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Monolithic (unshaped) refractory products —

Part 5: Preparation and treatment of test pieces

Produits réfractaires monolithiques (non façonnés) —

Partie 5: Préparation et traitement des éprouvettes

Reference number
ISO 1927-5:2012(E)

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Foreword

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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 1927-5 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 1927 consists of the following parts, under the general title *Monolithic (unshaped) refractory products*:

— *Part 1: Introduction and classification*

— *Part 2: Sampling for testing*

— *Part 3: Characterization as received*

— *Part 4: Determination of consistency of castables*

— *Part 5: Preparation and treatment of test pieces*

— *Part 6: Measurement of physical properties*

— *Part 7: Tests on pre-formed shapes*

— *Part 8: Determination of complementary properties*

Introduction

The values of the properties obtained using test pieces are used to assess the homogeneity of monolithic (unshaped) materials. They are reference values which do not necessarily correspond with those obtained in industrial applications. Other methods of test-piece preparation or treatment, which differ from those specified by this part of ISO 1927, can lead to different values.

Monolithic (unshaped) refractory products —

Part 5:
Preparation and treatment of test pieces

1 Scope

This part of ISO 1927 specifies methods for the preparation and treatment (curing, drying and firing) of test pieces from monolithic (unshaped) refractory materials.

The methods are applicable to dense and insulating castables and to ramming materials with the four types of chemical composition defined in ISO 1927-1.

The dimensions of the test pieces are specified and the preparation of the mixture, compaction methods, storage and post-treatment of the test pieces are described.

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 1927-1, *Monolithic (unshaped) refractory products — Part 1: Introduction and classification*

ISO 1927-2, *Monolithic (unshaped) refractory products — Part 2: Sampling for testing*

ISO 1927-4, *Monolithic (unshaped) refractory products — Part 4: Determination of consistency of castables*

ISO 10060, *Dense shaped refractory products — Test methods for products containing carbon*

3 Dimensions of test pieces

Shape A: Length: 230 mm; width: 114 mm; thickness: 64 mm;

Shape B: Length: 230 mm; width: 64 mm; thickness: 54 mm;

Shape C: Length: 230 mm; width: 64 mm; thickness: 64 mm;

Shape D: Length: 160 mm; width: 40 mm; thickness: 40 mm.

The width of the test piece as tested shall correspond to the height during preparation. The vibration of the test piece during preparation shall be recorded, and for shapes C and D the compaction surface shall be marked for reference. The selection of test pieces for each type of material shall be as given in Table 1, except that for basic dense castables, ramming materials, taphole mixes and dry vibration mixes, test pieces with a diameter of 50 mm and height of 50 mm ± 1 mm are permissible and can be prepared using the sand-rammer. Shape C shall be used as the referee shape for inter laboratory testing.

Table 1 — Type of shape for tests

Castables	Dense castables	Max grain size <15 mm		Shape B or C or D	Shape A
			Direct characterization [b]	X	
			Other tests		X
		Max grain size >15 mm	Direct characterization [b]	X [a]	
			Other tests		X
	Insulating castables				X
Ramming materials	Ramming mixes			X	
	Plastics			X	

[a] For these materials, shapes B and C are prepared by cutting from shape A.

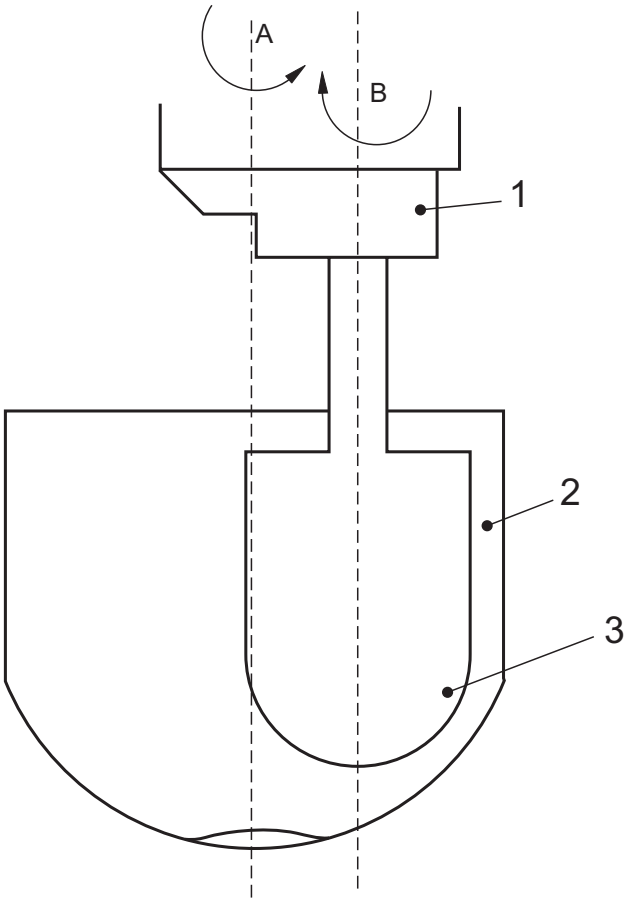
[b] Tests for which results are directly obtained from the bars without size modifications are modulus of rupture, cold compressive strength, and permanent change in dimensions on heating.

