INTERNATIONAL STANDARD

ISO 14422

First edition
1999-10-01

Carbonaceous materials used in the production of aluminium — Cold-ramming pastes — Methods of sampling

Produits carbonés utilisés pour la production de l'aluminium — Pâtes de brasquage à froid — Échantillonnage

Reference number
ISO 14422:1999(E)

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Printed in Switzerland

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Foreword

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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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14422 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-ramming pastes — Methods of sampling

1 Scope

This International Standard describes methods and conditions for sampling of cold-ramming pastes used in aluminium manufacture.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitutes provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 6257, *Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Sampling.*

3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 6257 and the following apply.

3.1
cold-ramming paste
mixture containing graded carbonaceous aggregate and bonding materials which harden when heated to form a carbonaceous bond

NOTE Such a mixture needs to be installed at or near ambient temperature by the use of mechanical or pneumatic tools or vibration.

3.2
increment
amount of material taken from a sample unit in a single operation with a sampling device

3.3
test portion
amount of material taken from the laboratory sample and prepared in a manner suitable for use in a particular determination

3.4
sample unit
item
defined quantity of material having a boundary

NOTE 1 The boundary may be physical, for example a container, or temporal, for example a particular time or time interval in the case of a stream of material.

NOTE 2 A number of sample units may be gathered together, for example in a package or box.

NOTE 3 In English, the terms "item", "unit" and "individual" are sometimes used as synonyms for "sample unit". In French, the term "individu" is sometimes used as a synonym for "unité d'échantillonnage".

4 General

The type of sampling equipment, and the preparation and reduction of the samples, which should not alter the properties to be determined, shall be agreed between the contracting parties.

When sampling material in movement, do so during loading or unloading of the consignment or while packaging units are being filled (see 6.4.5).

During sampling, sample division, and the preparation and storage of samples, protect the samples from any factors which may cause changes in the properties to be determined.

If required, subdivide the consignment into individual test lots (for example, if it is clear that the consignment consists of various lots or is to be treated in separate partial quantities).

Distribute the increments over the entire consignment and take them at regular ponderal or temporal intervals which do not vary throughout the duration of one sampling procedure. In addition, divide the consignment by mass and time into as many intervals as there are increments to be taken. Initial sampling is performed at a moment chosen at random within the first interval.

NOTE The term "ponderal" describes fixed intervals of mass.

5 Apparatus

Ensure that sample containers, sampling equipment and any ancillary equipment likely to come into contact with the product being sampled are clean and dry.

5.1 Quartering cross, of metal or other rigid impermeable material, with four blades joined together at the centre at right angles to each other. The height of the blades forming the cross shall be greater than that of the flattened conical pile of material on which it is used (see 6.4.2), and the length of the individual blades shall be greater than the radius of the flattened cone.

5.2 Sampling scoop, with an internal width at least six times the estimated size of the largest particles in the product to be sampled, and capable of containing the required minimum mass of an increment (see Table 1).

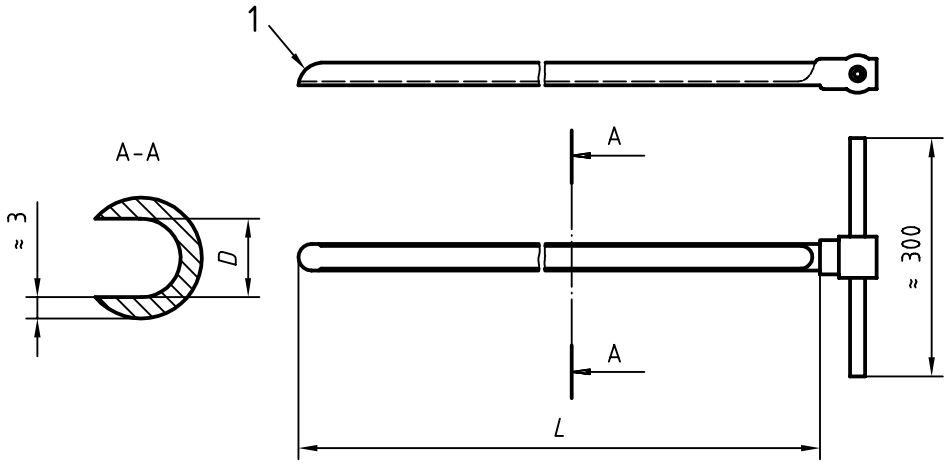
Table 1 — Minimum mass of increment

Maximum grain size mm	**Minimum mass of increment** g
20	2 000 ± 200
10	500 ± 50
3	200 ± 20
1	50 ± 5

