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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

Reference number
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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 3
4.3 Dead time 3
4.4 Peak-to-background ratio 4
4.5 Energy dependence of instrumental detection efficiency 4
5 Check of further performance parameters 4
5.1 General 4
5.2 Stability of the energy scale and resolution 4
5.3 Pile-up effects 4
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) Determination of the L/K ratio as a measure for the energy dependence of the instrumental detection efficiency 9
Bibliography 11

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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).

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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.

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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

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

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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

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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

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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 Subclause 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 intensity 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 Annex A, Clause 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 may 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°.

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, Clause 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. Additionally to this, 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

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(below 1 000 counts/s) on a reference material 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, one new material and new procedures are described in Reference [8].

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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, Teflon®) 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 $\leq$ 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 intensities in a PTFE spectrum enable 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 intensities 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

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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 [5] or [6]). 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 intensity. 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 may 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 Clause A.6 after a background subtraction as described in Clause A.5. The corresponding beam energy was 15 keV, the channel width 10 eV and TOA = 35°.

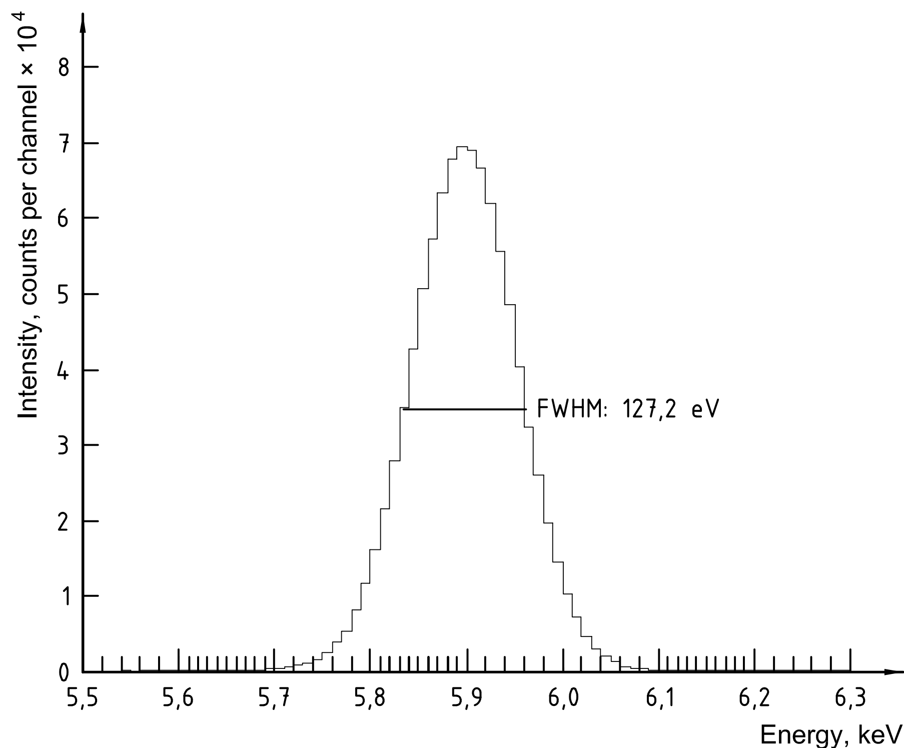

Figure A.1 — Manganese Kα spectrum emitted from an ^{55}Fe source and calculated FWHM

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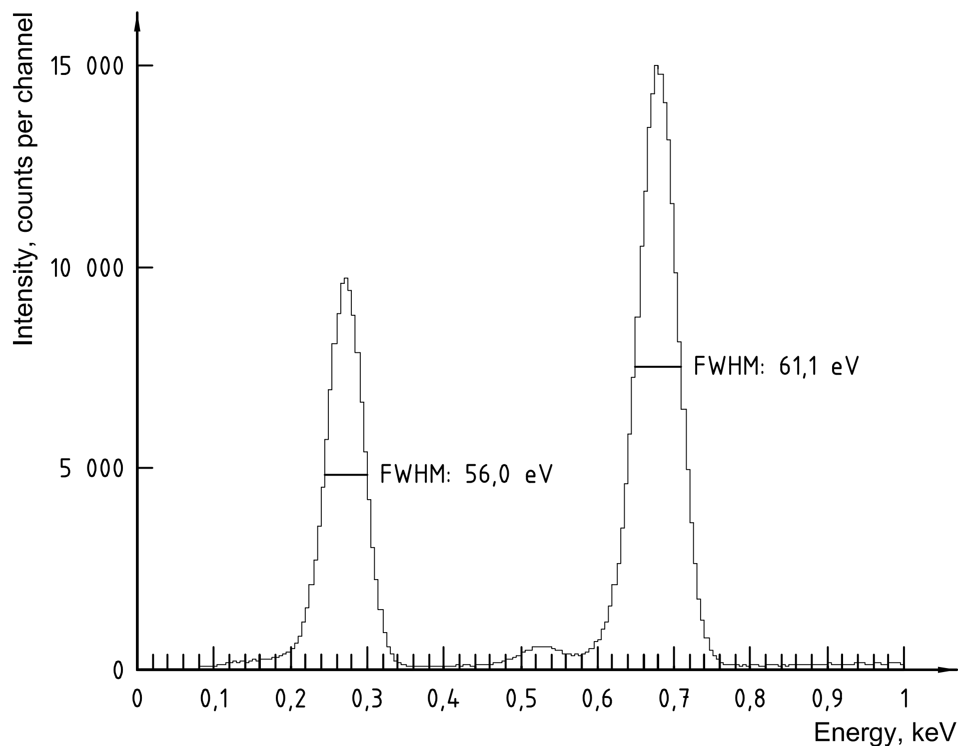

