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Aluminium oxide primarily used for the production of aluminium — Preparation and storage of test samples

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Foreword

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This document was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Aluminium oxide primarily used for the production of aluminium — Preparation and storage of test samples

1 Scope

This document describes methods for the sample preparation of smelter-grade aluminium oxide. It covers the reduction of a bulk sample between 1 kg and 20 kg to produce a sample that is suitable for chemical and physical analysis. The reduced sample produced will be representative of the initial bulk sample. The bulk sample is considered to be from one source.

The methods are suitable for dry, free-flowing aluminium oxide.

As the moisture content will influence many physical analyses, the method aims to minimize the exposure of the sample to the atmosphere to avoid water absorption.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
increment
quantity of material collected in a single operation of a sample device

3.2
sample preparation
process of preparing the sample for analysis or testing, which may involve air-drying, particle size reduction, mixing and division, and may be performed in several stages

3.3
sample preparation stage
stage in the sample preparation sequence of operations that may consist of sample drying, reduction in particle size or mixing, and which culminates in sample division

Note 1 to entry: The completion of each operation of sample division defines the commencement of the next sample preparation stage. Thus, the number of stages in sample preparation is equal to the number of divisions made.

4 Principle

The bulk sample is mixed using a rotary splitter and the sample reduced to the desired size, then further divided by rotary splitting into appropriately sized portions suitable for the analyses required.

5 Apparatus

5.1 Rotary splitter

Hopper, chute and sample containers should be made of a material which will not affect analytical results. Stainless steel is recommended. For large bulk samples, the sample receivers should have a capacity of up to 2 kg and the splitter should have a minimum of eight of these receivers. The splitter shall be configured so as to minimize loss of fine particles during operation; this is achieved by minimizing the fall distance of the aluminium oxide as it is fed onto the dividing plate and having a good seal between the sample receiver and the dividing plate exit. An example of a rotary splitter is shown in Annex A.

If the proportion of particles passing a 20 micron sieve is to be determined on a split portion, then test work to ensure that no more than 0,2% of less than 20 micron particles (referred to as "fines") are lost over the total sample division process shall be conducted.

5.2 Laboratory top-pan balance

Capable of weighing 4 kg to the nearest 1 g.

5.3 Sample bottles/containers

Of a sturdy plastic construction with an airtight screw-top lid. Sample containers shall be clean and dry. These containers should not have been used for storing any other material.

5.4 Small rotary splitter

Hopper, chute and sample containers should be made of a material which will not affect analytical results. Stainless steel is recommended. The sample receivers should have a capacity of up to 250 g and the splitter should have a minimum of eight of these receivers. If the material hopper discharges onto a chute, mechanical vibration will be required to assist flow of the aluminium oxide along the chute.

5.5 Brush

For brushing out residual aluminium oxide in containers.

5.6 Powder funnel

Suitable for specific mass of aluminium oxide to be transferred into a bottle/container (5.3).

6 Procedure

6.1 Sample integrity

It is important that, as far as is practicable, sample loss is minimized during the sample preparation stages. All equipment should be thoroughly cleaned before use. It is preferable to keep equipment and work areas solely for aluminium oxide preparation to avoid contamination. If a dust extraction or air-conditioning system is in use where splitting is performed, ensure that airflow is not sufficient to disturb the aluminium oxide while it is being processed.

Minimizing the time the sample is exposed to air will reduce the risk of dust and moisture contamination. Work quickly and limit the time the sample is left in the splitter prior to it being placed in sample containers. An increase in moisture will occur during the sample preparation process; this will impact on the material's loss of mass at 300 °C and, to a minor extent, 1 000 °C.

6.2 Mixing sample

This step may be omitted if the increment is homogenous and/or it is essential that loss of fines be minimized. The mixing procedure is as follows:

a) Transfer the sample to the hopper of a rotary splitter. Tap the sample container and brush to remove any dust still adhering to the inside and add to the rest of the sample in the hopper.

b) Operate the splitter as per the manufacturer's instructions. Turn on the splitter rotator and vibratory feeder (if fitted) and regulate aluminium oxide feed to form a steady flow to the receiving vessels. The carousel rotation and aluminium oxide flow should not be so fast as to generate dust and consequent loss of material.

c) When the sample has finished falling into the receivers, turn off the rotator and vibrator (if fitted). Tap and brush each receiver to remove any adhering dust and add to the hopper.

d) Return the split samples back into the splitter's hopper. Split the sample again [repeat 6.2 a) to c)]. This step will conclude the sample blending.

6.3 Division of sample

The sample division procedure is as follows:

a) Weigh one of the receivers and sample. Calculate the amount of sample in the receiver by deducting the container weight. Recombine an appropriate number of receivers into the hopper to produce a sample mass suitable for the size of the containers and analysis requirements.

NOTE An example of calculation for sample division is shown in Annex B.

b) Split the sample [follow 6.2 a) to c)].

c) Quantitatively transfer the contents of a receiver or several receivers to a sample container. Tap the receiver to remove all of the aluminium oxide. A funnel should be used in the transfer process to avoid sample spillage. If any loss of sample occurs between transferring the sample to the sample bottle, then this portion should be discarded. Repeat the transfer with the remaining segment containers. An appropriately sized sample at this stage would be 500 g to 2 000 g. Container lids shall be firmly fixed in place. Containers shall not be filled more than three quarters full to enable easy mixing of their contents by tumbling.

d) Store excess aluminium oxide in dry airtight containers if required for future reference.

6.4 Splitting of sample for analysis

Further reduction of the sample can be accomplished using the smaller rotary splitter (6.3). Tumble the aluminium oxide in its container to ensure good mixing and then quantitatively transfer it to the hopper of the splitter. Turn on the splitter rotator and vibratory feeder to split the sample among the receivers. It may be necessary to recombine a number of the receivers and split it again to obtain the desired mass; for example, 35 g to 50 g for sieve-based particle size analysis or 50 g to 60 g for attrition index. It is extremely important that all aluminium oxide is tapped and brushed from the receivers during the splitting and transferred to storage containers.

6.5 Identification of sample

Label each container with its identification, the date on which the sample was taken and any other details, if required.

6.6 Storage and use

Store sample containers at room temperature in a clean, dry environment.

Containers should be tumbled to ensure good mixing before an analytical portion is extracted.

Annex A
(informative)

Rotary splitter

Figure A.1 — Large rotary riffle splitter used for splitting 20 kg increments