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# Protective clothing — Assessment of resistance of materials to molten metal splash

Vêtements de protection — Évaluation de la résistance des matériaux aux projections de métal fondu



Reference number ISO 9185:2007(E)

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#### **Foreword**

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 9185 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 162, *Protective clothing including hand and arm protection and lifejackets*, in collaboration with Technical Committee ISO/TC 94, *Personal safety — Protective clothing and equipment*, Subcommittee SC 13, *Protective clothing*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 9185:1990), which has been technically revised.

This second edition includes the following significant technical changes compared to the first edition:

- a) new PVC sensor film included;
- b) Cryolite included as test metal.

#### Introduction

ISO 9185:1990 and EN 373:1995 have been used up until now with reasonable success as the principle test methods for materials used in the manufacture of clothing to protect against large splashes of molten metals. EN and ISO specifications cite these test methods and set levels of performance in terms of the mass of iron or aluminium that can be splashed onto test materials without producing damage to the heat sensor film.

The revision of the test methods contained within this International Standard incorporates changes based on experience that are intended to improve reproducibility and to respond to incident data from the aluminium smelter industry. A test procedure is therefore introduced to determine the protection provided by materials when splashed with molten cryolite. This revision also harmonises into one test procedure the previously slightly different procedures in ISO 9185 and EN 373 for testing with molten aluminium.

A new supply of PVC sensor film has been established together with a new world-wide distributor – see note in the text. One single specification for PVC film replaces the previously different ones in ISO 9185 and EN 373.

The test method in this International Standard is distinct from that in ISO 9150, which assesses the protective performance of materials intended to be manufactured into protective clothing for welding activities.

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# Protective clothing — Assessment of resistance of materials to molten metal splash

#### 1 Scope

This International Standard specifies a method for assessing the heat penetration resistance of materials intended for use in clothing to protect against large splashes of molten metal. It provides specific procedures for assessing the effects of splashes of molten aluminium, molten cryolite, molten copper, molten iron and molten mild steel.

The principle of the test method is applicable to a wider range of hot molten materials than those for which specific procedures are set out, provided that appropriate measures are applied to protect the test operator. It is important to note that good resistance of a material to a pure molten metal does not guarantee a good performance against any slag that can be present in a manufacturing process.

#### 2 Normative references

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

ISO 683-1:1987, Heat-treatable steels, alloy steels and free-cutting steels — Part 1: Direct-hardening unalloyed and low-alloyed wrought steel in form of different black products

#### 3 Terms and definitions

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

#### 3.1

#### damage

(PVC sensor film) any smoothing or modification to the embossing or pinholing of the PVC sensor film, extending in total for at least 5 mm across its width

NOTE Where the visual change in appearance is in discrete spots, damage occurs when the summation of the width of each spot exceeds 5 mm across any horizontal section. For cryolite, experience indicates that damage can be defined as less than 5 mm in width, but greater than 10 mm in length.

#### 3.2

#### molten metal splash index

figure equal to the minimum mass of molten metal poured which just causes damage to the PVC sensor film

#### 4 Principle

Materials are tested by pouring quantities of molten metal onto the test specimen supported at an angle to the horizontal on a pin frame. Damage is assessed by placing an embossed thermoplastic PVC sensor film directly behind, and in contact with, the test specimen and noting changes to the film after pouring. Any adherence of the metal to the test specimen surface is also noted. Depending on the result, the test is repeated, using a greater or smaller mass of metal, until the minimum quantity to cause damage to the film is observed.

#### 5 Apparatus and materials

**5.1 Metals and cryolite** complying with the specifications set out in Annex A. Other metals or substrates appropriate to the end use.

NOTE It is advisable that coarse filings or small pieces cut from solid bar or sheet be used, because fine filings have proved difficult to melt. A range of pouring temperatures used in industry for different metals and for cryolite is given in Annex A.

