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Microbeam analysis — Selected instrumental performance parameters for the specification and checking of energy-dispersive X-ray spectrometers (EDS) for use with a scanning electron microscope (SEM) or an electron probe microanalyser (EPMA)

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Contents

Page

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).

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).

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

For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 15632:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

— The title has been detailed;

— The definition of *dead time* (3.4) is more detailed;

— A Note (including a new Reference [5]) has been added to General description (4.1) related to the net active sensor area;

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Progress in energy-dispersive X-ray spectrometry (EDS) by means of improved manufacturing technologies for detector crystals and the application of advanced pulse-processing techniques have increased the general performance of spectrometers, in particular at high count rates and at low energies (below 1 keV). Meanwhile, the Si-Li detector technology has been successfully replaced by the silicon drift detector (SDD) technology which provides performance comparable to Si-Li detectors, even at considerably higher count rates. In addition, a smaller detector capacitance results in the capability of measuring even higher count rates and in the availability of larger area detectors. This document has therefore been updated with criteria for the evaluation of the performance of such modern spectrometers.

A spectrometer is commonly specified by its energy resolution at high energies defined as the full peak width at half maximum (FWHM) of the manganese Kα line. To specify the properties in the low energy range, values for the FWHM of carbon K, fluorine K or/and the zero peak are given by the manufacturers. Some manufacturers also specify a peak-to-background ratio, which may be defined as a peak-to-shelf ratio in a spectrum from an ^{55}Fe source or as a peak-to-valley ratio in a boron spectrum. Differing definitions of the same quantity have sometimes been employed. The sensitivity of the spectrometer at low energies related to that at high energies depends strongly on the construction of the detector crystal and the X-ray entrance window used. Although high sensitivity at low energies is important for the application of the spectrometer in the analysis of light-element compounds, normally, the manufacturers do not specify an energy dependence for spectrometer efficiency.

This document was developed in response to a worldwide demand for minimum specifications of an energy-dispersive X-ray spectrometer. EDS is one of the most applied methods used to analyse the chemical composition of solids and thin films. This document should permit comparison of the performance of different spectrometer designs on the basis of a uniform specification and help to find the optimum spectrometer for a particular task. In addition, this document contributes to the equalization of performances in separate test laboratories. In accordance with ISO/IEC 17025[1], such laboratories should periodically check the calibration status of their equipment according to a defined procedure. This document may serve as a guide for similar procedures in all relevant test laboratories.

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1 Scope

This document defines the most important quantities that characterize an energy-dispersive X-ray spectrometer consisting of a semiconductor detector, a pre-amplifier and a signal-processing unit as the essential parts. This document is only applicable to spectrometers with semiconductor detectors operating on the principle of solid-state ionization. This document specifies minimum requirements and how relevant instrumental performance parameters are to be checked for such spectrometers attached to a scanning electron microscope (SEM) or an electron probe microanalyser (EPMA). The procedure used for the actual analysis is outlined in ISO 22309[2] and ASTM E1508[3] and is outside the scope of this document.

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 23833, *Microbeam analysis — Electron probe microanalysis (EPMA) — Vocabulary*

ISO 22493, *Microbeam analysis — Scanning electron microscopy — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 23833, ISO 22493 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

NOTE With the exception of 3.1, 3.2, 3.2.1, 3.2.2, 3.9, 3.11, 3.12, 3.13 and 3.14, these definitions are given in the same or analogous form in ISO 22309[2], ISO 18115-1[4] and ISO 23833.

3.1
energy-dispersive X-ray spectrometer
device for determining X-ray signal intensity as a function of the energy of the radiation by recording the whole X-ray spectrum simultaneously

Note 1 to entry: The spectrometer consists of a solid-state detector, a preamplifier, and a pulse processor. The detector converts X-ray photon energy into electrical current pulses which are amplified by the preamplifier. The pulse processor then sorts the pulses by amplitude so as to form a histogram distribution of X-ray *signal intensity* (3.8) vs energy.