4 Apparatus

4.1 Separator. A riffle sampler, suitable for use with the maximum particle size of the product, or a shovel for the quartering method. The riffle separation shall be at least 2,5 times the maximum grain size.

4.2 Mixer, comprising the following.

4.2.1 Pan. The pan shall be symmetrical around axis A and shall have a capacity of 15 l to 30 l. Both the pan and the mixing blade (see 4.2.2) shall be constructed from a material that does not react with the test material (see Figure 1).



Key

1 Drive
2 Pan
3 Mixing blade

Figure 1 — Principle of the mixer

4.2.2 Mixing blade. The shape of the mixing blade shall be adapted to the internal dimensions of the pan. The radius swept shall be such that the distance between the blade and the wall of the pan and the space between the blade and the bottom of the pan are at least as large as the maximum grain size of the material. For products with a maximum grain size of 6 mm or less, the distance between the blade and the wall shall be 6 mm. For products with larger grain sizes (up to 25 mm), the blade is used in a manner such that the distance to the wall of the pan is 25 mm.

The mixing blade shall revolve at a speed between 40 r/min and 65 r/min around axis A (the symmetry axis of the mixing pan), the blade rotating simultaneously in the opposite direction at a speed between 120 r/min and 145 r/min around axis B (symmetry axis of the blade).

For low-intensity mechanical mixing of insulating castables the mixing blade shall revolve at a speed between 15 r/min and 25 r/min around axis A (the symmetry axis of the mixing pan), the blade rotating simultaneously in the opposite direction at a speed between 50 r/min and 80 r/min around axis B (symmetry axis of the blade).

4.3 Vibrating table. The vibrating table shall be flat and horizontal and shall perform only uniaxial vertical vibrations at a frequency of 50 Hz. The table shall be capable of being set at a double amplitude of 0,50 mm (see ISO 1927-1) with an accuracy of ± 0,05 mm for the entire procedure. There shall be an automatic adjustment to the required double amplitude according to the mass of the mould and material.

4.4 Pneumatic rammer. A compressed-air rammer shall have a rammer foot suitable for the width of the mould and have a smooth, flat working surface, i.e. 52 mm × 25 mm for shape B and 62 mm × 25 mm for shape C.

The mass of the rammer and the frequency of ramming shall be chosen in order to obtain a prescribed green bulk density, which shall be reported in the test report.

4.5 Sand-rammer, consisting of a mould of 50 mm inside diameter, and 140 mm in length, and a 6,67 kg ± 50 g weight sliding on the shaft of the apparatus and arranged to fall a distance of 50 mm before engaging a collar attached to the shaft. At the lower end of the shaft there is a plunger, the diameter of which is about 0,3 mm smaller than the inside diameter of the mould (see Figures 2 and 3).

NOTE A more detailed description of this piece of apparatus is given in ISO 1927-3.

