INTERNATIONAL
STANDARD

ISO
23833

Second edition
2013-04-15

Microbeam analysis — Electron probe microanalysis (EPMA) — Vocabulary

Analyse par microfaisceaux — Analyse par microsonde électronique (microsonde de Castaing) — Vocabulaire

Reference number
ISO 23833:2013(E)

COPYRIGHT PROTECTED DOCUMENT

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.org
Web www.iso.org

Published in Switzerland

Contents

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 23833 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 1, *Terminology*.

The European Microbeam Analysis Society (EMAS) made contributions to the preparation of the document.

This second edition of ISO 23833 cancels and replaces the first edition (ISO 23833:2006), of which it constitutes a minor revision.

This International Standard has a cross-reference relationship with the surface chemical analysis vocabulary prepared by ISO/TC 201 (ISO 18115-1:2010).

Introduction

Electron probe X-ray microanalysis (EPMA) is a modern technique used to qualitatively determine and quantitatively measure the elemental composition of solid materials, including metal alloys, ceramics, glasses, minerals, polymers, powders, etc., on a spatial scale of approximately one micrometer laterally and in depth. EPMA is based on the physical mechanism of electron-stimulated X-ray emission and X-ray spectrometry.

As a major sub-field of microbeam analysis (MBA), the EPMA technique is widely applied in diverse business sectors (high-tech industries, basic industries, metallurgy and geology, biology and medicine, environmental protection, trade, etc.) and has a wide business environment for standardization.

Standardization of terminology in a technical field is one of the basic prerequisites for development of standards on other aspects of that field.

This International Standard is relevant to the need for an EPMA vocabulary that contains consistent definitions of terms as they are used in the practice of electron probe microanalysis by the international scientific and engineering communities that employ the technique.

Microbeam analysis — Electron probe microanalysis (EPMA) — Vocabulary

1 Scope

This International Standard defines terms used in the practices of electron probe microanalysis (EPMA). It covers both general and specific concepts classified according to their hierarchy in a systematic order.

This International Standard is applicable to all standardization documents relevant to the practices of EPMA. In addition, some parts of this International Standard are applicable to those documents relevant to the practices of related fields (SEM, AEM, EDX, etc.) for definition of those terms common to them.

2 Abbreviated terms

BSE	backscattered electron
CRM	certified reference material
EDS	energy-dispersive spectrometer
EDX	energy-dispersive X-ray spectrometry
EPMA	electron probe microanalysis or electron probe microanalyser
eV	electronvolt
keV	kiloelectronvolt
RM	reference material
SE	secondary electron
SEM	scanning electron microscope
WDS	wavelength-dispersive spectrometer
WDX	wavelength-dispersive X-ray spectrometry

3 Definitions of general terms used in electron probe microanalysis

3.1
electron probe microanalysis
EPMA
technique of spatially-resolved elemental analysis based upon electron-excited X-ray spectrometry with a focussed electron probe and an electron interaction volume with micrometer to sub-micrometer dimensions

3.1.1
qualitative EPMA
procedure in EPMA leading to the identification of the elements present in the electron-excited interaction volume by a systematic method for the recognition and assignment of X-ray spectral peaks to specific elements

3.1.2
quantitative EPMA
procedure leading to the assignment of numerical values to represent the concentrations of elemental constituents that had been previously identified in the electron-excited interaction volume during the qualitative analysis phase in EPMA

Note 1 to entry: Quantitative analysis can be accomplished by comparing the unknown X-ray peak intensities to X-ray peak intensities measured under the same conditions using reference material(s) or by calculating the concentration from first principles (also known as standardless analysis).

3.2
electron probe microanalyser
instrument for carrying out electron-excited X-ray microanalysis

Note 1 to entry: This instrument is usually equipped with more than one wavelength spectrometer and an optical microscope for precise specimen placement.

