TECHNICAL SPECIFICATION

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Carbonaceous materials used in the production of aluminium — Cold-ramming pastes —Determination of effective binder content and aggregate content by extraction with quinoline, and determination of aggregate size distribution

Produits carbonés utilisés pour la production de l'aluminium — Pâtes de brasquage à froid — Détermination de la teneur effective en liant et en aggrégats par extraction à la quinoléine, et détermination de la granulométrie

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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 3.

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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed every three years with a view to deciding whether it can be transformed into an International Standard.

Attention is drawn to the possibility that some of the elements of ISO/TS 14423 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 14423 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.*

Introduction

In order to determine the particle-size distribution of the aggregate used in cold-ramming pastes, it is necessary to obtain the aggregate in a binder-free state. The most practicable way to achieve this is by extraction of the binder by a suitable solvent, and the most effective common solvent for coal-tar and bituminous binders is quinoline. Most coal tars are not totally soluble in quinoline, but have a small content of insoluble matter which principally comprises infusible solid carbonaceous particles smaller than approximately 50 μm. This insoluble matter is effectively part of the solid aggregate of the cold-ramming paste and is measured as such by this method. The effective binder is defined as that proportion of the product which is soluble in quinoline.

Although quinoline is the most effective common solvent for the present purpose, it has the disadvantage of being expensive and of having a high boiling point which makes the removal of the last traces from the extracted residue difficult by normal drying processes. However, when the most complete separation of the binder from the aggregate is required, e.g. for referee purposes, quinoline is the solvent of choice, and the method given in this Technical Specification should be employed.

NOTE The procedure described in this Technical Specification utilizes dichloromethane, but only to remove residual traces of quinoline solvent from filters prior to oven drying.

Carbonaceous materials used in the production of aluminium — Cold-ramming pastes — Determination of effective binder content and aggregate content by extraction with quinoline, and determination of aggregate size distribution

1 Scope

This Technical Specification describes a method for determining the content and particle-size distribution of the solid aggregate component, and also for determining the effective binder content, of cold-ramming pastes used in aluminium manufacture.

The method is applicable to cold-ramming pastes made with coal-tar or bituminous binders but not applicable to resin-based binders.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this Technical Specification. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this Technical Specification 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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 2591-1, *Test sieving — Part 1: Methods using test sieves of woven wire cloth and perforated metal plate.*

ISO 14422:1999, *Carbonaceous materials used in the production of aluminium — Cold-ramming pastes — Sampling.*

3 Principle

A test portion of the cold-ramming paste is digested under reflux with quinoline to dissolve the binder. The digestion mixture is poured on to a fine-aperture sieve, upon which the majority of the aggregate thus retained is washed free of residual binder with further portions of quinoline and then dried and weighed. The mixture and washings passing through the sieve are filtered through a filter pad and the very fine particles of aggregate thus recovered are likewise washed, dried and weighed.

The content of the aggregate is determined from the total mass of the particles retained on the sieve and the filter pad, and the content of the quinoline-soluble matter is calculated by difference.

The particle-size distribution of the solids retained on the fine-aperture sieve is determined by test sieving.

4 Terms and definitions

For the purposes of this Technical Specification, the terms and definitions given in ISO 14422 and the following apply.

4.1
effective binder content
proportion of the product soluble in quinoline, expressed as a percentage by mass

5 Reagents

WARNING — Refer to the reagent supplier's health and safety data sheets for the precautions which are to be observed for the safe use of quinoline and dichloromethane.

5.1 Quinoline, of purity at least 98 % by mass, freshly distilled, boiling between 235 °C and 237 °C at 101,36 kPa.

5.2 Dichloromethane (methylene dichloride), of purity at least 98 % by mass.

6 Apparatus

Ordinary laboratory apparatus and the following:

6.1 Distillation flask, round-bottomed, of borosilicate glass, capacity 2 litres, having a ground-glass socket.

6.2 Reflux air condenser, of effective length 550 mm, having a ground-glass cone by means of which it may be fitted to the socket of the distillation flask (6.1).

6.3 Electric heating mantle, capable of maintaining gentle boiling of the contents of the distillation flask (6.1) whilst under reflux.

6.4 Nest of test sieves, 200 mm in diameter, conforming to ISO 565, of stainless-steel wire cloth, with nominal apertures of between 53 μm and 5 mm inclusive, with lid and receiver.

NOTE Other nominal apertures recommended for inclusion are: 2 mm and 1 mm, 500 μm, 250 μm and 75 μm.