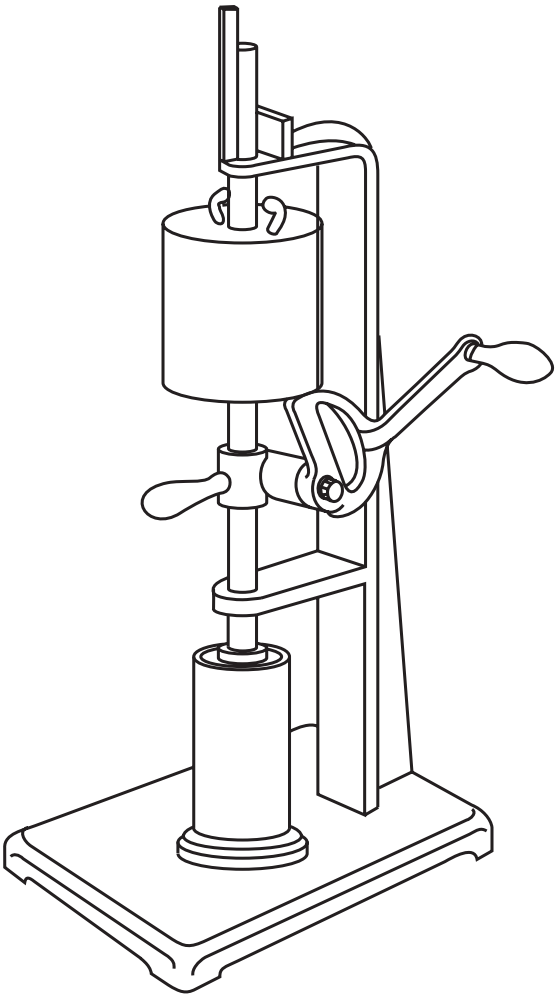


Figure 2 — Sand-rammer for shaping ramming mixes test pieces

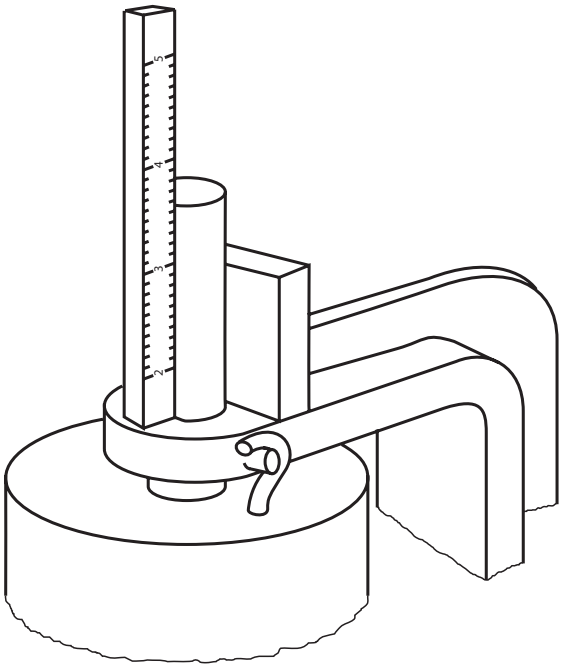


Figure 3 — Modification of sand-rammer for workability test

4.6 Hydraulic-type power press, equipped with suitable moulds for forming test pieces of the required size. The press shall be capable of applying a minimum of 10 MPa pressure to the moulded face.

NOTE The power press should only be used for shaping test pieces from plastics.

4.7 Trowel, pointing type or stiff-bladed spatula of typical size 150 mm length and 50 mm width.

4.8 Watertight moulds, capable of being dismantled and shall be watertight. They shall be made from a material that does not react with the material to be tested.

For compaction by the pneumatic rammer, the moulds shall be rigid so that they do not warp during ramming.

NOTE 1 Steel or similar material is recommended to withstand the stroke of the rammer.

The internal measurements of the moulds are determined by the dimensions of the test pieces. The surfaces 230 mm × 64 mm (shapes A and C) and 230 mm × 54 mm (shape B) are the horizontal surfaces during the compaction process. A variation of ± 0,5 mm is allowed for these dimensions. If multi-compartment moulds are used, the size of the mould shall be adapted for the number of test pieces prepared and this shall be indicated in the test report.

In order to overfill the mould, an overfill ring is required for all unshaped materials. This ring shall taper slightly upwards. For castables, it can at the same time serve as a clamp to the vibrating table. The mould, the overfill ring and the clamp shall have sufficient rigidity to ensure that only the induced vibrations and the required frequency and amplitude will occur.

NOTE 2 It is recommended that all internal surfaces of the mould are slightly oiled.

4.9 Two balances, one capable of weighing up to 25 kg with an accuracy of ± 10 g and the other capable of weighing up to 5 kg with an accuracy of ± 1 g.

4.10 Steel lath, of typical size 500 mm × 30 mm × 5 mm for scraping off overfill after casting.

4.11 Humidity cabinet, capable of maintaining a relative humidity of 90 % or greater, and of controlling the temperature between 18 °C and 22 °C.

4.12 Drying oven, fan-assisted and having openings to enable efficient ventilation.

4.13 Firing furnace. Electric or gas-fired furnace for firing test pieces with a temperature distribution over the hot zone of ± 10 °C. The heating rate shall be programmable.

Electric firing shall be used as the referee method.

4.14 Stopwatch or stopclock.

4.15 Thermometer.

4.16 Silicon carbide box, with cover, with dimensions such that it is capable of containing 2 or 4 test pieces (shape B or C) with approximately 20 mm spaces between the test pieces, and the test pieces, sides, top and bottom of the box. The box is filled with granules, 0,5 mm to 2 mm in size.

NOTE The wall thickness should be as thin as possible and the thermal conductivity as high as possible to minimize the temperature gradient.

4.17 Water. Pure mains water of drinking quality.

5 Preparation of castable test pieces

5.1 Dense castables

5.1.1 Preparation of the material for shaping

Reduce the amount of material to the required quantity with the riffle sampler or the shovel (see 4.1) in order to obtain the desired batch size for testing and thoroughly mix before use. The batch size depends on the number of test pieces to be prepared. The number of test pieces to be prepared shall be chosen in accordance with ISO 1927-2.

In cases where several components are supplied separately, mix each material first carefully by itself, then mix all carefully together. Before shaping the test pieces, maintain the material at a temperature between 18 °C and 22 °C for 24 h.

Water or a special mixing liquid (necessary for the mix and supplied by the manufacturer) can be used for mixing. When water is used, pure mains water with a maximum degree of hardness of 30° is used, and its temperature maintained between 18 °C and 22 °C.

Determine the amount of liquid either by a consistency test in accordance with ISO 1927-4, or use the amount stipulated by the manufacturer. Pour the amount of the dry material required into the mixer and mix for 30 s for homogenization. Add the liquid with an accuracy of 0,1 g per 100 g of dry substance. After having made a crater in the centre of the material, pour the liquid progressively into the crater and start the mixer. Add the remaining liquid in less than 1 min. Note the relation, E, between the quantity of water and the quantity of dry material (in ml/g).

Mix the batch for 2 min in the case of regular castables and for 4 min in the case of defloculated castables. If necessary, switch the mixer off after half the mixing time, in order to scrape off the adhering material at the edges of the mixer.

NOTE 1 The wet mixing time should not be less than 2 min nor exceed 8 min, depending on the product type or the time recommended by the manufacturer.