Figure A.2 — 10 keV spectrum from a PTFE specimen and calculated FWHMs for the carbon and fluorine K-lines

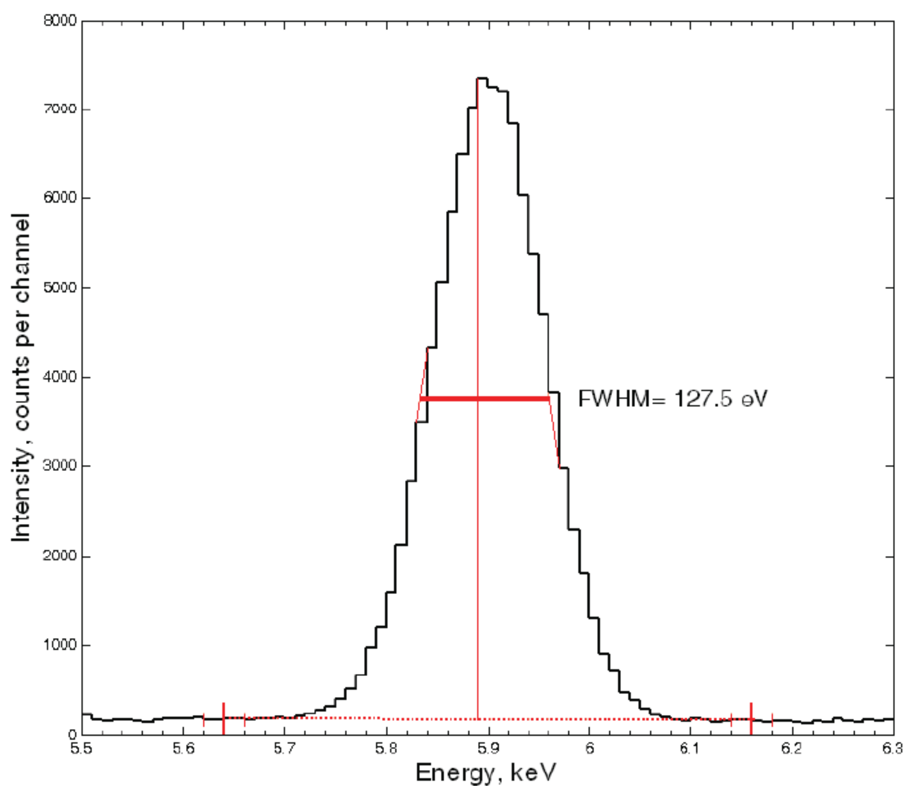

Figure A.3 — Manganese Kα peak from a 15 keV spectrum of a polished manganese specimen showing the linear background interpolated as in Clause A.5 and the FWHM calculated as in Clause A.6.

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Annex B
(normative)

Determination of the L/K ratio as a measure for the energy dependence of the instrumental detection efficiency

B.1 Specimens

Flat, polished and sufficiently cleaned nickel or copper specimens shall be used.

B.2 Measurement conditions

— Adjust the beam energy to 20 keV.

— Mount the specimen to effect perpendicular incidence of the electron beam as well as to effect a TOA of 35°.

— Peak intensities shall exceed 10 000 counts.

B.3 Calculation of L/K ratio

For the calculation of the L/K ratio, the following procedure shall be applied: Determine the net peak areas for the Ka peak and the L series by a linear background subtraction as described in Annex A. Calculate the L/K ratio as the area ratio.

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

B.4 Conversion of the L/K ratio for TOA ≠ 35°

In many scanning electron microscopes, the X-ray detector sees the specimen under a TOA ≠ 35°.

In these cases, the L/K ratio shall be converted to TOA ≠ 35° applying the absorption correction formalism of quantitative electron probe microanalysis. Figures B.1 and B.2 give nomograms for that conversion. The nomograms base on the XPP absorption correction model derived by Pouchou and Pichoir and their mass attenuation coefficients (MACs)[7].

Other formalisms for absorption correction may be applied as well, because in this case the differences are within the limits of error.

