INTERNATIONAL STANDARD

ISO 15632

First edition
2002-12-01

Microbeam analysis — Instrumental specification for energy dispersive X-ray spectrometers with semiconductor detectors

Analyse par microfaisceaux — Spécifications instrumentales pour spectromètres à rayons X à dispersion d'énergie avec détecteurs à semiconducteurs

Reference number
ISO 15632:2002(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 15632:2002
https://standards.iteh.ai/catalog/standards/sist/4978b842-8089-472d-b020-0c33c5e8cded/iso-15632-2002

© ISO 2002

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15632 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*.

Annexes A and B form a normative part of this International Standard.

Introduction

Recent progress in energy dispersive X-ray spectrometry (EDS) by means of improved manufacturing technologies for detector crystals, new materials for the detector's X-ray entrance window and the application of advanced pulse processing techniques have increased the general performance of spectrometers and extended their application to low energies (below 1 keV).

In the past, a spectrometer was commonly specified by its energy resolution at high energies defined as the full width at half maximum (FWHM) of the Mn-Kα line. To specify the properties in the low energy range, values for the FWHM of C-K, F-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 Fe^{55} source or as peak to valley ratio in a boron spectrum. Definitions of the same quantity may be different. 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 EDS spectrometers. 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, 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 — Instrumental specification for energy dispersive X-ray spectrometers with semiconductor detectors

1 Scope

This International Standard defines the most important quantities that characterize an energy dispersive X-ray spectrometer (EDS) 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 for such spectrometers attached to an electron probe microanalyser (EPMA) or a scanning electron microscope (SEM). Realization of the analysis is outside the scope of this International Standard.

2 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

NOTE With exception of 2.1 these definitions are given in the same or analogous form in ISO 18115.

2.1
energy dispersive spectrometer
spectrometer that records the whole X-ray spectrum simultaneously

NOTE The spectrometer consists of a solid state detector, a preamplifier and a pulse processor to convert the X-ray photons in electronic pulses, pulse height analysis being used to sort the pulses formed by the processor according to X-ray energy in spectral channels.

2.2
spectral channel
interval of the measured energy for a measured spectrum with a width defined by a regular energy increment

2.3
instrumental detection efficiency
ratio of quantity of detected photons and quantity of the photons available for measurement

2.4
signal intensity
strength of the signal in counts per channel or counts per second per channel at the spectrometer output after pulse processing

2.5
peak intensity
signal intensity of a spectral peak measured as height of the peak above a defined background

2.6
peak area
net peak area
area under a peak in a spectrum after background removal

2.7
background signal
signal present in a spectral channel due to bremsstrahlung or instrumental background

2.8

background, instrumental

signal contribution, generally unwanted, arising from one or more parts of the spectrometer, which adds to that emitted from the sample, so distorting the measured spectrum

3 Requirements

3.1 General description

The manufacturer shall describe, using appropriate reference texts, the essential constructive elements of the spectrometer in order to permit the user to evaluate the performance of the spectrometer. Constructive elements that are indispensable for the evaluation of the suitability of a spectrometer for a certain field of applications shall be given explicitly. These are the type of crystal material (silicon or germanium), the thickness of the crystal, the active crystal area 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. maximum count rates, construction principle of cooling etc. shall be explained in the reference text.

3.2 Energy resolution

The energy resolution shall be specified as FWHM of the Mn-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 C-K and the F-K lines. The specified FWHM shall be an upper limit. The resolution determined in accordance with annex A is guaranteed to be less than the specified value. The resolution figure shall be accompanied by a statement of count rate for which the specification is valid (e.g. $<$ 1 000 counts/s.

3.3 Peak to background ratio

The peak to background ratio shall be derived from an acquired spectrum of an ^{55}Fe source as a characteristic spectrometer parameter. The ratio shall be given by the peak intensity of the Mn-Kα line divided by the instrumental background. The instrumental background shall be calculated as the mean number of counts per channel within the energy range from 0,9 to 1,1 KeV. Sufficient counts shall be recorded in the spectrum to make the measure statistically significant and the electronics shall be set up to be 100 % effective in responding to pulses produced by signals in the 0,9 to 1,1 KeV energy band.

NOTE 1 The peak to background ratio depends on spectrometer resolution. Therefore, the ratio is only relevant for the comparison of spectrometers with similar resolution performance.

NOTE 2 In an electron microscope, the bremsstrahlung background from a manganese specimen may be considerably greater than the instrumental background, due to degraded events. Therefore, a manganese specimen cannot be used to measure the peak to background ratio.

