INTERNATIONAL STANDARD

ISO 15632

Third edition
2021-02

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)

Reference number
ISO 15632:2021(E)

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Published in Switzerland

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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;

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.

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.

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.

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 collimation) and the type of the window (beryllium, thin film window or windowless). Parameters which may not be encompassed by this document, 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.

NOTE In many cases the specific geometry of the EDS detector at a particular SEM/EPMA chamber can result in a reduction of the net active sensor area as expected after subtraction of shadowing area caused by the window grid. For example, a falsely mounted collimator, electron trap or shadowing by other parts in the SEM chamber can reduce additionally the illumination of the detector with X-rays. A practical procedure how to determine experimentally the effective area of an EDS detector and under which conditions is described in Reference [5].

4.2 Energy resolution

The performance of an EDS depends on the pulse processing details. 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 photon is identical. However, it is possible to reduce the dead-time fraction by relaxing the criterion for pulse rejection and allowing the measurement time per photon to vary according to the arrival time of photons (“adaptive filtering”). In this case, the process time is not defined, the peak shape and resolution will change with count rate and this could cause analytical results to vary with count rate. Therefore, any specification of dead-time fraction should include a description of the essential design elements of the electronics as per 4.1.

4.4 Peak-to-background ratio

The peak-to-background ratio shall be derived at the point of manufacture of the spectrometer from an acquired spectrum of an ^{55}Fe source as a characteristic spectrometer parameter. The ratio shall be given by the peak height of the manganese Kα line divided by the background. The background shall be calculated as the mean number of counts per channel within the energy range from 0.9 keV to 1.1 keV. Sufficient counts shall be recorded in the spectrum to make the measure statistically significant (as per A.4) and the electronic threshold(s) shall be set up so that any energy cut-off occurs well below the specified range.

NOTE 1 Beside other factors, 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 from a manganese specimen can be considerably greater than the background component caused by degraded events. Therefore, a manganese specimen cannot be used to measure the peak-to-background ratio.

4.5 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 of the L series lines divided by the net peak area of Kα series 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°. The specimens to be used, the measurement conditions, the calculation of L/K ratio and its conversion for TOA ≠ 35° are given in Annex B.

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 B.4).

5 Check of further performance parameters

5.1 General

The parameters described in 4.2, 4.3 and 4.5 shall be specified by the manufacturers and checked periodically by the users. In addition, the users may check other parameters and effects which are relevant for the performance of the spectrometer, especially at high count rates.

5.2 Stability of the energy scale and resolution

For the range of count rates that will be used in practice, the stability of the energy scale, i.e. of the peak positions, can be checked. For example, the change in centroid position for Mn Kα peak over an ICR range of 1 000 to 50 000 counts/s can be evaluated on a periodical basis. For the determination of the peak centroid position only data at or near the FWHM energy locations should be used. The stability of resolution with count rate can also be checked.

5.3 Pile-up effects

At high count rates, pile-up effects produce both sum peaks and pile-up continuum artefacts, so that relative peak areas are more or less altered in the X-ray spectrum at high count rates even after electronics and software corrections. The pile-up effects might be checked e.g. by measuring a spectrum at a low count rate (below 1 000 counts/s) of a reference material (pure aluminium is a good choice) with known beam current for a known time. The beam current should then be increased and a new spectrum obtained at higher count rate. The spectra should then be scaled by dividing by the beam current and time and then compared. This will reveal any distortions due to pile-up and any energy-dependent distortion in the live-time correction.

5.4 Periodical check of spectrometer performance

The periodical check of system performance may be carried out by users using a known reference material that is appropriate for the type of analysis to be conducted. For example, a dedicated test material and measurement procedures are described in References [6] and [7].

Annex A
(normative)

Measurement of line widths (FWHMs) 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) piece or foil for the carbon and fluorine K-lines. In order to check periodically the FWHMs at high count rates as well as 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 ≤10 eV.

Operate at 10 keV primary electron energy to record the carbon K and fluorine K-lines.

NOTE Under this condition, the similarity of carbon and fluorine peak heights in a PTFE spectrum enables the FWHM determination of both lines from one spectrum.

