INTERNATIONAL STANDARD

ISO 20202

First edition
2004-07-15

Carbonaceous materials used in the production of aluminium — Cold and tepid ramming pastes — Preparation of baked test pieces and determination of loss on baking

Produits carbonés utilisés pour la production de l'aluminium — Pâtes de brasquage froides et tièdes — Préparation d'éprouvettes cuites et détermination de la perte à la cuisson

Reference number
ISO 20202:2004(E)

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Published in Switzerland

Foreword

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ISO 20202 was prepared by Technical Committee ISO/TC 47, *Chemistry*, Subcommittee SC 7, *Aluminium oxide, cryolite, aluminium fluoride, sodium fluoride, carbonaceous products for the aluminium industry*.

Carbonaceous materials used in the production of aluminium — Cold and tepid ramming pastes — Preparation of baked test pieces and determination of loss on baking

1 Scope

This International Standard describes a method of baking green test specimens made by compacting carbonaceous ramming pastes. It includes the baking procedure (heating rate and holding time) and the determination of the loss on baking (relative mass loss).

Baked test specimens of ramming paste need to be prepared for the determination of properties after baking, for instance apparent density, crushing strength and porosity.

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 4955, *Heat-resisting steels and alloys*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 14422, *Carbonaceous materials used in the production of aluminium — Cold-ramming pastes — Methods of sampling*

ISO 14427, *Carbonaceous materials used in the production of aluminium — Cold and tepid ramming pastes — Preparation of unbaked test specimens and determination of apparent density after compaction*

ASTM E 220, *Standard Test Method for Calibration of Thermocouples by Comparison Techniques*

3 Principle

Compacted test specimens of carbonaceous ramming paste are baked in a furnace by raising the temperature at a specified rate to 1 000 °C and holding the temperature at this value for 2 h. The mass of each test specimen is measured before and after baking and the loss on baking calculated.

4 Apparatus

4.1 **Balance**, accurate to 0,1 g.

4.2 **Furnace**, e.g. an electrically heated muffle furnace or chamber furnace, with a temperature-control facility enabling it to meet the requirements given in Clause 6.

4.3 Baking container with cover, capable of holding the test specimens and made from heat-resistant steel (e.g. X15CrNiSi2521 steel as specified in ISO 4955). The distance between the test specimens and between the test specimens and the container walls shall not be less than 10 mm. An example of a suitable container is shown in Figure 1.

4.4 Packing material, consisting of electrographite or calcined petroleum coke, maximum particle size 1 mm.

4.5 Temperature-measuring device, capable of determining the temperature to within ± 5 °C up to 1 000 °C, e.g. a type K or S thermocouple calibrated in accordance with ASTM E 220.

5 Sampling

Sample the paste in accordance with ISO 14422. Prepare the test specimens in accordance ISO 14427.

6 Procedure

Determine the mass m_0 of each unbaked test specimen to the nearest 0,1 g.

Insert the test specimens in the baking container (4.3). Surround the test specimens on all sides by at least 10 mm of packing material (4.4). Insert the container, with its cover fitted, into the furnace (4.2). Heat the furnace in accordance with the heating programme given in Table 1.

Table 1 — Heating programme

	Temperature range	Heating rate
Initial rate of heating	Room temperature to 500 °C	(35 ± 5) °C·h^{-1}
Subsequent rate of heating	500 °C to 1 000 °C	(100 ± 10) °C·h^{-1}

Keep the furnace at a final temperature of $(1\,000 \pm 10)$ °C for 2 h to ensure that all the test specimens reach this temperature. Then allow the furnace to cool down to room temperature. Take the test specimens out of the baking container. Remove any packing material which may be sticking to the surface of the test specimens. Determine the mass m_1 of each baked test specimen to 0,1 g.

7 Expression of results

Calculate the relative mass loss during the baking process, called the loss on baking, using the following equation:

$$w = \frac{m_0 - m_1}{m_0} \times 100 \qquad (1)$$

where

w is the loss on baking, expressed as a mass fraction in %;

m_0 is the mass of the green test specimen, in g;

m_1 is the mass of the baked test specimen, in g.

Round the results to the nearest 0,1 % (absolute).

