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Carbonaceous materials used in the production of aluminium — Determination of baking level expressed by equivalent temperature

Produits carbonés utilisés pour la production de l'aluminium — Détermination du niveau de cuisson par estimation de la température de cuisson équivalente

Reference number
ISO 17499:2006(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 17499 was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

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Introduction

The equivalent temperature is used to express the baking level of a single anode or cathode, or the overall baking level and distribution of a section of any type of baking furnace constructed for baking carbon anodes or cathodes for the production of aluminium.

The equivalent temperature is also useful for monitoring and comparing the baking level of laboratory test samples.

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Carbonaceous materials used in the production of aluminium — Determination of baking level expressed by equivalent temperature

1 Scope

This International Standard covers Carbonaceous materials used in the production of aluminium — Determination of baking level expressed by equivalent temperature.

This International Standard specifies the determination of the equivalent temperature of one anode or cathode in a baking furnace, and the calculation of the overall baking level in a section in the baking furnace.

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 20203, *Carbonaceous materials used in the production of aluminium — Calcined coke — Determination of crystallite size of calcined petroleum coke by X-ray diffraction*

3 Terms and definitions

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

3.1
equivalent temperature
baking level of an anode or cathode measured by the heat treatment of a reference coke attached to the anode or cathode, and quantified in equivalent degrees, °E, determined from the mean crystallite height, L_c, of the reference coke using a calibration curve

NOTE The calibration curve is derived by subjecting a series of separate samples of the reference coke to a 2 h heat treatment at different hold temperatures, in degrees Celsius, and measuring the mean crystallite height, L_c The equivalent temperature, T_{eq}, is numerically equal to the temperatures, in degrees Celsius, plotted on the calibration curve.

4 Principle

The equivalent temperature of an anode or cathode is determined by placing a graphite container with a test portion of the reference green coke in a stub-hole or other suitable depression prior to loading the anode or cathode in the baking furnace.

Following calcination in the baking furnace, the graphite container is unloaded, and the reference coke is recovered and analyzed with regard to the mean crystallite height, L_c, according to ISO 20203.

NOTE ISO 20203 uses the term crystallite size or thickness, which is the same as the crystallite height.

The pre-determined calibration curve linking equivalent temperature with the crystallite height is used to determine the equivalent temperature from the measured L_c-value.

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5 Reference coke

5.1 General

A calibration curve is unique for the specific green, single-source petrol coke batch used as the reference coke.

5.2 Selection and preparation

Store a sufficient amount of dry, green, single-source petrol coke to be the reference coke and give it a batch reference number. The coke should be – 5 mm, and it should be in grains and not powder for ease of recovery from the graphite container. If required, mix, preferably by splitting and recombining.

NOTE Using portions of 20 g, an expected production of 400 anodes/day and a measurement frequency of 2 % daily gives an annual routine consumption of 60 kg. A complete mapping of the baking level in a 168-anode section will consume 3,4 kg.

6 Calibration curve

Determine the calibration curve for the reference coke by taking separate samples of the reference coke, subjecting them to a series of heat treatments with a hold temperature, T_h, and analyzing them with regard to mean crystallite height, L_c using the X-ray diffractometer (7.2), in accordance with ISO 20203.

Care should be taken to have a sufficient number of heat treatments, at least 6, and at least 2 in the upper range due to the effect of the curvature.

Each heat treatment is performed by rapidly heating the reference coke to the hold temperature, T_h, keeping it at that temperature for a constant soaking time of 2 h and then immediately quenching.

According to the definition, the equivalent temperature is numerically equal to the hold temperature, $T_{eq} = T_h$, thus we have a series of (L_c, T_{eq}) data points. A typical series is shown in Figure 1.

Experience indicates that a 3rd-order polynomial as shown in Equation 1 gives the best calibration-curve fit. The resulting expression is of the form

$$T_{eq} = a \cdot (L_c)^3 + b \cdot (L_c)^2 + c \cdot L_c + d \quad (1)$$

where

T_{eq} is the equivalent temperature;

a, b, c, d are coefficients of the 3rd-order equation;

L_c is the mean crystallite height.

NOTE 1 The calibration curve of a new batch of reference coke can be determined from an old reference coke by heat-treating a number of test portions in parallel, which gives equivalent temperatures for the new batch. By measuring the L_c of the new test portions, a series of (L_c, T_{eq}) data points is obtained for the new batch, and the new calibration curve can be determined.

NOTE 2 The green coke calcination is sensitive to time as well as temperature. Figure 2 illustrates how the crystallite height and equivalent temperature increased with time at the same calcination temperature, underlining the importance of keeping the same 2 h hold time during the heat treatments.

