and m_2).

N.4 Reporting of results

N.4.1 Calculate the quantities of material retained, as a percentage of the original sample, as follows :

$$M_{500} = \frac{m_1}{m_0} \times 100$$

$$M_{90} = \frac{m_2}{m_0} \times 100$$

where

m_0 is the mass of the original sample in grams;

m_1 is the mass retained on the 500 μm sieve in grams;

m_2 is the mass retained on the 90 μm sieve in grams.

N.4.2 Report the results to the nearest 0,5 %.

Method P: Impregnated reinforcement — Extraction of impregnant

P.1 Principle

The test consists of extracting the reinforcement impregnated with bitumen or coal tar derived material by a suitable solvent.

P.2 Procedure

According to the determinations to be carried out subsequently, one of the following two methods is used :

P.2.1 Method Pa

This method shall be used before carrying out the following determinations :

- number of threads per 100 mm in each direction for woven glass (method Q) ;
- mass per unit area after calcination and loss of mass on ignition (method R).

P.2.1.1 Apparatus

- P.2.1.1.1 **Extraction apparatus**, as shown in figure 11.
- P.2.1.1.2 **Pair of forceps**.
- P.2.1.1.3 **Drying oven with fan**, controlled at 105 ± 5 °C.
- P.2.1.1.4 **Desiccator**.
- P.2.1.1.5 **Solvent**, chemically pure (for example toluene).

P.2.1.2 Method of extraction

P.2.1.2.1 Place the reinforcement of suitable dimensions (consult the relevant test methods) in the extraction apparatus (P.2.1.1.1) and extract with the suitable solvent (P.2.1.1.5) until the liquid which runs down is colourless.

P.2.1.2.2 Remove the extracted glass material, with the forceps (P.2.1.1.2).

P.2.1.2.3 Dry the extracted material, first at room temperature, then in the oven (P.2.1.1.3) controlled at 105 ± 5 °C; then cool it in the desiccator (P.2.1.1.4).

P.2.2 Method Pb

This method shall be used before carrying out the following determinations :

- tensile strength (method S);
- resistance to water (method T).

P.2.2.1 Apparatus

P.2.2.1.1 **Bath** of dimensions 400 mm × 400 mm × 50 mm fitted with air-tight lid.

P.2.2.1.2 **Fume cupboard**.

P.2.2.1.3 **Wash bottle**.

P.2.2.1.4 **Drying oven with fan** controlled at 105 ± 5 °C.

P.2.2.1.5 **Desiccator**.

P.2.2.1.6 **Suitable solvent**, chemically pure.

P.2.2.2 Method of extraction

P.2.2.2.1 Put the test pieces of the correct dimensions (according to the relevant methods of test) in the bath (P.2.2.1.1) filled with a suitable chemical solvent (P.2.2.1.6).

P.2.2.2.2 Carry out the extraction in a fume cupboard (P.2.2.1.2).

P.2.2.2.3 Extract until, after removal of the material from the bath, the bare glass fibre matting or woven glass is completely visible.

P.2.2.2.4 Rinse the material thoroughly with fresh solvent using a wash bottle (P.2.2.1.3).

P.2.2.2.5 Subsequently dry the material, initially at room temperature and then for 30 min in the oven (P.2.2.1.4) at 105 ± 5 °C and allow to cool to room temperature in the desiccator (P.2.2.1.5).