3.4 Energy dependence of instrumental detection efficiency

The minimum specification for the energy dependence of the instrumental detection efficiency shall be the intensity ratio of a low energy line and a high energy line in the characteristic X-ray spectrum of a given material. This ratio shall be given as the net peak area ratio of the L series and Kα lines in the spectrum of a pure nickel or copper specimen, excited by a 20 keV electron beam perpendicular to the specimen surface and collected by the detector at a take-off angle of 35°.

NOTE 1 These measures are only appropriate for a detector thick enough to absorb at least 95 % of the incident X-ray energy at 8 keV.

NOTE 2 If the specimen chamber containing ports to fix the detector does not allow a take-off angle of 35°, the L/K ratio at any other take-off angle can be converted to that for 35° by applying the absorption correction formalism of quantitative electron probe microanalysis (see annex B).

Annex A
(normative)

Measurement of line widths (FWHM) to determine the energy resolution of the spectrometer

A.1 Specimens

Specimens shall be an encapsulated ^{55}Fe source to measure the manganese Kα line and a polytetrafluoroethylene (PTFE, Teflon®) piece or foil for the carbon and fluorine K lines. In order to check the FWHMs in the user laboratory, a polished manganese specimen can be used if ^{55}Fe is not available or may not be used in the laboratory due to safety regulations. Alternatives to PTFE to measure the carbon and fluorine K-lines are respectively glassy carbon and a fluorine-containing mineral such as CaF_2.

A.2 Specimen preparation

The PTFE specimen shall be coated with an approximately 20 nm thick evaporated or sputtered carbon layer in order to make the surface conductive. If ^{55}Fe is used with the spectrometer already attached to the scanning electron microscope, sources similar to Amersham Buchler ^{55} Fe gamma reference source VZ-1977 shall be also coated with carbon on one side only. This makes it easy to find the position of the encapsulated radioactive pill and to locate the source in the microscope at the correct working distance for X-ray spectrometry.

A.3 Preparatory work

Calibrate the energy scale of the spectrometer using manufacturer's recommended procedure and record the X-ray lines and energies used for that calibration.

A.4 Measurement conditions

- Select a channel width $\leqslant$ 10 eV.
- Operate at 10 keV primary electron energy to record the C-K and F-K lines.

  NOTE Under this condition the similarity of carbon and fluorine peak intensities in a PTFE spectrum enable the FWHM determination of both lines from one spectrum.
- Operate at 15 keV in the case of a manganese specimen, to record the Mn-K spectrum.
- Adjust the beam current so that the count rate during measurement does not exceed the limit of count rate (e.g. 1 000 counts/s) for which the specification is valid.
- Peak intensities shall exceed 10 000 counts.

A.5 Background subtraction

The background shall be neglected in spectra from a ^{55}Fe source, PTFE or glassy carbon specimens.

NOTE The peak to background ratio for the K-lines in the case of a PTFE or glassy carbon specimen is above 100. Neglecting the background slightly overestimates the peak FWHM by about 1 eV or less but avoids all problems associated with background modelling and subtraction. It can be considered as a reliable and reproducible measure for system to system comparison.

A linear background shall be subtracted before FWHM calculation in the case of manganese- and fluorine-containing mineral specimens. The linear background shall be fixed at the low and high energy side of the peak in a distance of at least the FWHM × 2 with count values taken as the mean value from five neighbouring channels representing the background signal.

A.6 Calculation of FWHM

For the calculation of the FWHM the following procedure shall be applied (in agreement with [3] or [4]). Find those two channels at the low and high energy side of the peak with the counts just above and just below half of the counts in the peak maximum. Interpolate between each pair to find those fractions of the channel width that correspond exactly with the half maximum. Count the channel distance between the half maximum positions. Multiply the distance with the channel width in eV found after calibration of the spectrometer.

The final result shall be the mean of at least 5 individual measurements.

NOTE For some special pulse processor designs the resolution may show a minor dependence on the signal intensity and the spectrum content. In such cases the measured FWHM might slightly depend on the used specimen.

A.7 Example

Figures A.1 and A.2 give examples of a manganese spectrum from an ^{55}Fe source measured with 10 eV channel width and a PTFE spectrum measured at 10 kV beam voltage, 5 eV channel width, and TOA $=$ 30° together with the calculated FWHMs.