NOTE A flat specimen surface and the correct TOA are very important. A tilt of the specimen surface by a few degrees changes the TOA by up to the same amount. Establishing the correct TOA requires that the height of the specimen be properly chosen. The manufacturer of the microscope should provide this height. Furthermore, there is some uncertainty in attenuation coefficients for nickel and copper L-lines, which will make the nomogram less reliable for correcting measurements at the extremes where the TOA is far different from 35°.

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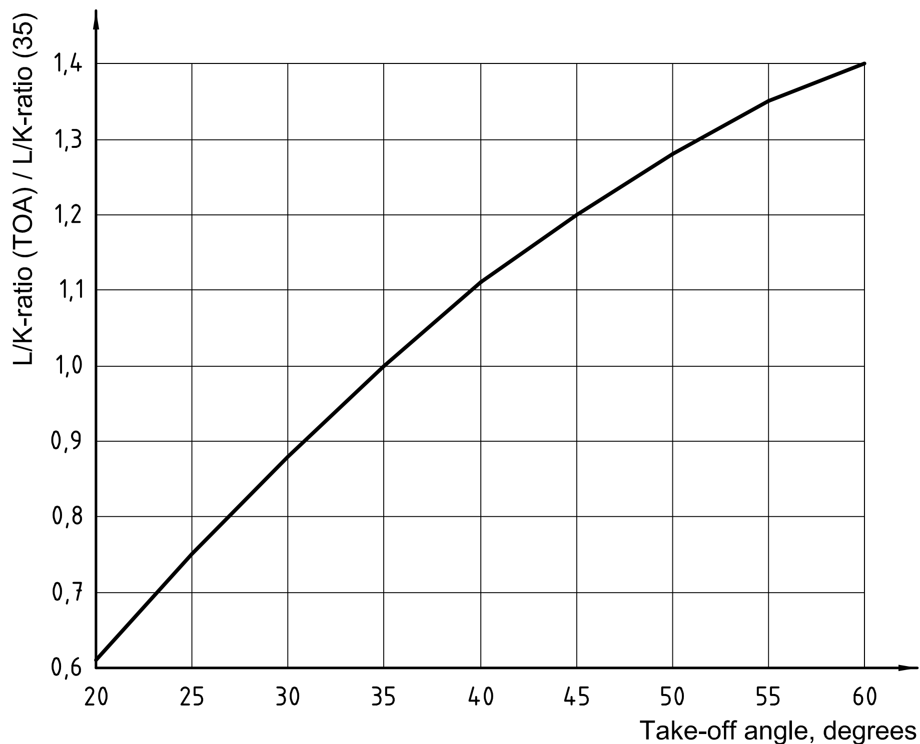

Figure B.1 — Nomogram for conversion to TOA = 35° in the case of nickel

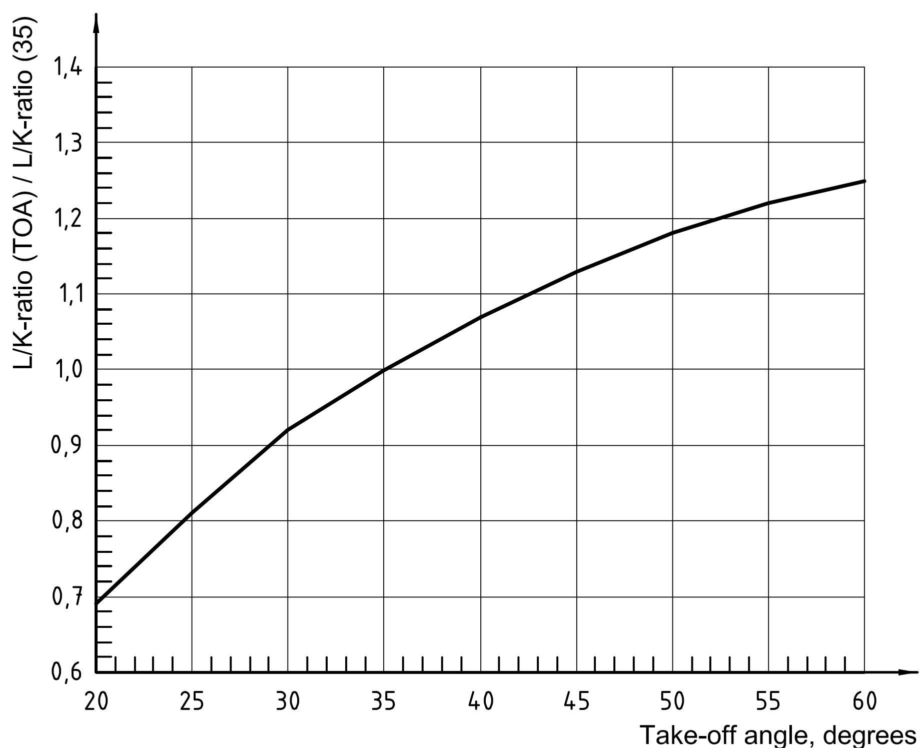

Figure B.2 — Nomogram for conversion to TOA = 35° in the case of copper

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