Dimensions in millimetres

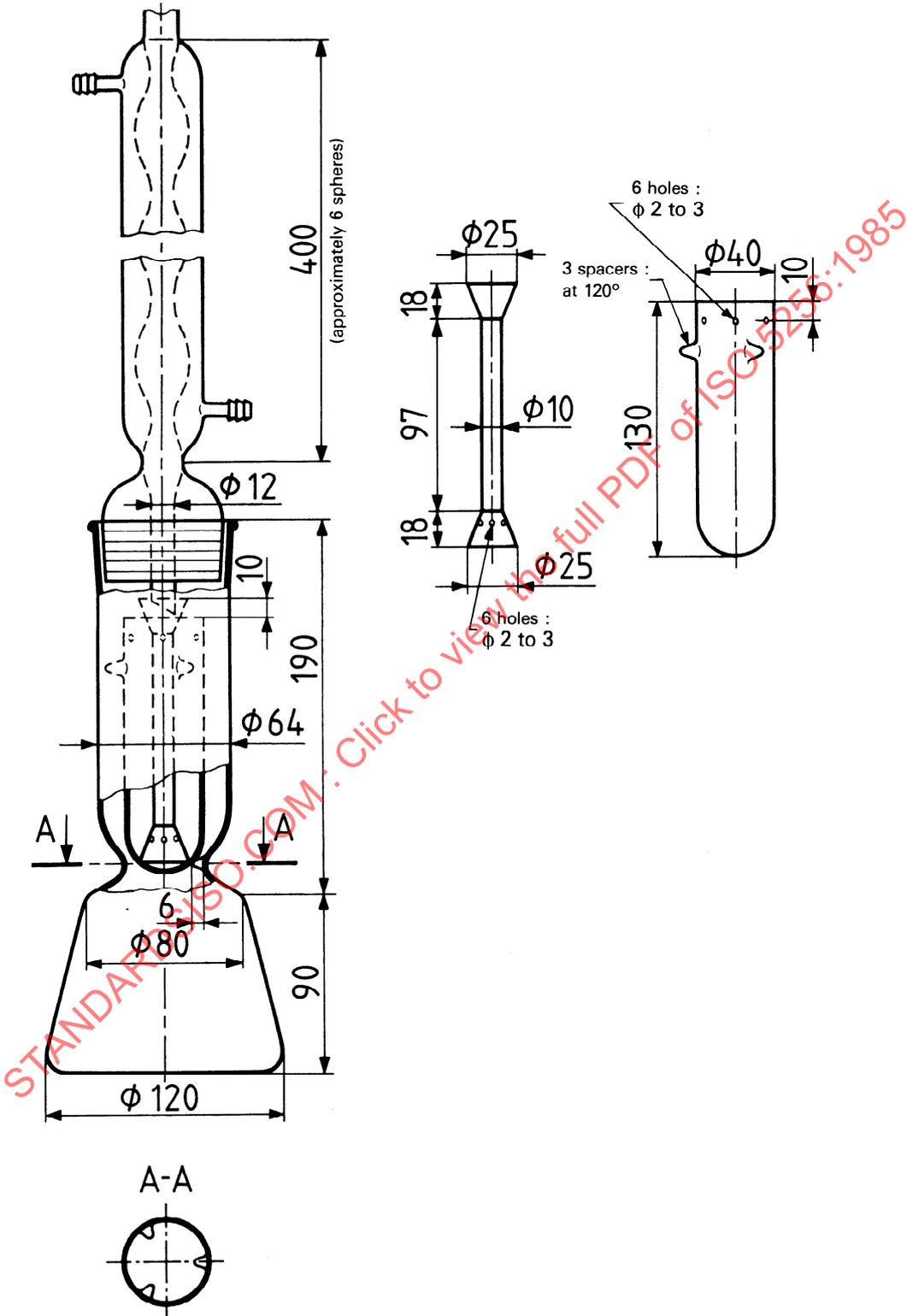


Figure 11 — Extraction apparatus

Method Q: Woven glass reinforcement — Number of threads per 100 mm in each direction (according to ISO 4602)

Q.1 Principle

The test consists of determining in a woven glass reinforcement the number of threads per 100 mm in each direction.

Q.2 Apparatus

Q.2.1 Strong cardboard sheet with a square aperture of 100 mm side cut out in the middle.

Q.3 Procedure

Q.3.1 In the case of impregnated woven glass, prepare the sample according to method Pa, if necessary.

Q.3.2 Carry out the test in triplicate on specimens slightly larger than 100 mm by 100 mm.

Q.3.3 Place the specimen on a perfectly plane surface, superimpose the cardboard sheet (Q.2.1), taking care that one side of the square cut out is exactly parallel to the edge of the woven glass.

Q.3.4 Count the number of threads both along a line parallel to the edge and along a line perpendicular to the edge of the woven glass. Note that in certain woven fabrics the transverse threads are grouped in pairs.

Q.3.5 The first count gives the number of threads per 100 mm in the longitudinal direction, and the second the number of threads per 100 mm in the transverse direction of the sample.

Q.4 Reporting of results

Calculate for the longitudinal and transverse directions the arithmetic mean of the three values and round off the results to the next higher whole number.

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Method R: Reinforcement — Mass per unit area after calcination and loss of mass on ignition

R.1 Principle

The test consists of determining the mass per unit area of the de-impregnated reinforcement before and after calcination at the specified temperature. The loss of mass in ignition is then calculated from the results.

R.2 Apparatus

R.2.1 Dishes of porcelain or fused silica of square shape of dimensions 110 mm × 110 mm.

R.2.2 Balance, accurate to 0,001 g.

R.2.3 Muffle furnace, which can reach a temperature of at least 800 °C.