3.3
electron scattering
deviation in trajectory and/or kinetic energy suffered by an impinging energetic beam electron as a result of an interaction with a specimen atom or electron

3.3.1
angle of scattering
scattering angle
angle between the direction of the incident particle or photon and the direction that the particle or photon is traveling after scattering

[ISO 18115-1:2010]

3.3.2
backscattering
escape of beam electrons from the specimen following sufficient scattering events to cause the trajectories to intersect the entrance surface of the specimen

3.3.2.1
backscatter coefficient
η
fraction of beam electrons that are backscattered, given by the equation

$$\eta = n(\mathrm{BS})/n(\mathrm{B})$$

where $n(\mathrm{B})$ is the number of incident electrons and $n(\mathrm{BS})$ is the number of backscattered electrons

3.3.2.2
backscattered electron
electron ejected from the entrance surface of the specimen by a backscattering process

3.3.2.3
backscattered electron angular distribution
distribution of backscattered electrons as a function of the angle relative to the surface normal

3.3.2.4
backscattered electron depth distribution
distribution of backscattered electrons as a function of the maximum depth into the specimen reached before travelling back to the entrance surface to exit the specimen

3.3.3
continuous energy loss approximation
mathematical description of energy loss by fast electrons propagating through matter in which all of the discrete inelastic processes are approximated as a single continuous energy loss process

Note 1 to entry: Also known as the continuous slowing-down approximation (CSDA).

3.3.4
elastic scattering
interaction of an energetic electron from the impinging beam and a specimen atom during which the electron's energy remains essentially unaltered but its trajectory is changed by an angle from 0 up to π rad (180°) with an average of approximately 0,1 rad

3.3.5
inelastic scattering
interaction of an energetic electron from the impinging beam and a specimen atom or electron during which kinetic energy is lost to the specimen by various mechanisms (secondary electron generation, bremsstrahlung, inner shell ionization, plasmon and photon excitation)

Note 1 to entry: For inelastic scattering, the electron trajectory is modified by a small angle, generally less than 0,01 rad.

3.3.6
scattering cross-section
number of scattering events per unit area
mathematical description of the probability of a scattering event (elastic or inelastic)

Note 1 to entry: See *ionization cross-section* (3.4.4).

Note 2 to entry: Scattering cross-section is usually expressed simply as an area, in cm^2. The number of scattering events per unit area is expressed in events/(atoms/cm^2).

3.3.7
scattering effect
measurable physical phenomenon, such as electron backscattering or loss of X-ray generation, that results from modification of the trajectory and/or kinetic energy of an impinging energetic beam electron by scattering processes in the specimen

3.3.8
secondary electron
Electron emitted from the specimen as a result of inelastic scattering of the primary beam electron by loosely bound valence-level electrons of the specimen

Note 1 to entry: Secondary electrons have conventionally an energy less than 50 eV.

3.4
X-ray
photon of electromagnetic radiation created by fluorescence of an inner shell electron vacancy or by deceleration of an energetic electron in the Coulombic field of an atom

3.4.1
characteristic X-ray
photon of electromagnetic radiation created by the relaxation of an excited atomic state created by inner shell ionization following inelastic scattering of an energetic electron or ion, or by absorption of an X-ray photon

3.4.2
continuous X-ray
photon of electromagnetic radiation created by deceleration (an inelastic scattering mechanism) of an energetic electron in the Coulombic field of an atom

3.4.3
fluorescence yield
ω
fraction of inner shell ionization events that give rise to characteristic X-ray emission during subsequent de-excitation

Note 1 to entry: The fluorescence yield is independent of the method of ionization.

3.4.4
ionization cross-section
number of ionization events per unit area
mathematical description of the probability of ionizing an atom by removing an atomic electron from a particular bound electron shell into the unbound vacuum, or continuum, energy level or state

Note 1 to entry: See *scattering cross-section* (3.3.6).

Note 2 to entry: Ionization cross-section is usually expressed simply as an area, in cm^2 or in barns (10^{-24} cm^2). Ionization event probability is expressed in events/(atoms/cm^2).

Note 3 to entry: Ionization cross-section is usually denoted by Q, which is defined by the mathematical expression $\mathrm{d}n = Q(N\rho/A)\mathrm{d}x$ where $\mathrm{d}n$ is the number of ionization events which occur in each increment $\mathrm{d}x$ of electron path and $N\rho/A$ is the number of atoms per unit volume.

3.4.5
ionization energy
critical excitation energy
edge energy
minimum energy required to ionize an atomic electron, i.e. to remove a bound electron from a shell (K, L, etc.) to (as a minimum) the continuum of energy states in a solid

Note 1 to entry: Ionization energy units are eV or keV.

