

Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy

ICS 71.040.50

Confirmed February 2009

National foreword

This British Standard is the UK implementation of ISO 14594:2003, incorporating corrigendum February 2009.

The start and finish of text introduced or altered by corrigendum is indicated in the text by tags. Text altered by ISO corrigendum February 2009 is indicated in the text by AC1 AC1.

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.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 11 August 2004

© BSI 2009

ISBN 978 0 580 67362 7

Amendments/corrigenda issued since publication

Date	Comments
31 December 2009	Implementation of ISO corrigendum February 2009

INTERNATIONAL STANDARD

ISO
14594

First edition
2003-08-01

Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy

*Analyse par microfaisceaux — Microanalyse par sonde à électrons —
Lignes directrices pour la détermination des paramètres expérimentaux
pour la spectroscopie à dispersion de longueurs d'onde*



Contents

Page

Foreword.....	iv
1 Scope.....	1
2 Normative references	1
3 Terms and definitions.....	1
4 Abbreviated terms.....	2
5 Experimental parameters	2
5.1 General	2
5.2 Parameters related to the primary beam	2
5.3 Parameters related to wavelength dispersive (WD) spectrometers	3
5.4 Parameters related to the sample	4
6 Procedures and measurements	5
6.1 General	5
6.2 Beam current	5
6.3 Parameters related to measured peaks	6
6.4 Parameters related to the sample	8
7 Test report.....	9
Annex A (informative) Methods of estimating analysis area	10
Annex B (informative) Methods of estimating analysis depth	12
Annex C (informative) Method of estimating X-ray analysis volume by applying the Monte Carlo (MC) simulation	13
Bibliography	17

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 14594 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 2, *Electron probe microanalysis*.

Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy

1 Scope

This International Standard gives the general guidelines for the determination of experimental parameters relating to the primary beam, the wavelength spectrometer and the sample that need to be taken into account when carrying out electron probe microanalysis. It also defines procedures for the determination of beam current, current density, dead time, wavelength resolution, background, analysis area, analysis depth and analysis volume.

This International Standard is intended for the analysis of a well-polished sample using normal beam incidence, and the parameters obtained may only be indicative for other experimental conditions.

This international standard is not designed to be used for energy dispersive X-ray spectroscopy.

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 Guide 25:1990, *General requirements for the competence of calibration and testing laboratories*

3 Terms and definitions

For the purposes of this International Standard the following terms and definitions apply.

3.1

analysis area

two-dimensional region of sample surface $\boxed{AC_1}$ text deleted $\boxed{AC_1}$ from which the full signal or a specified percentage of that signal is detected

3.2

analysis depth

distance from the sample surface to the bottom normal to the surface from which the full signal or a specified percentage of that signal is detected

3.3

analysis volume

three-dimensional region of a sample from which the full signal or a specified percentage of that signal is detected

3.4

background

non-characteristic component of an X-ray spectrum arising from the X-ray continuum

- 3.5**
beam current
electron current contained within the focused beam
- 3.6**
beam current density
beam current incident on the sample per unit area
- 3.7**
dead time
time associated with the measurement of a signal photon in a detector and/or counting system, representing the time that the system is unavailable to process the next photon
- 3.7**
wavelength resolution
full peak width at half maximum of a peak obtained from a single X-ray transition

4 Abbreviated terms

- EPMA: Electron Probe MicroAnalysis
- FWHM: Full Width at Half Maximum
- WD: Wavelength Dispersive

5 Experimental parameters

5.1 General

The parameters given in 5.2.1 to 5.2.4 should be known and recorded. Checking the calibration of beam energy, beam current and magnification together with counter dead time should be included in the maintenance schedule of the instrument.

5.2 Parameters related to the primary beam

5.2.1 Beam energy

The beam energy may typically range from 2 keV to 30 keV. In most cases the calibration of the beam energy is not critical for qualitative analysis.

NOTE Calibration is very critical in the case of use of low overvoltage ratio or during measurements relating to layer thickness or elemental depth distributions.

5.2.2 Beam current

Because X-ray peak intensity is directly proportional to beam current, the precision of the measurement of the beam current should be better than the precision required for quantitative analysis.

NOTE The beam current stability over long periods of time is absolutely essential for consistent quantitative analysis. The beam current stability should be tested periodically, especially prior to quantitative calibration and analysis. It is possible to compensate for small changes in beam current if this is recorded prior to or following each measurement. Then all X-ray peak and background measurements should be scaled appropriately by I_i/I_m where I_i is the initial beam current and I_m is the beam current at the time of the measurement.