R.2.4 Desiccator.

R.3 Procedure

R.3.1 Determination of initial mass per unit area

R.3.1.1 Cut eight specimens of reinforcement of dimensions 100 mm × 100 mm.

R.3.1.2 Proceed in the case of impregnated reinforcement, to extract the bitumen or coal tar derived material from these eight specimens according to method Pa.

R.3.1.3 Then determine the mass per unit area (ρ_{A0}) of the de-impregnated reinforcement, expressed in grams per square metre, from the total mass of the eight specimens.

R.3.2 Determination of mass per unit area after calcination

R.3.2.1 Take four of the specimens used for determining the initial mass ρ_{A0} .

R.3.2.2 Calcine them together in the muffle furnace (R.2.3) at a temperature of 625 ± 25 °C for 30 min, to destroy the binder and/or the protective coating on the glass fibres.

R.3.2.3 Allow the specimens to cool to room temperature in a desiccator (R.2.4).

R.3.2.4 Weigh the specimens to the nearest 0,001 g (m_0) and calculate the mass per unit area, expressed in grams per square metre, after calcination with the formula

$$\rho_{A1} = 25 m_0.$$

R.4 Reporting of results

R.4.1 Mass per unit area after calcination

Report the result ρ_{A1} expressed in grams per square metre, to the nearest whole number.

R.4.2 Loss of mass on ignition

Calculate the loss on ignition, expressed as a percentage, on the residual glass after calcination by the following formula :

$$\frac{\rho_{A0} - \rho_{A1}}{\rho_{A1}} \times 100$$

where

ρ_{A0} is the mass per unit area of de-impregnated reinforcement, expressed in grams per square metre;

ρ_{A1} is the mass per unit area of de-impregnated reinforcement after calcination expressed in grams per square metre.

Method S : Reinforcement — Tensile strength

S.1 Definition

tensile strength of a reinforcement : The force exerted on a rectangular strip, 50 mm wide, at the instant it begins to break when, under the conditions specified below, it is subjected to a gradually increasing tensile force at a constant rate of extension.

S.2 Apparatus

S.2.1 Air-conditioned room having the following characteristics :

- temperature : 23 ± 2 °C
- relative humidity : (65 ± 5) %

S.2.2 Tensile testing apparatus, mechanically driven. The speed of the moving cross-head shall be such that the specimen fractures within 10 ± 5 s for a specimen width of 50 mm and an initial distance between the clamps of 200 mm.

In the case of apparatus with a lever arm the deflection of the arm shall be between 9 and 45°; with apparatus of other types the first tenth and the last tenth of the measuring range shall not be used. It is recommended that those parts of the scale which are not permitted shall be clearly marked.

Provision shall be made for pre-stressing the sample; it shall be fastened in the upper clamp and shall hang freely through the open lower clamp.

The pre-stressing force shall be 5 N, evenly applied over the whole strip width.

S.2.3 Timing device (stop watch).

S.2.4 Spring clamp with jaws at least 50 mm wide.

S.2.5 Latex, natural or synthetic.

S.3 Sampling

S.3.1 Unimpregnated reinforcement

Two pieces of 300 mm × 280 mm, one in the longitudinal and one in the transverse direction.

S.3.2 Impregnated reinforcement

Two pieces of 350 mm × 350 mm. After extraction of the bitumen or coal tar derived material according to method Pb,

mark the test pieces so that the longitudinal and transverse directions can be identified; then cut them to the dimensions required above for unimpregnated reinforcements.

NOTE — If the width of the sample roll is too small to provide specimens of this size, smaller test specimens may be used, provided this is stated specifically in the test.

S.4 Procedure

S.4.1 Preparation of tensile specimens

S.4.1.1 Take the pieces sampled as in clause S.3 and mark on each short side a strip of 280 mm × 50 mm.

S.4.1.2 Treat these two strips with latex of a suitable viscosity so that the centre of each specimen of dimensions 280 mm × 200 mm is kept free from latex.

S.4.1.3 Allow the latex to dry.

S.4.1.4 Cut from each sample five specimens of 300 mm × 50 mm, each end of the test pieces to be clamped by latex on a length of 50 mm having now been suitably prepared to prevent fibres slipping out of the clamps.

S.4.1.5 Condition the cut specimens for at least 20 h in the air-conditioned room (S.2.1) at 23 ± 2 °C and (65 ± 5) % relative humidity.