8 Precision (determined in accordance with ISO 5725-2)

Repeatability (r) = 0,9 % (absolute)

Reproducibility (R) = 2,3 % (absolute)

The number of degrees of freedom, i.e. number of laboratories (6) × number of samples (4), was 24.

NOTE The precision will be influenced by variability in the material.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for identification of the sample tested;

c) the temperature used for preparation of the green test specimens;

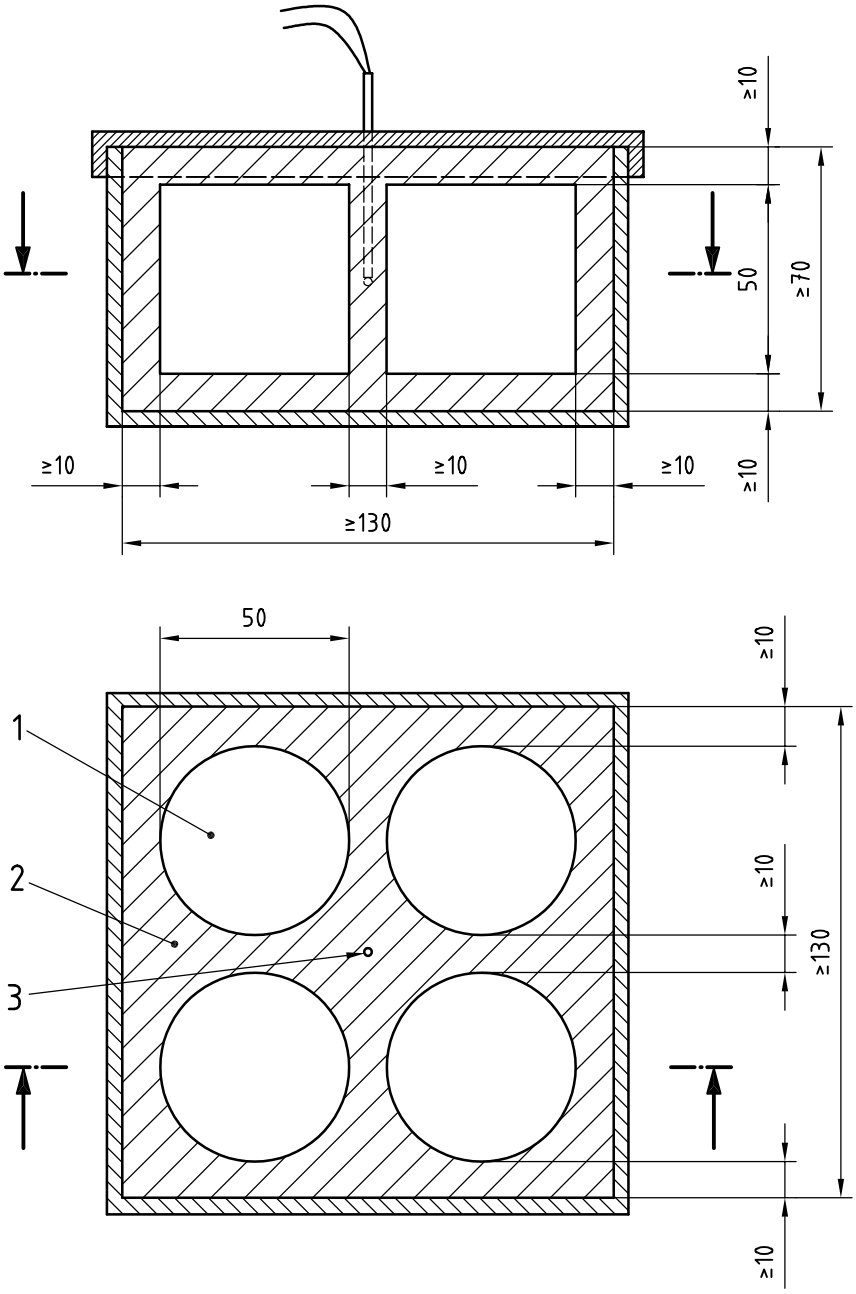
d) the results of the test, i.e. the loss on baking;

e) the date of the test;

f) details of any unusual features noted during the determination;

g) details of any operation not included in this International Standard or regarded as optional.

Dimensions in millimetres



Key

1 test specimen
2 packing material
3 thermocouple

Figure 1 — Example of a baking container for four specimens

ICS 71.100.10

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