



BSI Standards Publication

Microbeam analysis — Quantitative analysis using energy-dispersive spectrometry (EDS) for elements with an atomic number of 11 (Na) or above

National foreword

This British Standard is the UK implementation of ISO 22309:2011. It supersedes BS ISO 22309:2006, which is withdrawn.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 — Quantitative
analysis using energy-dispersive
spectrometry (EDS) for elements with an
atomic number of 11 (Na) or above**

*Analyse par microfaisceaux — Analyse élémentaire quantitative par
spectrométrie à sélection d'énergie (EDS) des éléments ayant un
numéro atomique de 11 (Na) ou plus*





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Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Specimen preparation	5
5 Preliminary precautions	6
6 Analysis procedure	7
7 Data reduction	9
7.1 General	9
7.2 Identification of peaks	9
7.3 Estimation of peak intensity	9
7.4 Calculation of k-ratios	10
7.5 Matrix effects	10
7.6 Use of reference materials	10
7.7 Standardless analysis	10
7.8 Uncertainty of results	11
7.9 Reporting of results	12
Annex A (informative) The assignment of spectral peaks to their elements	13
Annex B (informative) Peak identity/interferences	15
Annex C (informative) Factors affecting the uncertainty of a result	17
Annex D (informative) Analysis of elements with atomic number < 11	19
Annex E (informative) Example data from a reproducibility study within a laboratory and between laboratories	20
Bibliography	22

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 22309 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*.

This second edition cancels and replaces the first edition (ISO 22309:2006), of which it constitutes a minor revision. The main changes are as follows:

- the title has been modified to make it quite clear that the method is not suitable for light elements with an atomic number of less than 11;
- Subclause 6.2 has been modified to take into account the use of silicon drift detectors.

Introduction

X-rays generated when a high-energy electron beam interacts with a specimen have energies (wavelengths) which are characteristic of the chemical elements (atom types) present in the specimen. The intensity of these X-rays from each element is related to the concentration of that element in the specimen. If these intensities are measured, compared with those from a suitable reference material or set of reference materials, and corrected in an appropriate manner, the concentration of each element can be determined. “Standardless” procedures also provide quantitative information, but involve a comparison with previously measured reference intensities that are stored within the software package or are calculated theoretically; such procedures may, depending on any assumptions made, be inherently less accurate than the method employing reference materials (see References [1] to [8] in the Bibliography). There are two common methods of detecting the characteristic X-rays that are produced, one which relies on wavelength dispersive spectrometry (WDS) and the other which uses energy-dispersive spectrometry (EDS). This International Standard relates to the latter, EDS.

Using EDS, the quantitative analysis of light elements (i.e. atomic number $Z < 11$, below Na) is more complex and some of the problems are discussed in this International Standard.

Microbeam analysis — Quantitative analysis using energy-dispersive spectrometry (EDS) for elements with an atomic number of 11 (Na) or above

1 Scope

This International Standard gives guidance on the quantitative analysis at specific points or areas of a specimen using energy-dispersive spectrometry (EDS) fitted to a scanning electron microscope (SEM) or an electron probe microanalyser (EPMA); any expression of amount, i.e. in terms of percent (mass fraction), as large/small or major/minor amounts is deemed to be quantitative. The correct identification of all elements present in the specimen is a necessary part of quantitative analysis and is therefore considered in this International Standard. This International Standard provides guidance on the various approaches and is applicable to routine quantitative analysis of mass fractions down to 1 %, utilizing either reference materials or “standardless” procedures. It can be used with confidence for elements with atomic number $Z > 10$.

Guidance on the analysis of light elements with $Z < 11$ is also given.

NOTE With care, mass fractions as low as 0,1 % are measurable when there is no peak overlap and the relevant characteristic line is strongly excited. This International Standard applies principally to quantitative analyses on a flat polished specimen surface. The basic procedures are also applicable to the analysis of specimens that do not have a polished surface but additional uncertainty components will be introduced.