Operate at 15 keV beam energy in the case of a manganese specimen, to record the manganese K-spectrum.

Adjust the beam current so that the ICR during measurement is as specified in 4.2.

Peak heights shall exceed 10 000 counts.

A.5 Background subtraction

The background shall be neglected in spectra from an ^{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 of fluorine-containing mineral specimens. The linear background shall be interpolated between background points located twice the FWHM above and below the peak centre. Each background point shall be established as the mean value of five adjacent channels centred on the chosen background point.

A.6 Calculation of FWHM

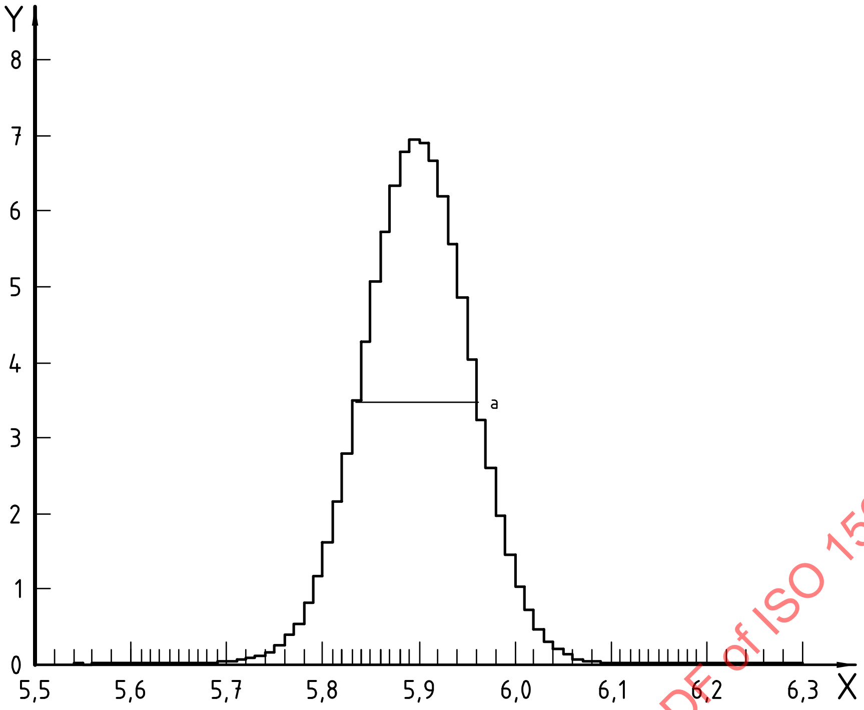
For the calculation of the FWHM, the following procedure shall be applied (in agreement with Reference [8] or [9]). Find those two channels at the low- and high-energy side of the peak with the counts just above and just below half of the peak height. Interpolate between each pair to find those fractions of the channel width that correspond exactly with the half maximum. Compute the FWHM as the interval, in channels, between the two half-maximum positions (including fractional parts), multiplied by the channel width, in eV, established by calibration of the spectrometer.

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

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

A.7 Examples

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 keV beam energy, 5 eV channel width, and TOA = 30° together with the calculated FWHMs. Figure A.3 illustrates the application of the procedure for the calculation of the FWHM to the manganese Kα peak as described in A.6 after a background subtraction as described in A.5. The corresponding beam energy was 15 keV, the channel width 10 eV and TOA = 35°.



