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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

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 17499:2006), which has been technically revised.

The main changes are as follows:

— technical description errors have been corrected in 3.2, 5.1, 5.2 and 7.3;

— technical details relating to procedure have been clarified in Clause 4 and 8.1;

— the precision statement text has been corrected and reformulated (without changing the precision values) in Clause 10;

— the description of the graphite crucible with a drawing has been moved from the appendix into the body text as 7.3;

— some editorial corrections have been made.

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.

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 anodes or cathodes and other samples.

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

1 Scope

This document 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 a baking furnace.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

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

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

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

3.1
equivalent temperature
baking level of an anode or cathode measured by the heat treatment of a reference coke attached to or placed with 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 1 to entry: The calibration curve is derived by subjecting a series of separate samples of the green 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.

3.2
reference green coke
raw unprocessed petroleum coke from a refinery coker unit

Note 1 to entry: For use as a reference coke, the green coke should be a single source, low sulfur, anode grade coke, preferably with a sulfur mass fraction of <2,0 % and dried at 110 °C prior to use.

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 the baking of the anode or cathode in the baking furnace, the graphite container is collected, and the now-calcined 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 predetermined calibration curve linking equivalent temperature with the crystallite height is used to determine the equivalent temperature from the measured L_c value.

5 Reference coke

5.1 General

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

5.2 Selection and preparation

Store a sufficient amount of dry, green, single-source petroleum coke to be the reference coke and give it a batch reference number. The coke should be −4 mm and should be in grains and not powder for ease of recovery from the graphite container. If required, mix well, preferably by splitting and recombining.

NOTE Using portions of 12 g, an expected production of 400 anodes/day and a measurement frequency of 2 % daily gives an annual routine consumption of 40 kg. A complete mapping of the baking level at each anode position in a 168-anode section will consume 2,2 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 six, including at least two in the upper range due to the effect of the curvature.

Perform the heat treatment by rapidly heating the reference coke to the hold temperature, T_h, keeping it at the temperature for a constant soaking time of 2 h and then immediately quenching (see Figure 2).

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

Experience indicates that a third-order polynomial, as shown in Formula (1), gives the best calibration-curve fit.

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

where

T_{eq} is the equivalent temperature;

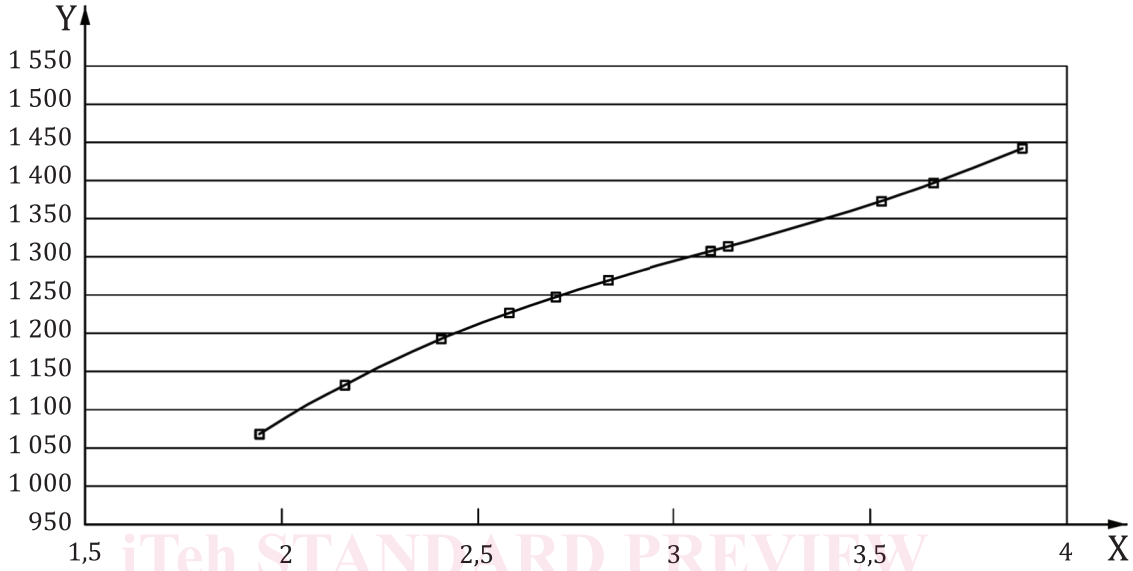
a, b, c, d are coefficients of the third-order equation;

L_c is the mean crystallite height.

