

BS ISO 16592:2012



BSI Standards Publication

# Microbeam analysis — Electron probe microanalysis — Guidelines for determining the carbon content of steels using a calibration curve method

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**National foreword**

This British Standard is the UK implementation of ISO 16592:2012. It supersedes BS ISO 16592:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/9, Microbeam analysis.

A list of organizations represented on this committee can be obtained on request to its secretary.

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**Microbeam analysis — Electron  
probe microanalysis — Guidelines for  
determining the carbon content of steels  
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*Analyse par microfaisceaux — Analyse par microsonde électronique  
(microsonde de Castaing) — Lignes directrices pour le dosage du  
carbone dans les aciers par la droite d'étalonnage*





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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 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 16592 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 2, *Electron probe microanalysis*.

This second edition cancels and replaces the first edition (ISO 16592:2006), of which it constitutes a minor revision involving the following changes:

- instructions concerning the establishment of a conductive path from the specimen to ground to prevent charging and instructions concerning the minimization of outgassing of the specimen-mounting medium have been added to Subclause 2.3.2;
- in Subclause 2.3.3, the meaning of carbon-free ultrasonic cleaning has been clarified;
- the second sentence in Subclause 2.4.2 has been reworded to make it clear that the requirements apply to both the reference material and the unknown specimen.

# Microbeam analysis — Electron probe microanalysis — Guidelines for determining the carbon content of steels using a calibration curve method

## 1 Scope

This International Standard gives guidance on a method for the determination of the carbon content in steels containing other alloying elements (less than 1 % to 2 % by mass) using the calibration curve method. It specifies the sample preparation, X-ray detection, establishment of the calibration curve and the procedure for the determination of the uncertainty of the measured carbon content. It is applicable to steels containing a mass fraction of carbon of less than 1,0 %. The method is not applicable to steels with higher carbon contents, which could significantly affect the accuracy of the analysis results.

This International Standard applies to analyses performed using normal beam incidence and wavelength-dispersive X-ray spectrometry; it is not designed to be used for energy-dispersive X-ray spectrometry.

## 2 Procedure

### 2.1 General

In order to determine the carbon content in steels using a calibration curve, suitable reference materials should be prepared. For accurate analysis, extreme care should be used to prevent carbon contamination which would otherwise increase the apparent carbon content of the specimen.

The measurement of C  $K\alpha$  intensity should be carried out using the same procedures for the specimen and the reference materials; that is, specimen preparation, beam energy, beam current, beam diameter, point counting mode, step between points in case of line analysis, and also the method of background subtraction.

### 2.2 Reference materials

To establish the calibration curve to determine the carbon content, a suitable reference material or set of reference materials should be used. Examples of reference materials are as follows:

- Fe-C solid-solution reference materials which are manufactured by quenching from the austenite region at high temperature; these reference materials should be homogeneous and contain different carbon concentrations;
- Fe-C compound  $\text{Fe}_3\text{C}$ <sup>[1]</sup>.

Reference materials with a different C  $K\alpha$  peak shape compared to the unknown materials should not be used because the use of these reference materials causes a lowering of the quantitative accuracy.

### 2.3 Specimen preparation

#### 2.3.1 General

The presence of carbon and/or its compounds as contamination on the specimen surface as a result of specimen preparation significantly affects the accuracy of carbon analysis. Extreme care should be used to prevent this contamination. The specimen preparation (mounting, grinding and polishing) procedures should be the same for both the reference material and the unknown material.