3.4.6
J-value
mean ionization energy, a critical parameter in mathematical descriptions of the continuous energy loss approximation

3.4.7
stopping power
$\mathrm{d}E/\mathrm{d}s$
rate of energy loss experienced by a primary electron (from all inelastic scattering processes) with distance travelled in the specimen

Note 1 to entry: Stopping power is expressed as energy loss/unit distance (e.g. eV/nm).

3.4.8
X-ray fluorescence effect
secondary fluorescence
photoelectric absorption of an X-ray (characteristic or bremsstrahlung) by an atom, resulting in an excited atomic state which will de-excite with electron shell transitions and subsequent emission of an Auger electron or the characteristic X-ray of the absorbing atom

3.4.9
X-ray generation
generation of X-rays in the specimen under the stimulation of an incident beam of radiation (electrons, ions or photons)

Note 1 to entry: X-rays can be generated through the ionization of inner atomic shells (characteristic X-rays) or through the "braking radiation" (bremsstrahlung) process (continuum or white radiation).

3.5

X-ray absorption

attenuation of the intensity of X-rays passing through matter, arising primarily from photoelectric absorption for X-ray energies appropriate to EPMA

3.5.1

absorption edge

critical ionization energy for a particular shell or subshell of an atom species

Note 1 to entry: Absorption edges are detected in spectra as discontinuities in the X-ray continuum (bremsstrahlung) background due to a sharp change in the X-ray mass absorption coefficient at the edge.

3.5.2

absorption factor

$f(\chi)$

ratio of emitted X-ray intensity to the generated intensity in a specific direction towards the X-ray detector

3.5.3

depth distribution function

$\phi(\rho z)$

function which describes the distribution of generated X-rays as a function of depth below the specimen surface

Note 1 to entry: ρz is expressed in units of density × thickness (depth).

3.5.4

jump ratio

<EPMA, TXRF> ratio of the X-ray absorption coefficient at an energy just above an absorption edge to that at an energy just below the edge

Note 1 to entry: X-ray absorption spectra can have complex shapes for X-ray energies in the vicinity of photoionization thresholds, and a well-defined edge is not always observed at the threshold.

[ISO 18115-1:2010]

3.5.5

mass absorption coefficient

μ_l/ρ

material parameter that relates the loss of X-ray intensity due only to photoelectric absorption during passage through matter, where μ_l is the linear absorption coefficient and ρ is the material density

Note 1 to entry: Its dimensions are area/mass (e.g. cm^2/g). It is sometimes referred to as the mass absorption cross section.

3.5.6

mass attenuation coefficient

μ/ρ

material parameter that relates the loss of X-ray intensity due to all absorption and scattering processes during passage through matter, given by the equation

$$I/I_0 = \exp[-(\mu/\rho)\rho s]$$

where I_0 is the incident X-ray intensity, I is the intensity remaining after passage through a distance s of the material, μ is the linear attenuation coefficient, ρ is the material density, s is the path thickness in cm

Note 1 to entry: Its dimensions are area/mass (e.g. cm^2/g). It is sometimes referred to as the mass attenuation cross section.

3.5.7
mass-depth distance
ρz
description of a dimension in terms of the product of linear distance (cm) and density (g/cm^3)

Note 1 to entry: It is expressed in grams per square centimetre.

3.5.8
X-ray take-off angle
ψ
angle between the specimen surface and the central axis of the X-ray spectrometer

3.6
X-ray spectrum
plot of the X-ray photon abundance as a function of the energy or wavelength

Note 1 to entry: Most often, the number of photons/unit time is plotted, but other measures of the intensity are possible.

