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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 microanalysis

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 second edition cancels and replaces the first edition (ISO 15632:2002), which has been technically revised (see the Introduction, first paragraph, for details).

Introduction

Recent 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). A revision of this International Standard became necessary because silicon drift detector (SDD) technology was not included. SDDs provide performance comparable to Si-Li detectors, even at considerably higher count rates. In addition, a larger detector active area results in the capability of measuring even higher count rates. This International Standard has therefore been updated with criteria for the evaluation of the performance of such modern spectrometers.

In the past, a spectrometer was 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 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 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[1], 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.

Microbeam analysis — Selected instrumental performance parameters for the specification and checking of energy-dispersive X-ray spectrometers for use in electron probe microanalysis

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[2] and ASTM E1508[3] 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*

3 Terms and definitions

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

NOTE With the exception of 3.1, 3.2, 3.2.1, 3.2.2, 3.11, 3.12 and 3.13, 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 intensity as a function of the energy of the radiation by recording the whole X-ray spectrum simultaneously

NOTE 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 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 detector per second

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

NOTE 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 For X-ray acquisition, in every practical case the real time always exceeds the live time.

3.4

dead time

τ

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

NOTE 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

NOTE 1 Live time = real time for an analysis minus cumulative dead time.

NOTE 2 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 available for measurement

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 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 intensity

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

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 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 center of the detector's sensor

NOTE 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.

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 (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 artifacts 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.

4.2 Energy resolution

The energy resolution shall be specified as the FWHM of the manganese Kα peak and determined in accordance with Annex A. Spectrometers that claim detection of X-rays lower than 1 keV shall also be specified by the FWHM of the carbon K and the fluorine K-lines. The specified FWHM shall be an upper limit in that the resolution determined in accordance with Annex A is guaranteed to be no greater than the specified value. The resolution value shall be accompanied by a statement of count rate for which the specification is valid. For most detector systems the best energy resolution is attained at an ICR $<$ 1 000 counts/s and the best energy resolution shall be specified. Where detector systems offer higher count rate capability, e.g. SDD EDS, the energy resolution shall also be specified at high ICR, e.g. 50 000 counts/s, 500 000 counts/s.

4.3 Dead time

In order to evaluate the process time of the EDS, complementary to the energy resolution specified in 4.2, the corresponding dead-time fraction should be specified. The calculation of the dead-time fraction is given in 3.4.

Dead time is a consequence of the electronics rejecting “bad” measurements in order to achieve high spectrum fidelity. In many systems, the rejection criterion is designed to ensure that the measurement time for each