**5.2 PVC** sensor film <sup>1)</sup>, comprising an embossed PVC sheet, of mass per unit area  $(300 \pm 30)$  g/m<sup>2</sup>, which when tested as described in Annex B shows no smoothing or modification of the embossing of the central area at a block temperature of  $(166 \pm 2)$  °C but which shows smoothing or modification of the central area at a block temperature of  $(183 \pm 2)$  °C. The procedure set out in Annex B shall be undertaken no more than 30 days before any one day of testing in accordance with this International Standard.

NOTE The reason for this continuous calibration of the PVC sensor film is that it is likely to change over time because of plasticizer loss. It is advisable that the PVC sensor film be stored in a cool and dark location so as to minimize such changes. Because of the economics and consistency of production, one batch of at least 1500 m is produced and then used by test laboratories over a period of several years.

**5.3 Crucible**, whose approximate external dimensions are a height of 97 mm, a top diameter of 80 mm, a bottom diameter of 56 mm and a capacity (brim full) of 190 ml (see Figure 1).

NOTE For most molten metals, including iron, a graphite impregnated material (if an induction furnace is used) has been found suitable for the crucible.

- **5.4 Detachable crucible holder**, to enable the crucible containing the molten metal to be moved quickly and safely from the furnace to the test apparatus.
- **5.5 Furnace**, capable of operating at a temperature 100 °C above the pouring temperature specified in Annex A. The furnace type may be either a muffle furnace or an induction type furnace.

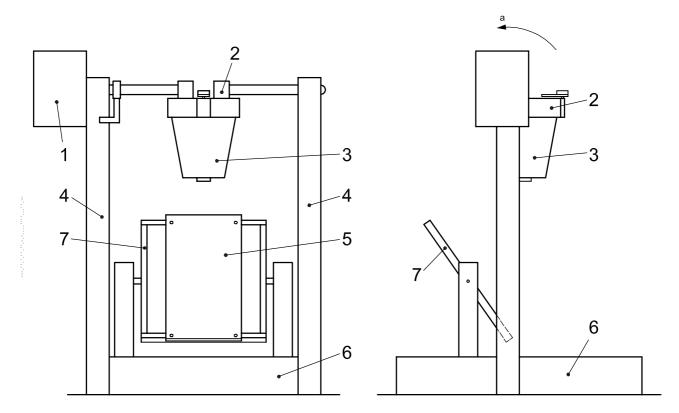
NOTE Muffle furnaces are capable of holding at least four crucibles (i.e. internal furnace size is typically  $135 \text{ mm} \times 190 \text{ mm} \times 780 \text{ mm}$ ), but they take several hours to melt metals such as steel, iron and copper. Induction furnaces melt a single crucible of these metals in less than half an hour.

- **5.6 Temperature probe**, either a small thermocouple  $^{2)}$  or an optical non-contact temperature device, capable of measuring molten metal temperatures up to 1 650 °C to an accuracy of  $\pm$  10 °C.
- **5.7 Pouring apparatus**, shown in Figure 1, consisting of the pouring device, a means of rotating the pouring device at constant angular velocity, a specimen holder with supporting frame and a sand tray.

The pouring device, consisting of crucible holder and drive shaft, shall be designed and constructed so that the point at which the molten metal pours from the crucible lies on the axis of rotation of the drive shaft. The pouring device shall be manufactured from steel.

<sup>1)</sup> The PVC sensor film is supplied by Health & Safety Laboratory, Harpur Hill, Buxton, SK17 9JN, England. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO. Equivalent products may be used if they can be shown to lead to the same results.

<sup>2)</sup> A suitable device is a long U-tube thermocouple unit known as a dipstick, which can be obtained from Heraeus Electro – Nite Ltd., Chesterfield, S41 9ED, England. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO. Equivalent products may be used if they can be shown to lead to the same results.



#### Key

- 1 motor
- 2 crucible holder
- 3 crucible
- 4 adjustable support
- 5 test specimen
- 6 sand tray
- 7 specimen holder
- a Direction of tip.