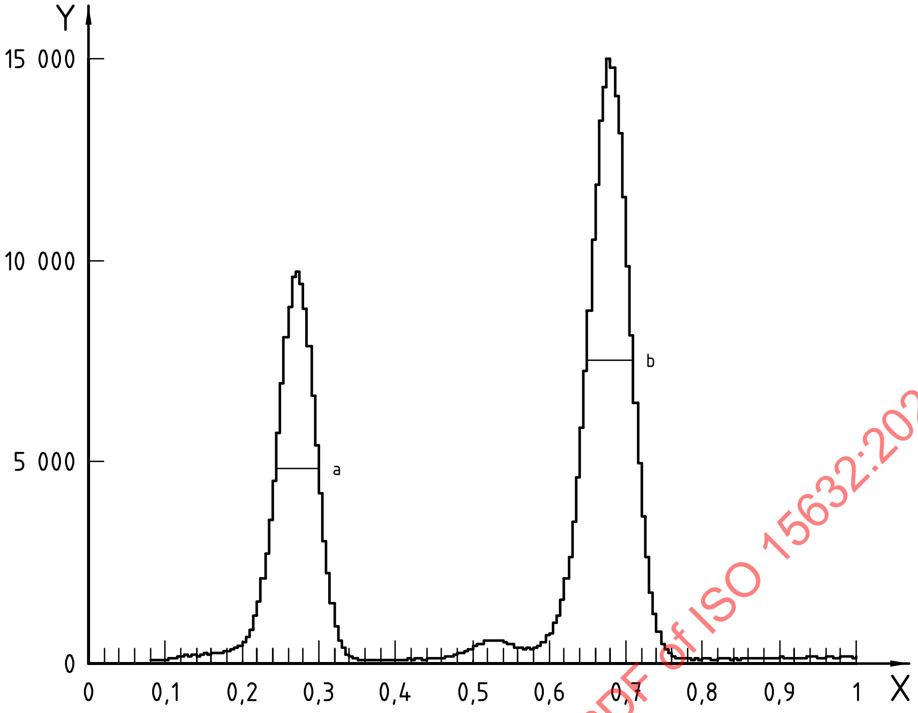
Key

X energy, keV

Y intensity, counts per channel × 10^4

[a] FWHM: 127,2 eV.

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



Key

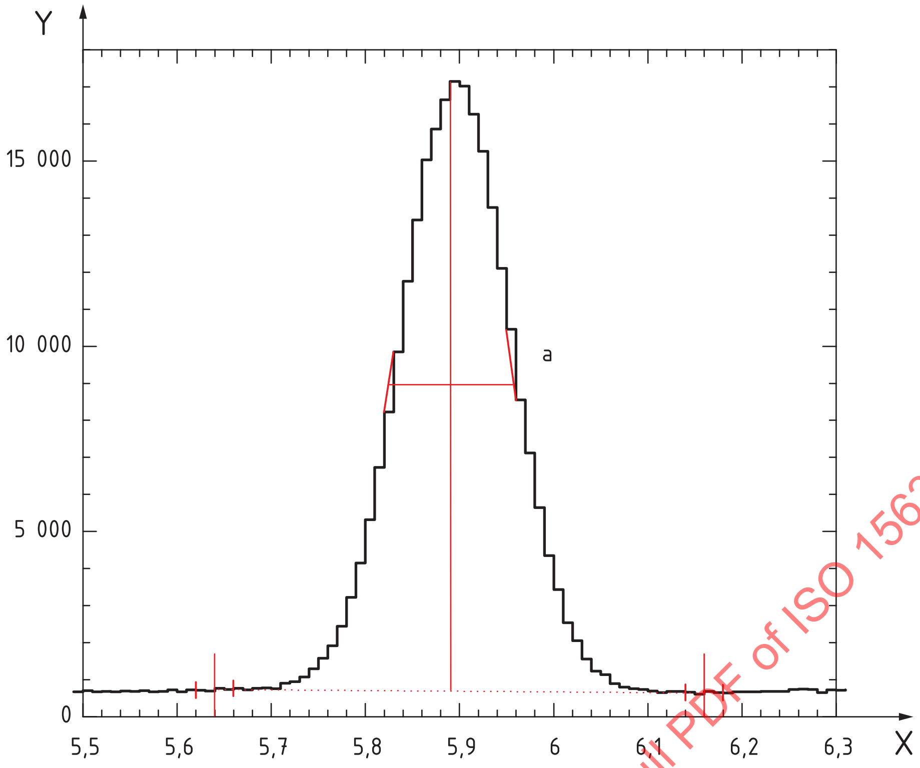
X energy, keV

Y intensity, counts per channel

[a] FWHM: 56,0 eV

[b] FWHM: 61,1 eV

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



Key

X energy, keV

Y intensity, counts per channel

[a] FWHM = 139,2 eV

Figure A.3 — Manganese K*α* peak from a 15 keV spectrum of a polished manganese specimen showing the linear background interpolated as in A.5 and the FWHM calculated as in A.6.