NOTE 2 Generally, deflocculated castables require a longer mixing time than regular castables, and a minimum of 4 min can be expected

Record the temperature of the batch before shaping.

5.1.2 Shaping of test pieces

5.1.2.1 Compaction by vibration

The mix prepared in accordance with the instructions in 5.1.1 is compacted by vibration. The total time for the preparation of the mix and for making the test pieces shall not exceed 12 min.

Fill the mould fitted with its overfill ring, vibrate the mix according to the time and as indicated in Table 2. During the initial vibration, add material to the mould so that the level of material fills the overfill ring at completion of vibration.

Table 2 — Time and double amplitude for compaction by vibration

Material	Type of castable	Total time of vibration min	Double amplitude mm
Alumino-silicate and special dense castables	Regular	1	0,50
	Defloculated + chemically bonded	5 max.	0,50 [a]
Basic dense castables [b]		4	0,50 [a]
Carbon containing dense castables	Regular	1	0,50
	Defloculated + chemically bonded	5 max.	0,50 [a]

[a] Alternative double amplitudes for these materials can be used by agreement between parties and are reported.

[b] For basic dense castables, the sand-rammer can also be used for preparing test pieces (see 6.1.2.2).

Check the double amplitude of the vibration table during the compaction process and correct if necessary. Remove the overfill ring and level the surface of the material in the mould with the steel lath (see 4.10). Remove the mould from the vibrating table.

5.1.2.2 Compaction by self-flowing

The mix prepared according to the instructions described in 5.1.1 is compacted by free de-airing. The total time for the preparation of the mix and for making the test pieces shall not exceed 10 min.

Fill the mould, without its overfill ring, with a moderate speed so as to obtain an accurate levelling between the top of the mould and the product. Let it lie without any movement until set, leaving the mould at the same place in the humidity cabinet (see 4.11) or airtight plastic bag in the curing room (see 7.1.1).

5.2 Insulating castables

5.2.1 Preparation of the material for shaping

5.2.1.1 General

Reduce the amount of the material for shaping with the riffle sampler or the shovel (see 4.1) in order to obtain the desired batch size for testing and thoroughly mix before use. The batch size depends on the number of test pieces to be prepared. The number of test pieces to be prepared shall be chosen in accordance with ISO 1927-2.

In cases where several components are supplied separately, mix each material first carefully by itself, then mix all carefully together. Before the shaping of test pieces, maintain the material for testing at a temperature between 18 °C and 22 °C for 24 h.

Water or a special mixing liquid (necessary for the mix and supplied by the manufacturer) can be used for mixing. When water is used, pure mains water with a maximum degree of hardness of 30° is to be used, and its temperature shall be maintained between 18 °C and 22 °C.

Determine the amount of liquid by a consistency test in accordance with ISO 1927-4 when the castable is shaped by vibration. When the castable is shaped by rodding, use the amount of liquid recommended by the supplier. Add the liquid with an accuracy of 0,2 g of water per 100 g of dry substance.

Pour the amount of dry material required either into a trough or into the low-intensity mixer bowl (see 4.2) and mix for 30 s for homogenization. Make a crater in the centre of the material and proceed in accordance with either 5.2.1.2 or 5.2.1.3. The method of shaping (i.e. rodding or vibration) shall be defined in agreement with the recommendations of the supplier and in accordance with the consistency test (see ISO 1927-4).

5.2.1.2 Manual mixing (for compaction by rodding or vibration)

Pour the mixing water progressively into the crater and start the manual mixing immediately. Note the relation, *E*, between the quantity of water and the quantity of dry material (in ml/g).

The total time of manual mixing is 5 min.

NOTE Manual mixing is recommended for lightweight aggregates.

5.2.1.3 Mechanical mixing (for compaction by rodding or vibration)

Pour the mixing water progressively into the crater and start the mixer immediately.

Note the relation, E, between the quantity of water and the quantity of dry material (in ml/g).

Mix the batch for between 2 min and 6 min, according to the manufacturer's instructions. If necessary, switch off the mixer after 2 min mixing time, in order to scrape off the adhering material at the edges of the mixer.

5.2.2 Shaping of test pieces

5.2.2.1 Compaction by rodding

The mix prepared according to the instructions described in 5.2.1.1 and 5.2.1.2 or 5.2.1.3 is compacted by rodding. The total time for the preparation of the mix and for making the test pieces shall not exceed 10 min. The consistency of the fresh castable shall be such that no pressure is needed for deformation, but not be so fluid that segregation can occur.

Weigh the empty mould and record the mass as P_1 and note its nominal volume V_m. Fill the mould with its overfill ring and carry out the rodding action on the surface with a trowel, down to the bottom of the mould. Move the trowel uniformly in the mould, the spatula forming an angle of 45° with the sidewall of the mould.

Fill the mould with its overfill ring again and carry out the same operation. After moulding, remove the overfill ring and level the surface of the material in the mould with the steel lath (see 4.10). Smooth the surface without adding any more water.

Weigh the mould (P_2).

5.2.2.2 Compaction by vibration

The mix prepared according to the instructions described in 5.2.1.1 and 5.2.1.2 or 5.2.1.3 is compacted by vibration. The total time for the preparation of the mix and for making the test pieces shall not exceed 10 min.