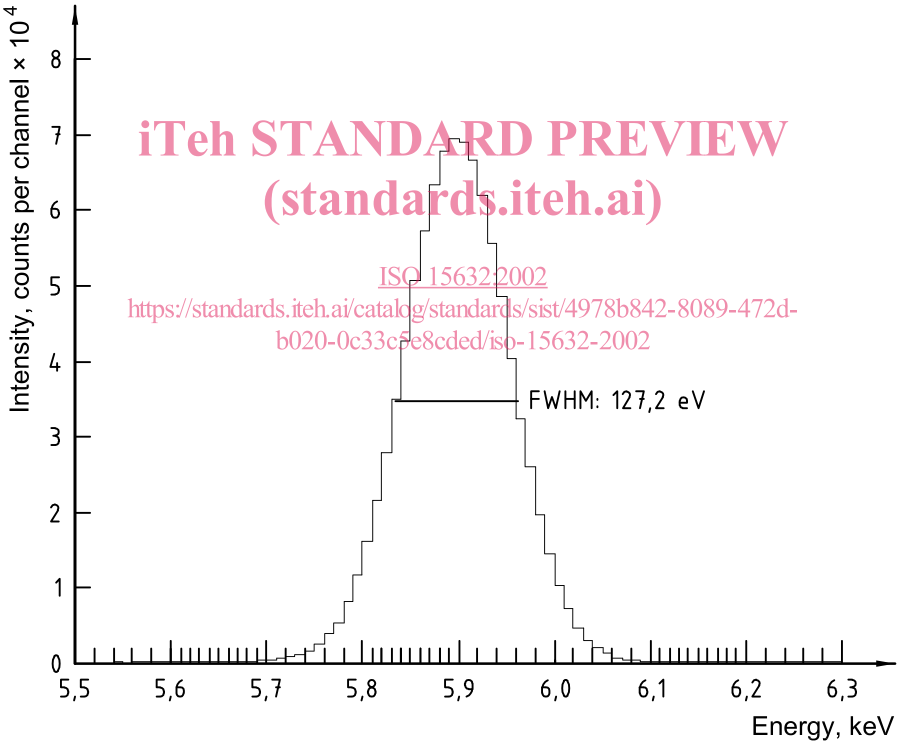

Figure A.1 — Manganese Kα spectrum emitted from an ^{55}Fe source and calculated FWHM

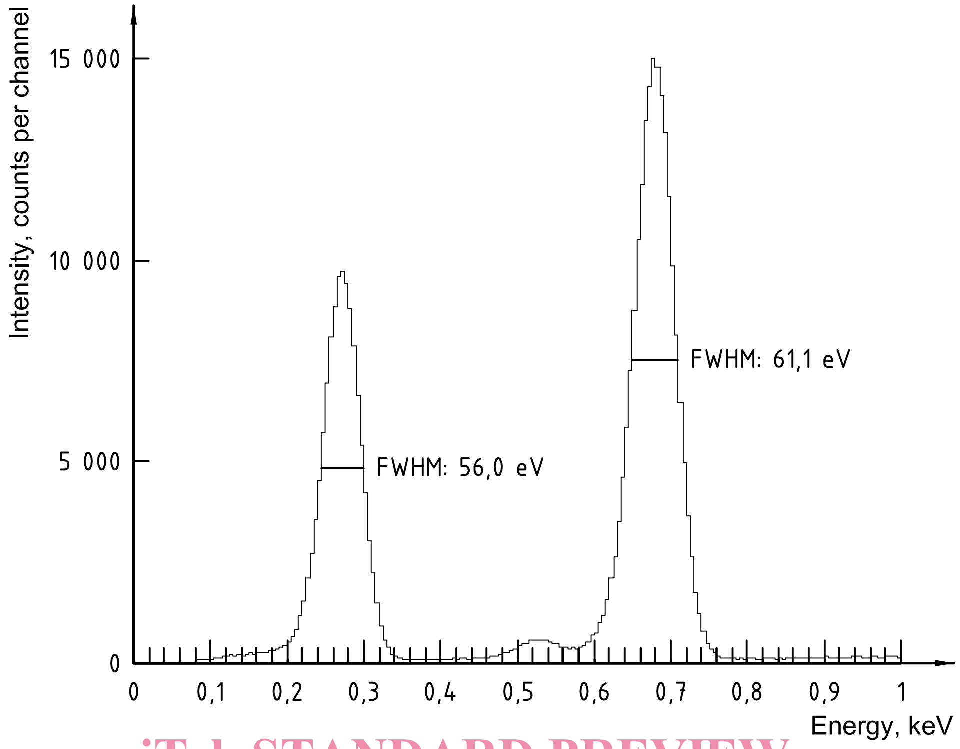

Figure A.2 — 10 keV **spectrum from a PTFE specimen and calculated FWHMs for the carbon and fluorine K lines**