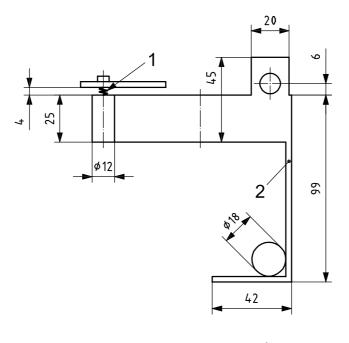
Figure 1 — Motor driven crucible

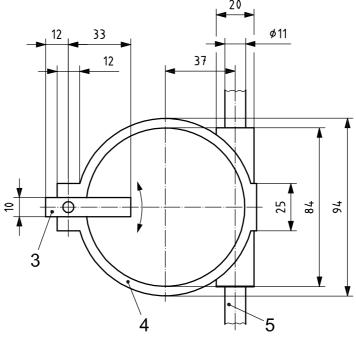
Figure 2 shows an example of a suitable design, using a straight drive shaft and a crucible holder into which the crucible fits with its top almost flush to the top surface of the crucible holder.

Figure 3 shows an example of equipment that incorporates a cranked drive shaft with a crucible holder into which the crucible fits with its top on the pivot axis. Thus, in this equipment, the top of the crucible does not fit flush with the top surface of the crucible holder.

However, in both these pouring devices, the axis of rotation passes through the pouring edge of the crucible, as required.

Dimensions in millimetres

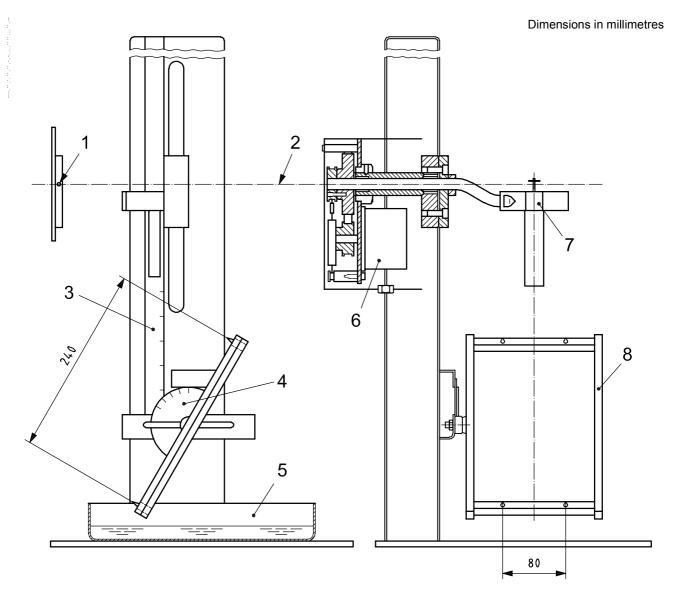




#### Key

- 1 coil spring
- 2 crucible support
- 3 retaining clip
- 4 ring
- 5 shaft

Figure 2 — Pouring device



#### Key

- 1 pin as indicator
- 2 pivot
- 3 scale for pour height
- 4 scale for test specimen angle
- 5 sand tray
- 6 stepper motor
- 7 crucible holder
- 8 specimen holder

Figure 3 — Alternative pouring device with cranked shaft drive

The specimen holder shall consist of a rectangular pin frame,  $(160 \pm 2)$  mm x  $(248 \pm 2)$  mm external dimension from 8 mm square steel. It shall have four pins, two on the centreline of the top of the frame and two on the centreline of the bottom of the frame, spaced  $(80 \pm 2)$  mm apart in the width direction,  $(240 \pm 2)$  mm in the length direction and  $(40 \pm 2)$  mm from the respective corners.

The pin frame shall be supported on a suitable frame which enables the angle of the specimen to the horizontal to be varied (see Annex A) and the position of the test specimen relative to the pouring device to be adjusted. It is recommended that the initial impact of molten metal or cryolite be not below the centre of the test specimen. This initial impact shall not be within the upper 25 mm of the test specimen.

Examples of suitable pouring apparatus are shown in Figures 1 and 2, both fitted with electric stepper motors as the means of rotating the pouring device at constant angular velocity.