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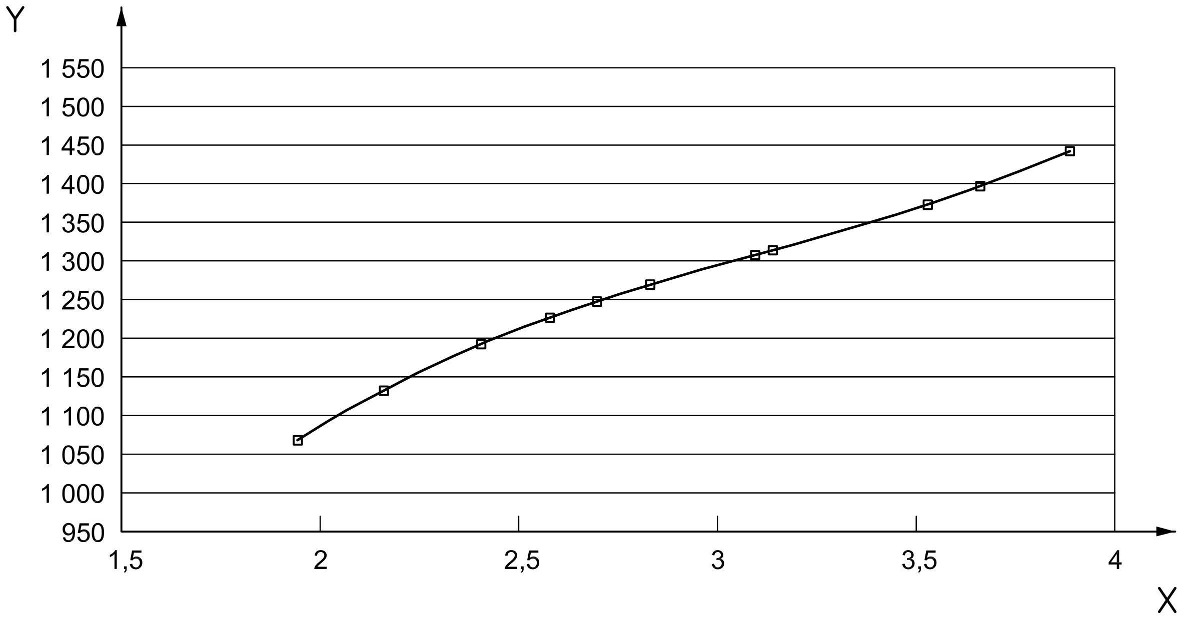
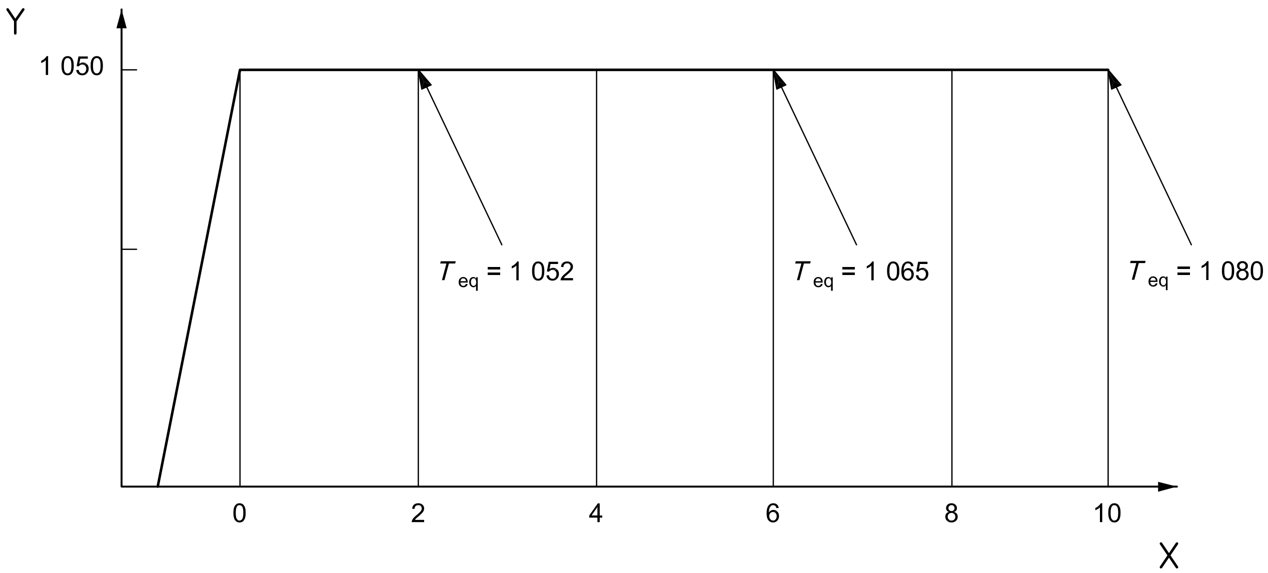