There is no accepted method for accurate quantitative EDS analysis of light elements. However, several EDS methods do exist. These are the following:

- a) Measuring peak areas and comparing intensities in the same way as for heavier elements. For the reasons explained in Annex D, the uncertainty and inaccuracy associated with the results for light elements will be greater than for the heavier elements.
- b) Where the light element is known to be combined stoichiometrically with heavier elements ($Z > 10$) in the specimen, its concentration can be determined by summing the appropriate proportions of concentrations of the other elements. This is often used for the analysis of oxygen in silicate mineral specimens.
- c) Calculation of concentration by difference where the light element percentage is 100 % minus the percentage sum of the analysed elements. This method is only possible with good beam-current stability and a separate measurement of at least one reference specimen and it requires very accurate analysis of the other elements in the specimen.

Annex D summarizes the problems of light element analysis, additional to those that exist for quantitative analysis of the heavier elements. If both EDS and wavelength spectrometry (WDS) are available, then WDS can be used to overcome the problems of peak overlap that occur with EDS at low energies. However, many of the other issues are common to both techniques.

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 14594, *Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy*

ISO 15632:2002, *Microbeam analysis — Instrumental specification for energy dispersive X-ray spectrometers with semiconductor detectors*

ISO 16700, *Microbeam analysis — Scanning electron microscopy — Guidelines for calibrating image magnification*

ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*

3 Terms and definitions

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

3.1
absorption correction
matrix correction arising from the loss of X-ray intensity from an element due to photoelectric absorption by all elements within the specimen while passing through it to the detector

3.2
accuracy
closeness of agreement between the “true” value and the measured value

3.3
accelerating voltage
potential difference applied between the filament and anode in order to accelerate the electrons emitted from the source

NOTE Accelerating voltage is expressed in kilovolts.

3.4
atomic number correction
matrix correction which modifies intensity from each element in the specimen and standards to take account of electron backscattering and stopping power, the magnitudes of which are influenced by all the elements in the analysed volume

3.5
beam current
electron current contained within the beam

NOTE Beam current is expressed in nanoamperes.

3.6
beam stability
extent to which beam current varies during the course of an analysis

NOTE Beam stability is expressed in percent per hour.

3.7
bremsstrahlung
background continuum of X-rays generated by the deceleration of electrons within the specimen

3.8
certified reference material
CRM
reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body

3.9
characteristic X-ray
photon of electromagnetic radiation created by the relaxation of an excited atomic state caused by inner shell ionization following inelastic scattering of an energetic electron, or by absorption of an X-ray photon

3.10
dead time

time that the system is unavailable to record a photon measurement because it is busy processing a previous event

NOTE This is frequently expressed as a percentage of the total time (see also live time).

3.11
energy-dispersive spectrometry
EDS

form of X-ray spectrometry in which the energy of individual photons are measured and used to build up a digital histogram representing the distribution of X-rays with energy

3.12
electron probe microanalysis

a technique of spatially resolved elemental analysis based on electron-excited X-ray spectrometry with a focused electron probe and an interaction/excitation volume with micrometre to sub-micrometre dimensions

3.13
escape peaks

peaks that occur as a result of loss of incident photon energy by fluorescence of the material of the detector

NOTE 1 These occur at an energy equal to that of the incident characteristic peak minus the energy of the X-ray line(s) emitted by the element(s) in the detector (1,734 keV for silicon).

NOTE 2 They cannot occur below the critical excitation potential of the detector material, e.g. Si K escape does not occur for energies below 1,838 keV.

3.14
fluorescence

photoelectric absorption of any X-ray radiation (characteristic or bremsstrahlung) by an atom which results in an excited atomic state which will de-excite with electron shell transitions and subsequent emission of an Auger electron or the characteristic X-ray of the absorbing atom

3.15
fluorescence correction

matrix correction which modifies the intensity from each element in the specimen and standards to take account of excess X-rays generated from element "A" due to the absorption of characteristic X-rays from element "B" whose energy exceeds the critical (ionization) energy of "A"

3.16
full width at half maximum
FWHM

measure of the width of an X-ray peak in which the background is first removed to reveal the complete peak profile

NOTE FWHM is determined by measuring the width at half the maximum height.

3.17
incident beam energy

energy gained by the beam as a result of the potential applied between the filament and anode

3.18
k-ratio

net peak intensity (after background subtraction) for an element found in the specimen, divided by the intensity, recorded or calculated, of the corresponding peak in the spectra of a reference material