The pouring device shall be firmly supported by a means that allows adjustment of the pour height, measured at the vertical distance from the drive shaft to the centre of the pin frame, in order that the value specified in Annex A can be achieved.

The pouring apparatus should incorporate a means of holding the pouring device stationary in its rest position. In the pouring apparatus shown in Figure 1, this is achieved by a metal stop.

The sand tray shall have minimum dimensions of approximately 250 mm wide x 350 mm long x 50 mm deep and shall be filled with dry sand to a depth of 30 mm to 40 mm.

- 5.8 Balance, capable of weighing to an accuracy of 1,0 g.
- 5.9 Template, in the form of a rigid rectangle  $(260 \pm 2) \text{ mm} \times (100 \pm 2) \text{ mm}$ , with four holes of 5 mm diameter, one in each corner and  $(10 \pm 1)$  mm from the two adjacent edges, their centres forming the corners of a rectangle (240  $\pm$  2) mm x (80  $\pm$  2) mm.

#### Conditioning 6

Condition the test specimens for at least 24 h in an atmosphere having a temperature of (20  $\pm$  2)  $^{\circ}$ C and a relative humidity of  $(65 \pm 5)$  %.

If testing is not carried out immediately after conditioning, place the conditioned test specimens in a sealed container. Begin testing each specimen within 2 min of removing it from either the conditioning atmosphere or the sealed container.

For testing, an atmosphere substantially free from draughts and having a temperature of between 10 °C and 30 °C and a relative humidity of between 15 % and 80 % shall be used.

#### Preparation of test specimens 7

Lay out the laboratory sample without tension, but free from wrinkles and creases, on a flat, smooth surface. First mark and cut seven test specimens using the template with the longer length in the machine direction, except where this does not apply (e.g. leather, when the direction of cutting is unimportant). Make use of the template to mark the position for the pins (of the pin frame) on the material by spots approximately 2 mm in diameter at the centres of the holes in the template. Cut a similar number of pieces of PVC sensor film but do not mark the position of the pins (see 9.3).

The assessment uses an iterative procedure, and therefore the exact number of test specimens needed NOTE cannot be stated. Seven test specimens are usually sufficient to give a result. If there is previous experience of the material or if a material is being assessed for compliance with a specification, fewer test specimens will be needed.

#### Operator safety

Protective clothing and equipment meeting the requirements of ISO and CEN standards shall be worn by the operator, in order to protect against the hazard of accidental splashes of molten metal or cryolite. The risk assessment shall also consider protection against smoke and/or toxic fumes.

WARNING — In addition to the hazard of molten metal splashes, certain metals (e.g. sodium) ignite spontaneously when heated in air and produce toxic fumes when so heated. Additional safety measures will therefore be necessary when testing the resistance of materials to these metals. The characteristics of cryolite and of all molten metals intended to be used in this test shall be fully understood by the operator.

#### 9 Procedure

#### 9.1 Setting up the apparatus

Adjust the position of the pouring device to give the pour height specified in Annex A for the molten metal being used. The pour height is the vertical distance from the drive shaft to the centre of the pin frame. Adjust the angle of the pin frame to give the specimen angle to the horizontal specified in Annex A.

#### 9.2 Preparation of molten metal or cryolite

Place approximately 50 g (weighed to the nearest gram) of metal or cryolite, or multiple of 50 g if it is known that the material under test will withstand a higher amount, either coarse ground or cut from bar or sheet, into the dry crucible and melt it to a temperature slightly above that at which it will remain molten throughout the test (see Annex A).

#### 9.3 Attachment of test material to pin frame

Attach a piece of PVC film with the embossed side uppermost, so that it is under sufficient tension to be flat. Locate the test specimen over the PVC film by pushing the pins through the marked positions. Ensure that the test specimen and the PVC film are in contact over their entire area and that the test specimen is free from creases. In addition, ensure that the surface of the test specimen intended to be the outer surface of a garment is exposed to the molten metal or cryolite.