5.3 Sampling spear, as illustrated in Figures 1 and 2, with a minimum internal diameter D at least six times the estimated size of the largest particles of the material to be sampled, and capable of containing the required minimum mass of increment (see Table 1). The length L of the spear shall be approximately 10 % greater than the depth of the product to be sampled.

5.4 Sample containers, of sufficient capacity to be nearly filled by the samples, leaving only sufficient space for expansion and to avoid losses during opening, made of a material which will not alter the samples in any way.

Dimensions in millimetres

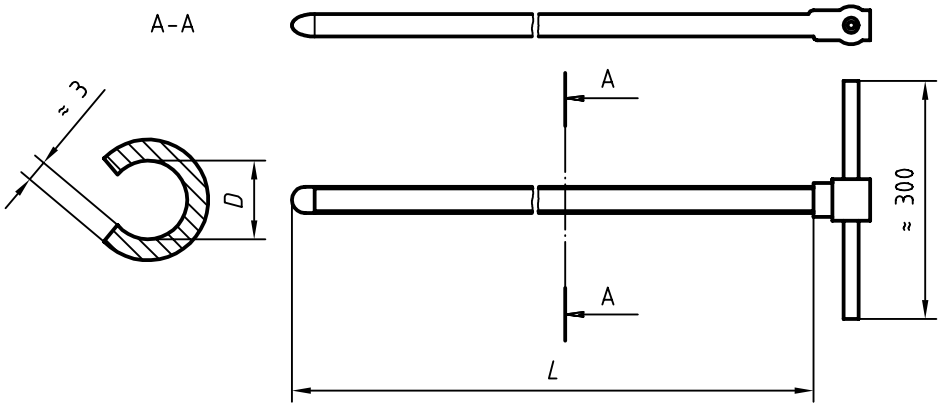


Key

1 Cutting edge (ground)
D Internal diameter (see 5.3)
L Length (see 5.3)

Figure 1 — Open-end sample spear

Dimensions in millimetres



Key

D Internal diameter (see 5.3)
L Length (see 5.3)

Figure 2 — Closed-end sampling spear

6 Procedure

6.1 Mass of increment

Estimate visually the maximum grain size of the product. Using the estimated maximum grain size, determine the minimum mass of the increment from Table 1.

NOTE This procedure avoids systematic errors during sampling.

6.2 Mass of final sample

Determine the minimum mass of the final sample from the total mass of the material to be sampled, by using the values in Table 2. If the value obtained is less than six times the total amount of material required for testing, increase the mass of the final sample to six times that required for testing.

Table 2 — Minimum mass of final sample

Mass of material to be sampled tonnes	**Mass of final sample** kg
< 1	8
1 to 5	12
5 to 10	16
10 to 50	24
50 to 100	32

6.3 Selection of items for sampling

If the sequence of manufacture of the individual items comprising the lot is known, e.g. from the container marks, take the increments so that they are distributed as uniformly as possible throughout the whole sequence. If the manufacturing sequence is not known, take the increments at random from the lot.

6.4 Sampling techniques

6.4.1 Selection of technique

If both the number of individual items and their masses are small, take the required increments by coning and quartering (see 6.4.2). If the individual items are too large to be sampled satisfactorily by coning and quartering, use the flattened-heap method (see 6.4.3) or the sampling-spear technique (see 6.4.4). If the increments are to be taken from the product during manufacture, use the procedure described in 6.4.5.

