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

*Analyse par microfaisceaux — Paramètres de performance instrumentale sélectionnés pour la spécification et le contrôle des spectromètres X à sélection d'énergie utilisés en microanalyse par sonde à électrons*

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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.

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 15632 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 (see the Introduction, first paragraph, for details).

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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 International Standard 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  $K\alpha$  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  $^{55}\text{Fe}$  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 International Standard 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 International Standard 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 International Standard contributes to the equalization of performances in separate test laboratories. In accordance with ISO/IEC 17025,<sup>[1]</sup> such laboratories have to periodically check the calibration status of their equipment according to a defined procedure. This International Standard 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 for use in electron probe microscope or an electron probe microanalyser (EPMA)

## 1 Scope

This International Standard 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 International Standard is only applicable to spectrometers with semiconductor detectors operating on the principle of solid-state ionization. This International Standard 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<sup>[2]</sup> and ASTM E1508<sup>[3]</sup> and is outside the scope of this International Standard.

## 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 International Standard, the terms and definitions given in ISO 23833, ISO 22493 and the following apply.

Note 1 to entry 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,<sup>[2]</sup> ISO 18115-1<sup>[4]</sup> 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 vs energy.

### 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 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", OCR) is less than the count rate of photons that cause signals in the detector ("input count rate", ICR). OCR may be equal to ICR, 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 always exceeds the live time.

### 3.4

#### **dead time**

$\tau$

time during which the system is unable to record a photon measurement because it is busy processing a previous event

Note 1 to entry: Dead-time fraction =  $1 - \text{OCR}/\text{ICR}$ .

### 3.5

#### **live time**

effective duration of an acquisition, in seconds, after accounting for the presence of dead-time

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

Note 2 to entry: Live-time fraction =  $1 - \text{dead-time fraction}$ .

### 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.

### 3.9

#### **peak height**

maximum signal intensity of a spectral peak measured as height of the peak above a defined background signal

### 3.10

#### **peak area**

#### **net peak area**

sum of signal intensities of a spectral peak after background removal



**3.11****background signal  
continuous X-ray spectrum  
continuum**

non-characteristic component of an X-ray spectrum arising from the bremsstrahlung and other effects

Note 1 to entry: Apart from the bremsstrahlung, degraded events occurring due to the operation of the spectrometer may contribute to the background. 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.

**3.12****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**

an 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 collimation) and the type of the window (beryllium, thin film window or windowless). Parameters which may not be encompassed by this International Standard, but that may influence detector performance, e.g. the construction principle of the cooling system, shall be explained in the reference text. Some detector systems are capable of very high count rates, but at high count rates other specifications like energy resolution may alter and artefacts may appear in the spectrum. All specifications should therefore be accompanied by a statement of the count rate at which they are measured and it should not be assumed that the specification will be the same at other count rates.