#### 9.4 Pouring

#### 9.4.1 Pouring of molten metal

Carefully transfer the crucible, using the detachable crucible holder, to the pouring ring. Allow the molten metal to cool to the pouring temperature (see Annex A) and then operate the pouring device so that the crucible turns through at least 130° from the horizontal at a constant rate of  $(36 \pm 2,5)$ ° per s. This rate is equivalent to a rotation of 90° in  $(2,5 \pm 0,2)$  s. Pour the metal over the rim of the crucible, not via any pouring lip, and ensure that an undamaged region of the rim surface is used.

NOTE Experience has shown that crucible rims can become degraded with use, such that they alter the flow of molten metal or cryolite over the rim. If the rim of a crucible becomes degraded such that it is no longer smooth and/or not of original profile, it is advisable that the crucible be discarded. It is advisable that crucibles be thoroughly decontaminated between tests by appropriate means such as scraping and/or baking, in order to remove solidified residues.

#### 9.4.2 Pouring of molten cryolite

When using this material, the crucible shall turn through at least  $130^{\circ}$  from the horizontal, but at a decreased constant rate of  $(18 \pm 1.5)^{\circ}$  per s.

#### 9.4.3 Additional procedures

Any molten metal or cryolite that has solidified on a test specimen shall not be reused.

NOTE It is recommended that metals or cryolite not be reused if there is evidence of contamination and/or degradation.

#### 9.5 Examination

- **9.5.1** 30 s after completion of pouring, remove the test specimen and examine the PVC film for any sign of damage (see 3.1). Note any such damage.
- **9.5.2** Note and record whether any molten metal has solidified and adhered to the surface of the test specimen.

#### Determination of mass of metal poured

Allow any metal remaining in the crucible to solidify sufficiently for it to be scraped out. Weigh this residue to the nearest gram and subtract it from the initial mass of metal melted. Record this as "metal poured".

#### 10 Iterative testing

- 10.1 If there is no damage to the PVC film, repeat the test procedure using new test specimens of material and PVC film and using a quantity of metal in the crucible 50 g greater than that used in the previous test. If the capacity of the crucible is reached, the test is not sufficiently severe to obtain film damage. If damage is observed, proceed to 10.2.
- 10.2 Repeat the test procedure using a quantity of metal in the crucible 10 g less than that used in the previous test. If damage to the PVC film is observed, repeat from 10.2. If no damage to the PVC film is observed proceed to 10.3.
- 10.3 Repeat the test procedure using the same quantity of metal in the crucible used in the previous test. If damage to the PVC film is observed, repeat from 10.2. If no damage to the PVC film is observed, repeat from 10.3 until four successive tests show no damage to the PVC film.
- **10.4** Note the highest value of the mass of metal poured (see 9.6) in these four successive tests, and the lowest mass of metal poured that caused damage.
- 10.5 Record the mean of these two values to the nearest gram as the "molten metal splash index".

#### 11 Void tests

If any of the following occurs during a test, declare the test void and repeat the test using the same mass of metal:

- a) the impact of the pour wanders horizontally across the test specimen;
- the metal runs off the side of the test specimen or strikes within 25 mm of the top edge; b)
- any of the molten metal does not first hit the test specimen; c)
- the metal is not completely molten when poured; d)
- the PVC film ignites due to metal solidifying to the pin frame.

#### 12 Test report

The test report shall include the following:

- a reference to this International Standard;
- for each individual test specimen, the approximate mass of metal used (see 9.2), whether any molten metal adhered to the material, the result of the assessment of the PVC film and the mass of molten metal or cryolite poured;
- the molten metal or cryolite splash index calculated as specified in Clause 10;
- the metal or specific source of cryolite used, the pouring temperature, the specimen angle to the horizontal and the pour height;
- any deviations from the test procedure likely to have had an influence on the test result.