Weigh the empty mould and record the mass as P_1 and note its nominal volume V_m. Fix the mould on the vibrating table and fill it with its overfill ring in place.

Set the double amplitude of the vibrating table for 0,75 mm. Check it during the compaction process and correct if necessary.

Vibrate the mix for 1 min. During vibration, add material to the mould so that the level of material reaches the top of the overfill ring, and continue the vibration to its completion. Remove the overfill ring and level the surface of the material in the mould with the steel lath.

Weigh the mould and record the mass as P_2.

5.2.3 Calculation of dry yield density

Calculate the dry yield density, V_R , in kilograms per cubic metre using the equation:

$$V_R = \frac{100(P_2 - P_1)}{V_m(E + 100)}$$

where

P_1 is the mass of empty mould, expressed in kilograms;

P_2 is the mass of full mould, expressed in kilograms;

V_m is the volume of mould, expressed in cubic metres;

E is the percentage of water by mass.

As an alternative to the calculation of dry yield density, the bulk density after firing to 400 °C may be determined in accordance with ISO 5016.

6 Preparation of test pieces from ramming materials, taphole clay and dry vibration mixes

6.1 Ramming mixes

6.1.1 Preparation of material for shaping

Reduce the amount of the material for shaping with the separator or shovel (see 4.1) in order to obtain the desired batch size for testing and mix thoroughly before use. The batch size depends on the number of test pieces which are to be prepared (see ISO 1927-2).

Before the shaping of test pieces, maintain the material for testing at a temperature between 18 °C and 22 °C for 24 h.

Where the ramming mix is supplied ready for use, it is normally formed into test pieces as received, but if necessary, after agreement with the supplier, some liquid may be added.

Where the ramming mix is supplied dry, mix the dry material for 30 s, and add the liquid for mixing evenly in the next 20 s. Continue mixing for between 2 min and 6 min. The amount of liquid to be added to dry ramming mixes will be stated by the manufacturer, who also may recommend a mixing time. If necessary, switch the mixer off after 1 min mixing time in order to scrape off the adhering material at the edges of the mixer. Report the water addition and mixing time.

6.1.2 Shaping of test pieces

6.1.2.1 Compaction with the pneumatic rammer

The ramming mix prepared according to the instructions described in 6.1.1 is compacted in several layers with a pneumatic rammer.

The quantity of material shall be such that a test piece is obtained which protrudes 5 mm to 15 mm above the top of the mould. The total quantity is derived from the green bulk density quoted by the manufacturer and the volume of the mould together with the overfill ring. The number of layers shall be four for shapes B and C. The weighed quantity for the individual layers shall be 1/4 of the total quantity.

Fill the amount of material for the first layer evenly into the mould. Start the ramming process with the pneumatic rammer at one end and complete in uniform steps towards the other end, then ram in the opposite direction so that the starting point is reached. Repeat this process until sufficient compaction is achieved for the quality of material.

Roughen the surface of the compacted layer with an appropriate tool to obtain a good key for the next layer to be compacted. Repeat this procedure to obtain the four layers required.

After compaction of the last layer, remove the overfill ring and scrape the mix over the rim with the steel lath (see 4.10), using a slight sawing action, and smooth the surface. Before dismantling, allow a setting time in accordance with the manufacturer's instructions.

Dismantle the mould and remove the test pieces. Use two support pieces against the sides in order to pick up the test piece and move it subsequently for measuring and weighing. Determine the mass to the nearest 1 g and the geometrical dimensions to the nearest 0,1 mm. Calculate the green bulk density.

Indicate the number of forming cycles per layer, and the mass of the rammer, in the test report.

6.1.2.2 Compaction with a sand-rammer

This procedure is applicable to all ramming materials. The mix prepared according to the instructions described in 6.1.1 is compacted in one layer with a sand-rammer (see 4.5).

When required, the appropriate amount of water, if not indicated by the manufacturer, shall be chosen within a range of 6 % to 8 %, so that test cylinders can be prepared by applying 40 strokes. If a different number of strokes is required, it shall be determined by the difference of the height of the cylinder after $(n - 10)$ strokes and (n) strokes, which shall be less than 2 %. In this case, indicate the number of strokes in the test report.

The height of the test piece should now be 50 mm $\pm$ 1 mm, otherwise repeat the procedure to obtain an appropriate green bulk density.

6.2 Plastics

6.2.1 Preparation of the material for shaping

Before preparation, maintain the blocks at a temperature between 18 °C and 22 °C for 24 h, taking care that no loss in moisture will occur during that time.

In the case of plastics delivered ready for use, the amount of the material for shaping shall be taken from blocks divided manually into small pieces of maximum dimension 25 mm. The material to be tested shall correspond to a total section of the block taken out in its middle.

Carry out the compaction immediately after dividing the blocks.

6.2.2 Shaping of the test pieces

6.2.2.1 Compaction with the pneumatic rammer

The material prepared according to the instructions in 6.2.1 is compacted in several layers with a pneumatic rammer.