S.4.2 Tensile test

S.4.2.1 Fix a specimen in the upper clamp. Before screwing the lower clamp on to the freely suspended bottom end of the specimen attach a separate spring clamp (S.2.4) with jaws and carrying a total mass of 500 g, so as to exert an initial force of 5 N over the full width of the specimen.

S.4.2.2 Now screw up the lower jaw.

S.4.2.3 Start the stop watch (S.2.3) and the loading mechanism (S.2.2) simultaneously.

S.4.2.4 Stop the watch at the instant the force reaches its maximum value as the specimen begins to break.

S.4.2.5 Record this maximum force.

S.4.2.6 If a specimen breaks in or near the clamps, ignore the value obtained and carry out an extra determination with a new specimen.

S.4.2.7 Make separate tests for the determination of tensile strength in the longitudinal and transverse directions.

S.5 Reporting of results

S.5.1 Calculate the tensile strength as the arithmetic mean of five determinations, performed under the conditions specified above.

S.5.2 Report the tensile strength in newtons per 50 mm to the nearest 5 N, as R_{10} in the longitudinal and R_{t0} in the transverse direction.

S.6 Reproducibility

The reproducibility of the method is $\pm 10\%$.

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Method T: Reinforcement — Resistance to water

T.1 Principle

The test consists of measuring the change in a characteristic of the reinforcement on immersion for a specified period in still water.

T.2 Apparatus

T.2.1 Glass dishes, approximately 400 mm × 200 mm × 50 mm.

T.2.2 Drying oven, controlled at $110\text{ °C} \pm 5\text{ °C}$.

T.2.3 Apparatus as for method S, tensile strength test.

T.2.4 Water, demineralized or distilled.

T.3 Procedure

T.3.1 In the case of an impregnated reinforcement an extraction of the bitumen or coal tar derived material according to method Pb shall first be carried out.

T.3.2 Make the test on a minimum of five specimens of dimensions 300 mm × 50 mm, identical to those specified for the tensile strength test (see method S).

T.3.3 Completely immerse the specimens in a horizontal position without overlap in the dishes (T.2.1) filled with water (T.2.4) and maintained at room temperature.

T.3.4 After 24 h immersion, examine the specimens :

— If the specimens have lost their uniformity, discontinue the test : the specimen is rejected.

— If the appearance of the specimens is unaffected, allow them to drain for 30 min and dry them in an oven (T.2.2) maintained at $110 \pm 5\text{ °C}$ for 2 h. Then subject each specimen to the tensile strength test (method S) but in the longitudinal direction only.

T.4 Reporting of results

T.4.1 Calculate the tensile strength R_{11} in newtons per 50 mm of specimen width as the arithmetic mean of the results of five tests, and then the ratio R_{11}/R_{10} , where R_{10} is the result of the tensile test according to method S on identical specimens, but which have not been subjected to the action of water.

T.4.2 The ratio R_{11}/R_{10} is the result of the resistance to water test.

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Method U : Reinforcement — Stability at application temperature of the test

U.1 Principle

The test consists of measuring the change in a characteristic of the reinforcement after conditioning for a specified period at a specified temperature.¹⁾

U.2 Apparatus

U.2.1 Oil bath, capable of holding 260 ± 2 °C for 2 min and of containing the specimens suspended vertically.

U.2.2 Inert thermal fluid, such as methyl-phenyl-silicone.

U.2.3 Thermometer, as specified in ASTM E 1, type 2 C⁽¹⁾.

U.2.4 Stop-watch.

U.2.5 Apparatus as for method S, tensile strength test.

U.3 Procedure

U.3.1 Carry out the test on a minimum of five specimens of dimensions 300 mm × 50 mm identical to those of the tensile strength test (see method S).

U.3.2 Place the specimens in the thermal fluid bath (U.2.1) brought to 260 ± 2 °C taking care not to crease the specimens, and ensuring that a length of about 50 mm projects out of the bath at each end (in an arc-shape); this is to avoid as far as possible alterations of those parts of the specimens to be clamped later in the tensile machine jaws. Immerse for 120 ± 5 s.

U.3.3 After 30 min draining and cooling, determine the tensile strength of each specimen by method S, but only in the longitudinal direction.

U.4 Reporting of results

U.4.1 Calculate the tensile strength, R_{12} , as the arithmetic mean of the five tensile tests in newtons per 50 mm specimen width, and then the ratio R_{12}/R_{10} , where R_{10} is the result of the tensile strength test by method S on identical specimens, not subjected to heating.

U.4.2 The ratio R_{12}/R_{10} is the result of the test for stability at application temperature on the reinforcement.

1) This experimentation is carried out only as a qualification test.