Key

X mean crystallite height, L_c (nm)

Y temperature, T_{eq} (°E)

Figure 1 — Calibration curve for the reference coke from 11 heat treatment (L_c, T_{eq}) data points. The curve fit is a 3rd order polynomial as shown in Equation 1



Key

X soaking time (h)

Y temperature (°C)

Figure 2 — Effect of longer soaking times relative to the equivalent temperature

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7 Apparatus

7.1 Small furnace, to provide heat treatments for a calibration curve for the green coke, that is able to heat (15 to 30) g of coke to (1 000 to 1 500) °C in (6 to 8) min and uses inert gas.

7.2 X-ray diffractometer, for L_c measurement according to ISO 20203.

7.3 Cylindrical graphite container with a graphite lid, with a small hole in the lid to enable the gas outlet from the green coke; typically with an outer diameter of 40 mm, inner diameter 20 mm and length 90 mm; with good treatment, graphite holders can last about 10 rounds.

7.4 Swing mill.

8 Test procedure

8.1 Equivalent temperature measurement

Place a suitably large test portion of the reference coke (Clause 5) in the cylindrical graphite container (7.3). A typical test portion is 15 g. Take care, each time the cylindrical graphite container is used, to ensure that the small hole in the lid is open before loading the test portion.

Place the graphite container in a stub-hole, or other suitable depression in the green anode or cathode, prior to loading it into the baking furnace. Fix the container in place with ceramic fibre or a similar suitable material.

Be aware that, when comparing baking levels in different bake furnaces, the position of the reference coke test portion on the anode relative to the flue wall is important and will influence the result. If the anodes are stacked with the reference coke samples towards the flue walls, a higher baking level will be observed than if the reference coke samples are in the middle of the pit.

Recover the graphite container after baking.

Remove the calcined reference coke test portion from the graphite container. Open the lid by giving a light tap if necessary. Remove the loose test portion, taking care to avoid contamination with graphite: Do not scrape the test portion from the walls of the graphite container, as this can cause graphite contamination. This is important due to the much higher L_c value of graphite.

Finely grind the recovered test portion in the swing mill (7.4) to a fineness suitable for the crystallite height, L_c, analysis using the X-ray diffractometer (7.2), in accordance with ISO 20203.

To obtain the equivalent temperature, the L_c-value from the XRD analysis is used with the calibration Equation (1) to determine the equivalent temperature.

8.2 Equivalent temperature of a section

To obtain the equivalent temperature distribution of a section in a baking furnace, load all or every second anode or cathode in the section with reference coke test portions. After the section has completed the cycle, all test portions are analyzed to obtain the L_c and equivalent temperature values.

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9 Expression of results

9.1 Equivalent temperature of a single anode or cathode

First the difference is calculated, with 2 parallels expressed as T_{eq} in equivalent degrees (°E). If the difference is within 10 °E, the equivalent temperature is the average of the two results. Report the average and the two results. If the difference exceeds 10 °E, 2 new parallels are measured and the new difference calculated. If the new difference is below 10 °E, the equivalent temperature is the average of the two new results, and the average and the 4 parallels are reported. If the new difference exceeds 10 °E, the equivalent temperature is the average of all 4 parallels; the average and the 4 parallels are reported.

9.2 Equivalent temperature of a section

The result is given as the average equivalent temperature for all test portions in the section with the standard deviation e.g. (1 200 $\pm$ 35) °E.

10 Test report

The test report shall include:

a) an identification of the sample (e.g. anode position, anode identification, date on which the anode was unloaded);

b) a reference to the method used, i.e. this International Standard;

c) the results;

d) the date on which the test was carried out;

e) any changes from the standard procedure.

11 Precision

The precision figures are from a ISO round robin carried out in 2003, with participants from 10 laboratories, which determined the equivalent temperature of 3 parallels of 10 samples, 5 samples from each of 2 reference cokes. The precision was:

— Repeatability: $r = 9$ °E

— Reproducibility (between-laboratories): $R = 14$ °E

in the range 1 050 °E to 1 400 °E.

The precision figures were independent of the measured equivalent temperature values.

The precision figures are 95 % confidence interval half-widths. Examples of use are as follows:

Repeatability: Given a number of anodes, all with equivalent temperature 1 200 °E, if the reference coke test portions are measured by the same operator in the same laboratory, the repeatability expresses that 95 out of 100 measurements will be within the range [1 191 to 1 209] °E.

Reproducibility: Given a number of anodes, all with equivalent temperature 1 200 °E, if the reference coke test portions are measured by different laboratories, the reproducibility expresses that 95 out of 100 measurements will be within the range [1 186 to 1 214] °E.

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