The quantity of material shall be such that a test piece is obtained which protrudes 5 mm to 15 mm above the top of the mould. The total quantity is derived from the green bulk density quoted by the manufacturer and the volume of the mould together with the overfill ring. The number of layers shall be 2 or 3 for shapes B and C and the weighed quantity for the individual layers shall be 1/2 or 1/3 of the total quantity.

Fill the amount of material for the first layer evenly into the mould. Start the ramming process with the pneumatic rammer at one end and complete in uniform steps towards the other end, then ram in the opposite direction so that the starting point is reached. Repeat this process until sufficient compaction is achieved for the quantity of material.

Roughen the surface of the compacted layer with an appropriate tool to obtain a good key for the next layer to be compacted. Repeat this procedure to obtain the two or three layers required.

After compaction of the last layer, remove the overfill ring and scrape the material over the rim with the steel lath (see 4.10) using a slight sawing action and smooth the surface.

Dismantle the mould and remove the test pieces. Use two support pieces against the sides in order to pick up the test piece and move it subsequently for measuring and weighing. Determine the mass to ± 1 g and the geometrical dimensions to ± 0,1 mm. Calculate the green bulk density.

Indicate the number of forming cycles, the mass of the rammer and the number of layers (two or three) in the test report.

6.2.2.2 Compaction with power press

The material prepared according to the instruction in 6.1.1 is compacted in a single layer with a power press.

Fill the mould and apply sufficient pressure to achieve a well compacted test piece, close to the green bulk density specified by the manufacturer's instructions. The pressure shall not be so high that a portion of the material is forced out of the mould by extrusion through the clearance space located between the plunger of the mould and the walls of the die cavity.

To eliminate possible entrapped air, apply an initial load, relieve this pressure and then increase to the selected pressure. Thrust the test piece out from the mould and remove it. If necessary, use two support pieces against the sides of the test piece in order to pick up the test piece and move it subsequently for measuring and weighing.

Determine the mass to the nearest 1 g and the geometrical dimensions to the nearest 0,1 mm. Calculate the green bulk density. If this is not close to that specified by the manufacturer, repeat the procedure at a different pressure. Report the pressure(s) used.

6.3 Taphole clay materials

6.3.1 Preparation of material for shaping

Before the shaping of test pieces, maintain the material for testing at a temperature between 36 °C and 44 °C for 12 h. Where the ramming mix is supplied ready for use, it is normally formed into test pieces as received,

The amount of the material for shaping shall be taken from blocks divided manually into small pieces of maximum dimension 25 mm. The material to be tested shall correspond to a total section of the block taken out in its middle.

6.3.2 Shaping of test pieces

The mix prepared according to the instructions described in 6.3.1 is compacted in one layer with a sand-rammer (see 4.5).

The compaction is made with 10 impacts on each end of the cylindrical mould to obtain a test piece of 50 mm ± 1 mm.

6.4 Dry vibrating mixes

6.4.1 Preparation of material for shaping

Before the shaping of test pieces, maintain the material for testing at a temperature between 18 °C and 22 °C for 24 h.

Using the riffle separator, select the right quantity for the sample. That quantity shall be enough to prepare all the test pieces required. Increase the amount by 50 % to ensure that the quantity is enough to allow correct mixing to eliminate segregation.

For the products containing a resin or an organic hardener, carry out a preliminary test for mould release and cohesion of the test pieces. If necessary, add 1,5 % of thermohardening resin.

Pour the product and resin, if needed, into the mixer pan and mix for 3 min at low intensity (see 4.2)

6.4.2 Shaping of test pieces

The mix prepared according to the instructions described in 6.4.1 is compacted in one layer with a sand-rammer (see 4.5) with appropriate half-core moulds.

Carefully start filling the assembled and lubricated mould to avoid segregating the product using the quantity determined by the preceding tests. The height of the test piece after compaction shall be 50 mm ± 2 mm.

Place the impact disc in the mould on the surface of the product, then place the mould under the sand-rammer.

Strike 20 times continuously.

Put the mould in the oven at 250 °C for 2 h.

Withdraw the mould from the drying oven, cool to 20°C and demould the test piece.

The height of the test piece after drying shall be 50 mm ± 2 mm.

Store the test piece in a desiccator avoiding moisture pick-up, for measurement and heat treatment.

7 Treatment of test pieces

7.1 Castables

7.1.1 Curing

When the shaping of the test pieces is complete, store the mould with the test pieces in air at a relative humidity of at least 90 %, in a humidity cabinet or an airtight plastic bag. The curing temperature is 18 °C to 22 °C. After a storage period of 24 h, withdraw the test pieces from the mould, and store for a further period of 24 h under the same conditions. For material containing magnesite (see ISO 1927-1:2012, 5.2) the curing time shall not exceed 12 h and the test pieces shall be dried immediately.

7.1.2 Drying

After curing according to 7.1.1, dry the test pieces to constant mass in the drying oven (see 4.12) at (110 ± 5) °C for a minimum of 16 h.

Ensure that the test pieces are exposed on all sides to warm air, so that the water vapour can escape unhindered. After drying, cool the test pieces to ambient temperature in a desiccator avoiding moisture pick-up.

For tests on unfired material, carry out the tests immediately after drying and cooling.