6.4.2 Coning and quartering

Empty the product from a container representing one item on to a clean, impervious surface and form it into a conical pile by shovelling. Work systematically around the pile, placing shovelfuls on the apex of the cone from every side so that the centre of the cone is not displaced. Flatten the cone by pressure with the back of the shovel, taking care that the resulting pile is uniform in height and diameter and that its centre coincides with the centre of the original cone. Repeat the coning and flattening operations twice.

Divide the pile into quarters along two lines intersecting at right angles, using a quartering cross (5.1). Remove and reject the product in two diagonally opposite quarters and repeat the coning and quartering until the mass of the unrejected product is within the limits for the required mass of the increment (see 6.1). Combine the unrejected

quarters to form the increment. Combine the individual increments (see 6.1 and 6.2) to form the final sample (see 6.5.1).

6.4.3 Flattened-heap technique

Empty the product from a container representing one item on to a clean, impervious surface and mix it thoroughly. Scrape the product into a heap, then flatten the heap. Using a sampling scoop (5.2), take at least 20 approximately equal increments each within the required mass limits (see 6.1). Dig to the bottom of the heap on all occasions and ensure that the increment taken contains product from throughout the depth of the heap. Combine the increments to form the final sample (see 6.5.1).

6.4.4 Sampling-spear technique

Thrust the sampling spear (5.3) at an angle into the product in a container representing one item, with the open side of the spear facing downwards. Give the spear two or three turns then, with the open side uppermost, withdraw it carefully so that it remains filled with the product. Transfer the withdrawn increment to the sample container. Repeat the technique at a number of points uniformly distributed over the accessible surface of the product until the mass of sample required (see 6.1 and 6.2) has been obtained. Combine the individual increments to form the final sample (see 6.5.1).

6.4.5 Sampling from product during manufacture

WARNING — It is essential to perform the following operations with the mixer *switched off and isolated*.

If the mixer is of a type allowing satisfactory access for sampling the whole of the batch, e.g. a pan mill, use a sampling scoop (5.2) to take a number of increments, each within the required mass limits (see 6.1), so that the total mass of the increments is no less than that required for the final sample (see 6.2). Dig through the full depth of the product in taking the increments, so that each increment contains product from throughout the depth of the charge. Combine the increments to form the final sample (see 6.5.1).

6.5 Sample subdivision

6.5.1 Final sample

If the laboratory and reference samples are to be drawn from more than one item, take the individual samples drawn from all the sampled items and mix them together thoroughly before performing any subdivision which may be necessary.

6.5.2 Laboratory and reference samples

The laboratory sample and the reference sample shall each be of at least three times the total mass required for the individual determinations which are to be undertaken (see 6.2). If the mass of the final sample (see 6.5.1) is more than twice the combined requirement for the laboratory and reference samples, subdivide the final sample by coning and quartering in accordance with 6.4.2 until the final operation produces two pairs of opposite quarters with each pair totalling not less than the required mass. Combine one pair of opposite quarters to give the laboratory sample, and combine the other pair to give the reference sample.

6.5.3 Test portions

6.5.3.1 Take test portions for individual determinations by subdividing the laboratory sample by coning and quartering in accordance with 6.4.2. Make any necessary adjustments to the test portion mass of less than 5 % by mass in accordance with 6.5.3.2.

6.5.3.2 Add randomly selected spot portions as necessary from the laboratory sample or subtract randomly selected spot portions from the test sample.

7 Sampling report

The sampling report shall contain the following information:

a) all details necessary for complete identification of the manufacturer and the product type;

b) the date of manufacture of the lot or consignment;

c) the number of containers in the lot or consignment, and their size and individual identifying marks;

d) the number of containers sampled and their individual identifying marks, in the case of material sampled in accordance with clause 6 after delivery;

e) the method of sampling used, including a reference to this International Standard, i.e. ISO 14422:1999, the number of increments taken, and the approximate mass of the laboratory and reference samples;

f) the identifying marks used for the laboratory and reference samples;

g) any unusual features noted during sampling;

h) any operation not included in this International Standard or regarded as optional.

ICS 71.100.10

Price based on 6 pages