3.6.1
characteristic X-ray spectrum
X-ray peaks or lines associated with a particular atom species and generated as a result of inner shell ionization (caused by an energetic electron, ion, or photon) followed by inter-shell electron transitions and emission of excess energy as a photon of electromagnetic radiation

3.6.1.1
family of X-ray lines
systematic set of characteristic X-rays produced as a result of all possible routes of ionization of a particular shell/subshell and the subsequent inter-shell electron transitions that occur

3.6.1.2
K-line
one of the characteristic line emissions following K-shell ionization

3.6.1.3
K-spectrum
series of characteristic X-rays arising from ionization of an electron bound in the K-shell of an atom

3.6.1.4
L-line
one of the characteristic line emissions following L-shell ionization

3.6.1.5
L-spectrum
series of characteristic X-rays arising from ionization of an electron bound in the L-shell of an atom

3.6.1.6
M-line
one of the characteristic line emissions following M-shell ionization

3.6.1.7
M-spectrum
series of characteristic X-rays arising from ionization of an electron bound in the M-shell of an atom

3.6.1.8
satellite line
low relative abundance feature near a characteristic peak that can result from any of a variety of processes or situations: “forbidden” transitions, “non-diagram” lines and doubly-ionized atoms

3.6.1.9
X-ray line table
table of fundamental parameters for qualitative analysis by EPMA

Note 1 to entry: The X-ray line table for qualitative analysis by EPMA contains the wavelengths (or energies) of K-, L- and M-lines of the elements to be analyzed. It may also include their relative intensities, the full width at half maximum (FWHM) of each peak, the names of the diffraction crystals used and their interplanar spacings, and the wavelengths (or energies) of satellite lines.

3.6.2
continuous X-ray spectrum
continuum
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 E_0 (Duane-Hunt limit)

3.6.2.1
Duane-Hunt limit
beam energy
E_0
maximum photon energy found in the continuous X-ray spectrum corresponding to complete conversion of an incident electron's energy in a single event

3.6.2.2
Kramers' law
experimental observation of the intensity of the X-ray continuum as a function of the incident electron beam energy, the photon energy and the atomic number of the specimen

4 Definition of terms used to describe EPMA instrumentation

4.1
electron optics
system of electrostatic and electromagnetic lenses that make up the beam forming system

4.1.1
condenser lens
first demagnification lens, i.e. in a two-lens system the lens located immediately below the electron gun

Note 1 to entry: Its principal function is to define the current in the beam.

4.1.1.1
condenser lens aperture
fixed diaphragm with an axial opening that defines the angular transmission of the condenser lens

4.1.1.2
condenser lens current
externally-adjustable current flowing in the coil of the condenser lens which generates the magnetic field that provides focussing action

4.1.2
demagnification
numerical value by which the diameter a beam exiting a lens is reduced in size, relative to the incident diameter, by the focussing action

Note 1 to entry: Equal to p/q, where p is the distance from the object to the center of the lens, and q is the distance from the image to the center of the lens

4.1.3
electron gun
source of electrons in EPMA, consisting of an emitter (heated tungsten or LaB_6 filament, or cold or thermally-assisted field emitter tip) and electrostatic extraction and accelerating optics

4.1.3.1
electron gun brightness
β
current per unit area per unit solid angle in the beam, given by the equation

$$\beta = 4I/(\pi^2 d^2 \alpha^2)$$

where I is the current (A), d is the beam diameter (cm) and α is the beam divergence (rad)

4.1.3.2
electron gun current
beam current
current in the beam as it exits the final element of the electron gun assembly

4.1.4
objective lens
electron probe forming lens, i.e. in a two-lens system the lens located immediately above the specimen and below the condenser lens

Note 1 to entry: The principal function of the lens is to focus the final probe.

4.1.4.1
objective lens aberration
any of various lens defects (spherical aberration, chromatic aberration, diffraction, etc.) that degrade the focussed probe

4.1.4.2
objective lens aperture
diaphragm with an axial opening placed above the objective lens that defines the angular divergence of the focussed beam and that reduces spherical aberrations

Note 1 to entry: Generally, a selection of apertures is provided on an externally adjustable slider.

4.1.4.3
objective lens current
externally-adjustable current flowing in the coil of the objective lens which generates the magnetic field that provides focussing action

4.1.5
working distance
distance between the lower surface of the polepiece of the objective lens and the specimen surface

Note 1 to entry: In the past, this distance was defined as the distance between the principal plane of the objective lens and the plane containing the specimen surface.

4.2
electron beam
⟨electron optics⟩ electron beam prior to focussing by the electron optical system onto the specimen

4.2.1
beam current
electron current contained within the electron beam

Note 1 to entry: See *electron beam* (4.2).

4.2.1.1

beam current density

beam current normalized by the beam area

Note 1 to entry: It is expressed in amperes per square centimetre.