For dense and insulating castables which do not develop a hydraulic bond, the manufacturers can specify other curing and drying conditions, which shall be agreed between the parties and given in the test report.

7.2 Ramming mixes and plastics

7.2.1 Alumino-silicate, special and carbon-containing products

7.2.1.1 Curing

After shaping and allowing any setting as stipulated by the manufacturer, withdraw the test pieces from the mould and store for 24 h at 18 °C to 22 °C in static air, on a perforated sheet.

7.2.1.2 Drying

After curing according to 7.2.1.1, dry the test pieces in the drying oven (see 4.12) at (110 ± 5) °C for a minimum of 24 h (to constant mass).

After drying, cool the test pieces to ambient temperature, taking care to protect them from humidity, before firing.

For tests on unfired material, carry out the test immediately after drying and cooling.

7.2.1.3 General

In special cases (such as materials with phosphate bonds), the manufacturer can specify other curing and drying conditions, which shall be agreed between the parties and given in the test report.

7.2.2 Basic products (including carbon-bonded basic ramming mixes)

7.2.2.1 Without tempering

For tests without tempering, carry out the tests immediately after preparing the test pieces, without curing or drying.

7.2.2.2 With tempering

Immediately after shaping, withdraw the test pieces from the mould and temper in a drying oven which is equipped with a gas exhaust. The tempering schedule shall be as shown in Table 3.

Table 3 —Heating rate for tempering

Description	Heating rate in °C/min	
	Resin bonded	Tar bonded
Ambient to 80 °C	2 to 5	-
80 to 140 °C	0,5	-
140 to 200 °C	1 to 2	-
Ambient to 300 °C	-	1 to 2
Soaking time	1 h at (200 ± 10) °C	1 h at (300 ± 10) °C
Cooling	naturally	naturally

8 Firing

8.1 Castables

8.1.1 General

The firing temperature, which shall be agreed between the parties, shall be either a whole multiple of 100 °C (up to 800 °C) or a multiple of 50 °C (above 800 °C).

NOTE One of two procedures can be used for firing, either firing under an oxidizing atmosphere or firing under a reducing atmosphere, chosen in accordance with the manufacturer's instructions.

8.1.2 Firing under oxidizing atmosphere

This type of firing is applicable to both dense and insulating castables.

Remove the test pieces from the drying oven, and protect from moisture pick-up. After cooling, place the test pieces in the furnace horizontally, such that the bottom surface during the preparation period is in contact with the furnace. Protect from direct radiation in an electrically heated furnace or from the flame of the gas burner in a gas-fired furnace. Do not superimpose test pieces one upon another. To allow free circulation of the hot gases, separate the test pieces from each other by a distance of not less than 20 mm, and not nearer than 50 mm to the walls of the furnace. Heat the furnace at the rates given in Table 4.

Table 4 — Heating rate for firing castables

Firing temperature in °C	Description	Heating rate in °C/min		
		Regular castables	Deflocculated castables and chemically bonded castables	Insulating castables
Below or equal to 1250 °C	From ambient to 600 °C	5 to 10	2 to 5	5 to 10
	Above 600 °C to 50 °C below the firing temperature	5 to 10	5 to 10	5 to 10
	The remaining 50 °C	1 to 2	1 to 2	1 to 2
Greater than 1250 °C	Above 600 °C to 1250 °C	5 to 10	5 to 10	5 to 10
	Above 1250 °C to 50 °C below the firing temperature	2 to 5	2 to 5	2 to 5
	The remaining 50 °C	1 to 2	1 to 2	1 to 2

Maintain the temperature for a soak period of 5 h for dense castables and 10 h for insulating castables. For shape D, soak for a period of 3 h for dense castables and 6 h for insulating castables.

NOTE 1 Other soak periods can be agreed between parties and be stated in the test report.

Maintain the temperature within ± 10 °C around the test temperature for the specified period and then switch off the furnace and cool naturally inside the furnace.

NOTE 2 Below 800 °C, the door can be progressively opened in order to cool quickly.

8.1.3 Firing under reducing atmosphere

This type of firing is applicable to dense castables only.

Remove the test pieces from the drying oven, and protect from humidity. After cooling, place the test pieces in silicon carbide boxes (see 4.16) containing metallurgical coke and shut off with a lid sealed with a jointing material. Leave a space of approximately 20 mm between the test pieces, the lid and the bottom of the box. Heat the furnace at the rates given in Table 4.

Maintain the temperature for a soak period of 5 h.

NOTE 1 Other soak periods and heating rates may be agreed between the parties concerned and stated in the test report.

Maintain the temperature inside the box within ± 10 °C around the test temperature for the specified period and then switch off the furnace and cool naturally inside the furnace.

Record the temperature within the box when the furnace has been at the test temperature for 30 min. The temperature inside the box shall be within 20 °C of the test temperature.

If not, then a different box material or dimension may be required for repeat tests.

NOTE 2 From 500 °C, the door can be progressively opened in order to cool quickly.

8.2 Ramming mixes and plastics

8.2.1 Alumino-silicate, special and carbon-containing products

8.2.1.1 General

The firing temperature, which shall be agreed between the parties, shall be either a whole multiple of 100 °C (up to 800 °C) or a multiple of 50 °C (above 800 °C).