The calibration curve of a new batch of reference coke from the same coke source or from a new source can be determined from a calibrated reference green coke by heat-treating a number of test portions of each coke in parallel. The calibrated coke yields the equivalent temperatures and, by measuring the L_c

of the new coke test portions, a series of (L_c, T_{eq}) data points are obtained for the new coke and the new calibration curve can be determined. It is critical to obtain data for a range of heat treatments; the new coke calibration curve should cover baking from 1 050 °E to 1 400 °E.

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



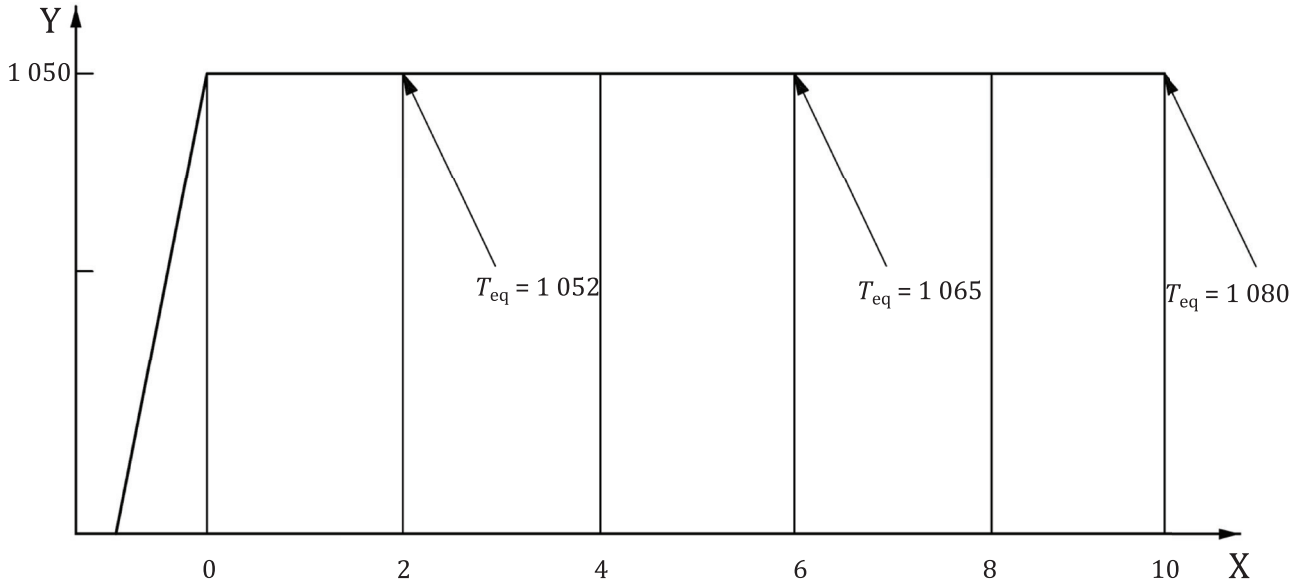
Key

X mean crystallite height, L_c (nm)

Y equivalent temperature, T_{eq} (°E)

NOTE The curve fit is a third-order polynomial, as shown in Formula (1).

Figure 1 — Calibration curve for the reference coke from 11 heat treatment (L_c, T_{eq}) datapoints



Key

X soaking time (h)

Y temperature (°C)

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

7 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

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. 1 400 °C is sufficent.

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, an inner diameter of 20 mm and a length of 90 mm. With good treatment, graphite holders can last ten rounds or more.

NOTE Any supplier of quality machined graphite items should be able to supply the crucible as drawn. The crucible displayed can be replaced wit crucibles with similar properties including the coned lid with the hole.

For an example of a graphite crucible with lid and body, see Figure 3. Lid plug measures A1 and A2 are adjusted so the lid plug can be pressed tight with a slight cone on the plug to fix the lid to the crucible. The lower edge of the upper part of the lid shall have a 2 mm to 3 mm space above the crucible when tightened.