

---

---

**Microbeam analysis — Electron  
probe microanalysis — Quantitative  
point analysis for bulk specimens  
using wavelength dispersive X-ray  
spectroscopy**

*Analyse par microfaisceaux — Microsonde de Castaing — Analyse  
quantitative ponctuelle d'échantillons massifs par spectrométrie à  
dispersion de longueur d'onde*

This document is a preview generated by EMS



**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2016, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Ch. de Blandonnet 8 • CP 401  
CH-1214 Vernier, Geneva, Switzerland  
Tel. +41 22 749 01 11  
Fax +41 22 749 09 47  
copyright@iso.org  
www.iso.org

# Contents

	Page
Foreword.....	iv
Introduction.....	v
<b>1 Scope.....</b>	<b>1</b>
<b>2 Normative references.....</b>	<b>1</b>
<b>3 Abbreviated terms.....</b>	<b>1</b>
<b>4 Procedure for quantification.....</b>	<b>2</b>
4.1 General procedure for quantitative microanalysis.....	2
4.1.1 Principle and procedure of quantitative microanalysis.....	2
4.1.2 Coverage of the quantitative analysis.....	2
4.1.3 Selection of reference materials.....	3
4.2 Specimen preparation.....	3
4.3 Calibration of the instrument.....	3
4.3.1 Accelerating voltage.....	3
4.3.2 Probe current.....	3
4.3.3 X-ray spectrometer.....	3
4.3.4 Dead time.....	4
4.4 Analysis conditions.....	4
4.4.1 Accelerating voltage.....	4
4.4.2 Probe current.....	4
4.4.3 Analysis position.....	4
4.4.4 Probe diameter.....	5
4.4.5 Scanning the focused electron beam.....	5
4.4.6 Specimen surface.....	5
4.4.7 Selection of X-ray line.....	5
4.4.8 Spectrometer.....	5
4.4.9 Method for measurement of X-ray peak intensity.....	6
4.4.10 Method for measurement of background intensity.....	6
4.5 Correction method based on analytical models.....	6
4.5.1 Principles.....	6
4.5.2 Correction models.....	7
4.6 Calibration curve method.....	7
4.6.1 Principle.....	7
4.6.2 Selection of reference materials.....	8
4.6.3 Procedure.....	8
4.7 Uncertainty.....	8
<b>5 Test report.....</b>	<b>8</b>
<b>Annex A (informative) Physical effects and correction.....</b>	<b>10</b>
<b>Annex B (informative) Outline of various correction techniques.....</b>	<b>12</b>
<b>Annex C (informative) Measurement of the <i>k</i>-ratios in case of “chemical effects”.....</b>	<b>14</b>
<b>Bibliography.....</b>	<b>15</b>

## 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](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

The committee responsible for this document is ISO/TC 202, *Microbeam analysis*, Subcommittee SC 2, *Electron probe microanalysis*.

This second edition cancels and replaces the first edition (ISO 22489:2006), of which it constitutes a minor revision to update the references and to revise text in [4.4.1](#) and [4.4.8](#).

## Introduction

Electron probe microanalysis is widely used for the quantitative analysis of elemental composition in materials. It is a typical instrumental analysis and the electron probe microanalyser has been greatly improved to be user friendly. Obtaining accurate results with this powerful tool requires that it be properly used. In order to obtain reliable data, however, optimum procedures must be followed. These procedures, such as preparation of specimens, measurement of intensities of characteristic X-rays and calculations of concentrations calculated from X-ray intensities, are given for use as standard procedures in this International Standard.



# Microbeam analysis — Electron probe microanalysis — Quantitative point analysis for bulk specimens using wavelength dispersive X-ray spectroscopy

## 1 Scope

This International Standard specifies requirements for the quantification of elements in a micrometre-sized volume of a specimen identified through analysis of the X-rays generated by an electron beam using a wavelength dispersive spectrometer (WDS) fitted either to an electron probe microanalyser or to a scanning electron microscope (SEM).

This International Standard also describes the following:

- the principle of the quantitative analysis;
- the general coverage of this technique in terms of elements, mass fractions and reference specimens;
- the general requirements for the instrument;
- the fundamental procedures involved such as specimen preparation, selection of experimental conditions, the measurements, the analysis of these and the report.

This International Standard is intended for the quantitative analysis of a flat and homogeneous bulk specimen using a normal incidence beam. It does not specify detailed requirements for either the instruments or the data reduction software. Operators should obtain information such as installation conditions, detailed procedures for operation and specification of the instrument from the makers of any products used.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 14595, *Microbeam analysis — Electron probe microanalysis — Guidelines for the specification of certified reference materials (CRMs)*

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

## 3 Abbreviated terms

EPMA	electron probe microanalyser
SEM	scanning electron microscope
EDS	energy dispersive spectrometer
PHA	pulse height analyser
P/B	peak-to-background ratio