NOTE One of two procedures can be used for firing, either firing under an oxidizing atmosphere or firing under a reducing atmosphere, chosen in accordance with instructions given by the manufacturer.

8.2.1.2 Firing under oxidizing atmosphere

Remove the test pieces from the drying oven, and protect from humidity. After cooling, place the test pieces in the furnace horizontally, on those surfaces which were also the bottom during the preparation period, and protect from direct radiation in an electrically heated furnace or from the flame of a gas burner in a gas-fired furnace. Do not superimpose test pieces one upon another. To allow free circulation of the hot gases, separate the test pieces from each other by a distance of not less than 20 mm, and not nearer than 50 mm to the walls of the furnace. Heat the furnace at the rates given in Table 5.

Table 5 — Heating rates for firing ramming materials and plastics

Firing temperature in °C	Description	Heating rate in °C/min
Below or equal to 1250 °C	From ambient to 50 °C below the firing temperature	5 to 10
	The remaining 50 °C	1 to 2
Greater than 1250 °C	From ambient to 1 250 °C	5 to 10
	From 1200 °C to 50 °C below the firing temperature	2 to 5
	The remaining 50 °C	1 to 2

Maintain the temperature for a soak period of 5 h.

For shape D and 50 mm diameter cylinders, soak for a period of 3 h.

NOTE 1 Other soak periods should be agreed between parties and be stated in the test report.

Maintain the temperature within ± 10 °C around the test temperature for the specified period and then switch off the furnace and cool naturally inside the furnace.

NOTE 2 Below 800 °C, the door can be progressively opened in order to cool quickly.

8.2.1.3 Firing under reducing atmosphere

Remove the test pieces from the drying oven, and protect from humidity. After cooling, place the test pieces in silicon carbide boxes (see 4.16) containing metallurgical coke and shut off with a lid sealed with a jointing material.

Leave a space of approximately 20 mm between the test pieces, the lid and the bottom of the box. Heat the furnace at the rates given in Table 5.

Maintain the temperature for a soak period of 5 h.

NOTE 1 Other soak periods should be agreed between parties and be stated in the test report.

Maintain the temperature within ± 10 °C around the test temperature for the specified period and then switch off the furnace and cool naturally inside the furnace.

Record the temperature within the box when the furnace has been at the test temperature for 30 min. The temperature inside the box shall be within 20 °C of the test temperature. If not, then a different box material or dimension may be required for repeat tests.

NOTE 2 Below 800 °C, the door can be progressively opened in order to cool quickly.

8.2.2 Carbon-bonded basic ramming mixes

8.2.2.1 General

The firing temperature, which shall be agreed between the two parties, shall be either a whole multiple of 100 °C (up to 1 000 °C), or a multiple of 50 °C (above 1 000 °C).

8.2.2.2 Drying

If the test pieces are cut or drilled out of larger shapes after tempering, dry them at (110 ± 5) °C for at least 24 h, to constant mass.

8.2.2.3 Carbonization

Carry out the carbonization in accordance with ISO 10060, except for the heating rate, which shall be in accordance with Table 6.

8.2.2.4 Firing under reducing conditions

The firing shall be carried out on the carbonized material cooled to room temperature, placed in the silicon carbide box (see 4.16) and fired according to the firing schedule given in Table 6.

Table 6 — Heating rates for coking and firing

Temperature in °C	Heating rate in °C/min
Ambient to 120	1,5 to 2
From 120 to 1000	3,5 to 3,8
Greater than 1000	2 to 5 (to 50 °C below the firing temperature)
	1 to 2 (the remaining 50 °C)

Maintain the temperature for a soak period of 5 h.

NOTE Other soak periods should be agreed between parties and be stated in the test report.

Maintain the temperature within ± 10 °C around the test temperature for the specified period and then switch off the furnace and cool naturally.

Record the temperature within the box when the furnace has been at the test temperature for 30 min. The temperature inside the box shall be within 20 °C of the test temperature.

If not, then a different box material or dimension may be required for repeat tests.

Open the box at a temperature below 100 °C.

Clean the test pieces from adhering coke particles.

9 Test report

The test report shall include the following information:

a) all information necessary for identification of the sample tested, including the date of production of the batch and the designation of the product tested, in accordance with ISO 1927-1;

b) a reference to this part of ISO 1927, i.e. ISO 1927-5:2012;

c) the preparation method used including;

   1) for castables: the water addition (in %), the duration of mixing, the method of placing, and the size of multi-compartment moulds (if used);

   2) for insulating castables: the dry yield density or bulk density after firing to 400 °C (see 5.2.3);

   3) for ramming materials: the water addition (in %) and the duration of mixing (where appropriate), the method of placing, including the number of forming cycles used and the mass of the rammer, or the forming pressure and the final green bulk density;

d) the name of the test laboratory;

e) any variations in curing conditions from those specified (see Clause 8);

f) firing schedule, including heating rate, maximum temperature and soak time at that temperature;

g) any deviations from the procedure specified;

h) any unusual features (anomalies) observed during the test;

i) the date of the test.

Bibliography

[1] ISO 5016, *Shaped insulating refractory products — Determination of bulk density and true porosity*

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