# Annex A

(normative)

### Test conditions for certain metals and for cryolite

As this International Standard consists solely of a test method, it does not specify performance levels for materials but does enable comparisons to be made between materials in terms of the protection provided against specific molten metals and cryolite. The conditions specified in Table A.1 have proven appropriate for the metals listed and for cryolite. The specimen angle shown for aluminium reflects the need to increase the sensitivity of the test for this metal in order to be able to compare materials more readily, and demonstrates the flexibility of the basic method in that it enables a wide range of metals to be assessed. For cryolite, the rate of rotation of the crucible, the specimen angle and the pour height need to differ from the values set for the metals because of the much lower viscosity for cryolite.

Temperatures to which metals and cryolite are heated before pouring are slightly higher than pouring temperatures, in order to allow for cooling during transfer from furnace to pouring apparatus. For substances poured at higher temperatures, the rate of cooling is greater than when poured at lower temperatures and therefore they need to be heated to a higher temperature to accommodate transference from furnace to crucible holder. The critical temperature is the pouring temperature, which can be estimated by use of predrawn temperature/time curves (cooling curves). The following "temperatures of removal from furnace" were found to be practical for the given metals using an induction furnace, and they enable the pouring temperatures specified in Table A.1 to be achieved:

 Aluminium	820 °C
 Copper	1 350 °C
 Iron	1 500 °C
 Mild steel	1 650 °C
 Cryolite	1 200 °C

Table A.1 — Pouring temperatures, pour heights and specimen angles to the horizontal for various molten metals

Metal	Pouring temperature	Pour height	Specimen angle to the horizontal		
	°C	mm	0		
Aluminium consisting of at least 99,5 % by mass of aluminium	780 ± 20	225 ± 5	60 ± 1		
Copper consisting of at least 99 % by mass of copper	1 280 ± 20	225 ± 5	75 ± 1		
Iron consisting of at least 93 % by mass of iron and containing the following:					
— C 2,8 % to 3,2 % — Si 1,2 % to 2,0 %	1 400 ± 20	225 ± 5	75 ± 1		
— P 0,3 % to 0,6 %					
Mild steel complying with designation C25, as specified in ISO 683-1:1987	1 550 ± 20	225 ± 5	75 ± 1		
Industrial grade cryolite	1 120 ± 20	300 ± 5	70 ± 1		

#### Annex B

(normative)

# Method of test for assessment of thermal characteristics of PVC sensor film

#### **B.1 Principle**

An aluminium block is heated to a specified temperature and placed on the PVC film. The block is removed after a specified time and the embossing on the PVC film is examined for signs of smoothing.

#### **B.2 Apparatus**

- **B.2.1 Cylindrical aluminium block**,  $(75 \pm 2)$  mm in diameter,  $(70 \pm 2)$  mm in height and of mass  $(880 \pm 50)$  g. One of the ends of the block shall be machined flat and the edge radiuses. A thermocouple for measuring its temperature shall be inserted into a hole drilled close to and parallel to the machined face.
- **B.2.2** Steel plate, not less than 100 mm in diameter and not less than 10 mm thick. One surface of the plate shall be machined flat.
- **B.2.3** Oven, capable of heating the aluminium block to 185 °C.
- B.2.4 Stop watch.

#### **B.3 Procedure**

Cut two circular test specimens not less than 100 mm in diameter from the PVC film. Position the steel plate (**B.2.2**) with its machined face uppermost.

Place a test specimen with the embossed face uppermost on the steel plate.

Heat the aluminium block (**B.2.1**) to a temperature of (166  $\pm$  2) °C.

Remove the aluminium block from the oven (**B.2.3**) and place on the test specimen with the machined surface in contact with the embossing. Start the stop watch (**B.2.4**). After 5 s, remove the aluminium block. Examine the surface of the test specimen in the centre of the area previously covered by the aluminium block for signs of smoothing or modification.

Repeat the procedure with the other test specimen using an aluminium block temperature of (183 ± 2) °C.

It is essential that the machined surface of the aluminium block is clean before the start of each test.

#### **B.4** Test report

Report whether or not any smoothing or modification of the embossing has occurred in the central area at either of the two aluminium block temperatures.

## **Bibliography**

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	nolten metal

Ī	2]	EN 373.	Protective clothing	a — Assessmen	t of	resistance o	f materials	to molte	en metal	splas	st

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