6.5 Receiver test sieve, 200 mm in diameter, conforming to the requirements of ISO 565, of stainless-steel wire cloth, of 53 μm nominal aperture. Prepare by heating in the electric oven (6.7) for 1 h at a temperature of 110 °C ± 5 °C then allow to cool to ambient temperature in a desiccator and store in the desiccator until required for use.

6.6 Dish, of glass or porcelain, capable of fitting below the receiver test sieve (6.5) and collecting all the liquids which pass through.

6.7 Electric oven, of size sufficient to contain the receiver test sieve (6.5) and capable of being maintained at 110 °C ± 5 °C.

6.8 Funnel, Hartley 3-piece or equivalent, having a disc diameter of 120 mm.

6.9 Glass-fibre filter pads, of diameter 120 mm, with a retention of at least 98 % by mass for particles of size 1,2 μm. Dry by heating for 1 h at 110 °C ± 5 °C then store in a desiccator until required for use.

6.10 Mechanical sieve shaker, capable of accommodating the nest of sieves (6.4) with the lid and receiver, as described in ISO 2591-1.

7 Sampling

Prepare a representative laboratory sample of cold-ramming paste in accordance with ISO 14422.

8 Preparation of test portion

Take 100 g ± 0,05 g from the laboratory sample (see clause 7) in accordance with 6.5.3 of ISO 14422:1999.

9 Procedure

9.1 Transfer the test portion quantitatively to the distillation flask (6.1). Add 1 litre of quinoline (5.1) and swirl the contents of the flask to mix the test portion with the quinoline. Place the flask in the electric heating mantle (6.3) and fit the reflux air condenser (6.2) to the flask. Bring the contents of the flask to a steady boil and continue boiling under reflux for 1 h.

Switch off the electric heating mantle and allow the flask and its contents to cool to 50 °C ± 5 °C. Weigh the receiver test sieve (6.5) to the nearest 0,01 g then place it over the dish (6.6). Carefully transfer the hot contents of the flask as completely as possible to the sieve. Rinse the flask using successive 20 ml portions of quinoline at 50 °C ± 5 °C until all the visible solids have been transferred to the sieve. Wash the contents of the sieve with quinoline at 50 °C ± 5 °C until the washings passing through the sieve show no additional colour due to dissolved binder. Allow the quinoline to drain from the sieve contents then separate the test sieve and the dish.

9.2 Transfer the contents of the dish quantitatively to a container to await recovery of the solids by filtration.

9.3 Replace the receiver test sieve over the dish and wash the aggregate on the sieve with 10 successive 100 ml portions of dichloromethane (5.2), allowing each portion of the solvent to drain off before adding the next. After the final wash, leave the sieve and its contents to drain for 1 h and discard the washings. Place the receiver test sieve with its contents in the oven (6.7) maintained at 110 °C ± 5 °C for 1 h, taking care that no boiling of residual dichloromethane occurs, leading to loss of solids.

Remove the receiver test sieve with its contents and allow to cool to ambient temperature in a desiccator. Weigh the receiver test sieve and its contents to the nearest 0,01 g. Repeat the operations of heating, cooling and weighing until the difference between two consecutive weighings does not exceed 0,01 g.

9.4 Weigh a dried filter pad (6.9) to the nearest 0,01 g. Assemble the funnel (6.8) with the filter pad and fit the assembly into the neck of a filter flask. Moisten the filter pad with a little quinoline at 50 °C ± 5 °C and apply suction. When the quinoline has passed through the filter, start filtering the retained digestion liquid and its associated quinoline washings (see 9.2).

When the filtration is complete, rinse the container with successive 20 ml portions of quinoline at 50 °C ± 5 °C to transfer any residual solids to the filter. When all the visible solids have been transferred, continue to wash the solids on the filter with successive 20 ml portions of dichloromethane, allowing each portion to drain off before adding the next. Release the suction, remove the filter pad with the residue and transfer any solid which remains on the walls of the funnel to the pad. Place the pad in the oven maintained at 110 °C ± 5 °C for 1 h.

Remove the pad from the oven and allow it to cool to ambient temperature in a desiccator. Weigh the pad bearing the residue to the nearest 0,01 g. Repeat the operations of heating, cooling and weighing until the difference between two consecutive weighings does not exceed 0,01 g.

9.5 Recover the dried aggregate from the receiver test sieve (see 9.3) by inverting the sieve over a suitably sized piece of clean smooth-surface paper. Remove any particles trapped in the mesh by tapping and gently brushing the underside of the sieve cloth.

9.6 Sieve the recovered aggregate using the nest of test sieves (6.4) and the mechanical sieve shaker (6.10) in accordance with ISO 2591-1 for dry sieving.