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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 ... **iv**

Introduction ... **v**

1 Scope ... **1**

2 Normative references ... **1**

3 Terms and definitions ... **1**

4 Requirements ... **3**
4.1 General description ... 3
4.2 Energy resolution ... 4
4.3 Dead time ... 4
4.4 Peak-to-background ratio ... 4
4.5 Energy dependence of instrumental detection efficiency ... 5

5 Check of further performance parameters ... **5**
5.1 General ... 5
5.2 Stability of the energy scale and resolution ... 5
5.3 Pile-up effects ... 5
5.4 Periodical check of spectrometer performance ... 5

Annex A (normative) **Measurement of line widths (FWHMs) to determine the energy resolution of the spectrometer** ... **6**

Annex B (normative) **Measurement of the L/K ratio as a measure for the energy dependence of the instrumental detection efficiency** ... **11**

Bibliography ... **13**

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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.

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).

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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;

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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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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)

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.

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3.2
count rate
number of X-ray photons per second

3.2.1
input count rate
ICR
number of X-ray photons absorbed in the active detector area per second that are input to the electronics

3.2.2
output count rate
OCR
number of valid X-ray photon measurements per second that are output by the electronics and stored in memory, including sum peaks

Note 1 to entry: When the electronics measures individual X-ray photon energies, there is some *dead time* (3.4) associated with each individual measurement. Consequently, the number of successful measurements is less than the number of incident photons in every practical case. Thus, the accumulation rate into the spectrum (*output count rate* (3.2.2)) is less than the count rate of photons that cause signals in the detector (*input count rate* (3.2.1)). *Output count rate* (3.2.2) may be equal to *input count rate* (3.2.1), e.g. at very low count rates and for very short measurements.

3.3
real time
duration in seconds of an acquisition as it would be measured with a conventional clock

Note 1 to entry: For X-ray acquisition, in every practical case the *real time* (3.3) always exceeds the *live time* (3.5).

3.4
dead time
time during which the system is unable to record a photon measurement because it is busy processing a previous event (frequently expressed as a percentage of the *real time* (3.3))

Note 1 to entry: Dead time = real time – live time.

Note 2 to entry: Dead-time fraction = 1 – OCR/ICR.

3.5
live time
effective duration of an acquisition, in seconds, after accounting for the presence of *dead time* (3.4)

Note 1 to entry: Live time = real time for an analysis minus cumulative dead time.

Note 2 to entry: Live-time fraction = 1 – dead-time fraction.

3.6
spectral channel
discrete interval of the measured energy for the histogram of recorded measurements with a width defined by a regular energy increment

3.7
instrumental detection efficiency
ratio of quantity of detected photons and quantity of the photons incident on the active detector area

3.8
signal intensity
strength of the signal in counts per channel or counts per second per channel at the spectrometer output after pulse processing

Note 1 to entry: This definition permits intensity to be expressed as either “counts” or “counts per second” (CPS). The distinction is not relevant to the procedures described in this standard so long as either one or the other is consistently employed.

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3.9
peak height
maximum *signal intensity* (3.8) of a spectral peak measured as height of the peak above a defined *background signal* (3.11)

3.10
peak area
net peak area
sum of *signal intensities* (3.8) of a spectral peak after *background signal* (3.11) removal

3.11
background signal
continuous X-ray spectrum
continuum
non-characteristic component of an X-ray spectrum arising from the *bremsstrahlung* (3.12) and other effects

Note 1 to entry: Apart from the *bremsstrahlung* (3.12), degraded events occurring due to the operation of the spectrometer may contribute to the *background signal* (3.11). Extraneous signals arising from one or more parts of the spectrometer, microscope chamber or specimen itself (by X-ray scattering) may also add to the *background signal* (3.11).

3.12
bremsstrahlung
braking radiation
non-characteristic X-ray spectrum created by electron deceleration in the coulombic field of an atom and having an energy distribution from 0 up to the incident beam energy

3.13
X-ray take-off angle
TOA
angle between the specimen surface and the direction where exiting X-rays will strike the centre of the detector's sensor

Note 1 to entry: With increasing solid angle encompassed by the detector, TOA may vary significantly within a range around that TOA corresponding to the central position on the X-ray sensor.

3.14
zero peak
strobe zero peak
noise peak
artificial peak generated by the pulse processor initiating measurements whenever there is a suitable gap between real photon pulses

Note 1 to entry: The zero peak effectively simulates the peak that would be obtained from zero energy photons and can be used to calibrate the energy scale and determine the electronic noise contribution. However, it is not always visible to the user. The strobe zero peak should not be confused with a structure in the energy histogram near zero energy that is the result of pulse measurements triggered by electronic noise fluctuations that are indistinguishable from low energy photon pulses. The strobe zero peak may sometimes be shown instead of this structure or the structure may be excluded from the display.

4 Requirements

4.1 General description

The manufacturer shall describe, using appropriate reference texts, the essential design elements of the spectrometer in order to permit the user to evaluate the performance of the spectrometer. Elements that are indispensable for the evaluation of the suitability of a spectrometer for a certain field of application shall be given explicitly. These are to include the type of EDS (Si-Li EDS, HpGe EDS, SDD EDS, etc.), the thickness of the sensor, the net active sensor area (excluding the window grid area and after

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