# INTERNATIONAL STANDARD

# ISO 17499

First edition
2006-02-01

## 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)

**PDF disclaimer**

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.





ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

# Contents

Page

Foreword ... iv
Introduction ... v
1 Scope ... 1
2 Normative references ... 1
3 Terms and definitions ... 1
4 Principle ... 1
5 Reference coke ... 2
6 Calibration curve ... 2
7 Apparatus ... 4
8 Test procedure ... 4
9 Expression of results ... 5
10 Test report ... 5
11 Precision ... 5

## 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 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*.



## 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.

# 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.

# 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;

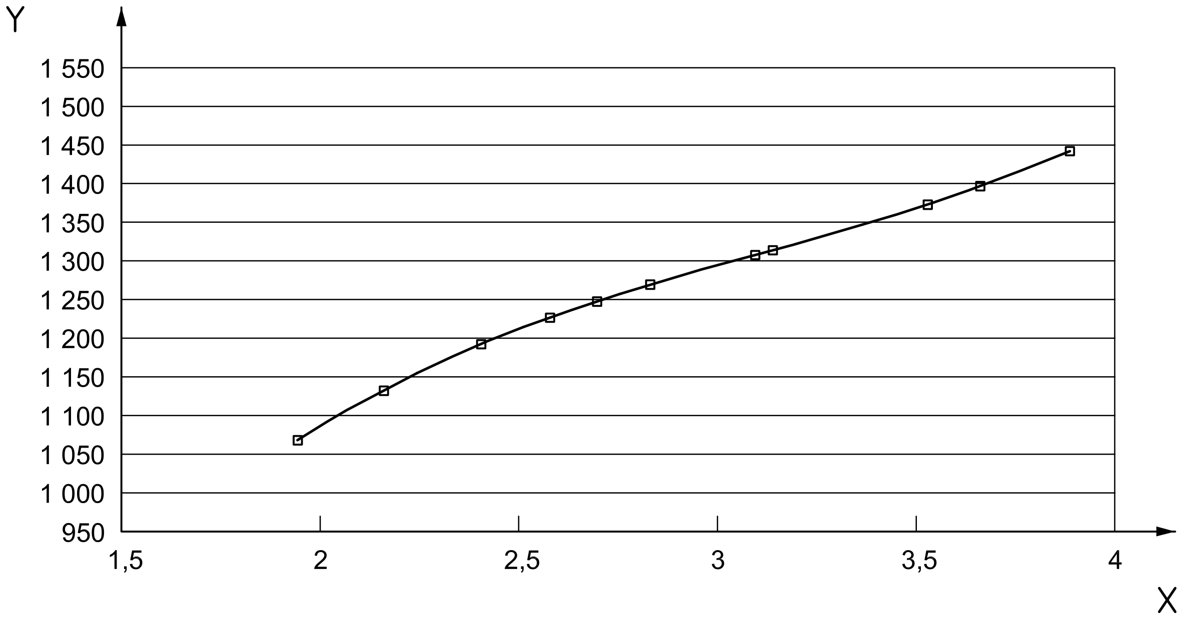
$a, b, c, d$ are coefficients of the 3rd-order equation;

$L_c$ 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.




**Key**

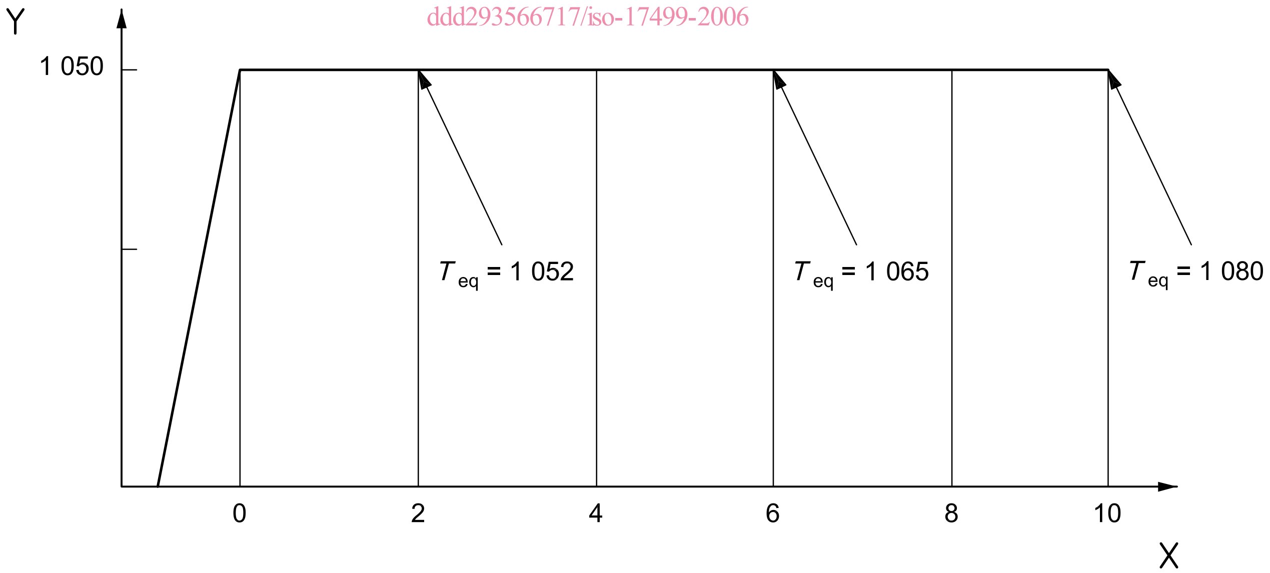
X mean crystallite height, $L_c$ (nm)

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**