4.2.1.2

beam current stability

variation with time of the beam current

Note 1 to entry: See *beam current* (4.2.1).

4.2.1.3

beam diameter

diameter of the electron beam containing a specified fraction of the total current, for example 0,8 (80 %) of the total

Note 1 to entry: See *electron beam* (4.2).

Note 2 to entry: The beam diameter can be measured as the full peak width at half maximum (FWHM) of the beam current across the diameter of the cross-section perpendicular to the beam.

4.2.2

Faraday cup

special target for capturing the incident beam current with negligible loss of current due to backscattering, secondary electron emission, or photoemission

Note 1 to entry: The Faraday cup typically consists of a blind hole drilled into a carbon block covered with a metal aperture with an opening of approximately 100 µm in diameter.

4.3

electron probe

⟨electron optics⟩ electron beam focussed by the electron optical system onto the specimen

Note 1 to entry: ⟨instrumentation⟩ See *electron probe microanalyser* (3.2).

Note 2 to entry: Typical beam diameters at the specimen plane range from nanometers to micrometers.

4.3.1

probe current

electron current contained within the electron probe

Note 1 to entry: See *electron probe* (4.3).

4.3.1.1

probe current density

probe current normalized by the probe area

Note 1 to entry: It is expressed in amperes per square centimetre.

4.3.1.2

probe current stability

variation with time of the probe current

4.3.2

probe diameter

diameter of the probe containing a specified fraction of the total current, for example 0,8 (80 %) of the total

Note 1 to entry: The probe diameter can be measured as the full peak width at half maximum (FWHM) of the probe current across the diameter of the cross-section perpendicular to the probe.

4.3.3
specimen stage
component of an electron probe X-ray microanalyser that operates to translate the specimen, usually in at least two dimensions (along *x*- and *y*-axes perpendicular to the beam)

Note 1 to entry: Movement in three dimensions (*x*-, *y*- and *z*-axes, where *z*- is parallel to the beam) and specimen tilt and rotation may also be provided.

4.3.4
absorbed current
specimen current
current flowing between the conducting specimen and ground, representing the difference between the beam current injected into the specimen and the current emitted as backscattered electrons and secondary electrons

4.4
electron scanning image
scanning image
image constructed by moving an incident electron beam to discrete locations on a specimen, collecting information generated therefrom (secondary electron signal, backscattered electron signal, X-rays, etc.) and displaying this signal at a corresponding point in a visual display such as a cathode ray tube or storing it as a numerical value at a computer memory location

4.4.1
area scanning
beam scanning action to cover a specific area of the specimen during the recording of an X-ray map

4.4.2
backscattered electron image
scanning electron beam image in which a signal is derived from a dedicated backscattered electron detector (e.g. passive scintillator, solid-state diode, channel plate or negatively-biased Everhart-Thornley detector)

4.4.3
compositional mapping
X-ray mapping in which all significant corrections are performed at each picture element to produce a map in which the quantitative concentration of an element is used to select the grey or colour scale for the final display

4.4.4
dot mapping
form of display for X-ray maps in which the X-ray intensity is used to select a two-state display, off (black) or on (white)

Note 1 to entry: Dot maps are purely qualitative in nature.

4.4.5
edge effect
phenomenon in which a secondary electron image appears bright, especially at a protuberance or the edge of an uneven part of the specimen, owing to an increase in the number of secondary electrons emitted from such an area

4.4.6
image contrast
C
difference in signal between two arbitrarily chosen points (P_1, P_2) of interest in the image field, normalized by the maximum possible signal available under the particular operating conditions

Note 1 to entry: $C=|S_2-S_1|/S_{max}$ $(0 < C < 1)$, where S_2 and S_1 are the signals from the two arbitrarily chosen points (P_1, P_2), respectively, and S_{max} is the maximum possible signal available under the particular operating conditions.

4.4.7

image magnification

ratio of the linear dimension *L* of the scan display (e.g. along an edge) to the corresponding length *l* of the scan field on the specimen

$$M = L/l$$

Note 1 to entry: Historically, the magnification was related to the size of the roll film used to photograph the CRT image. For digital images displayable on screens of various sizes, it is more appropriate to specify the size of the scan field.

4.4.8

image resolution

minimum spacing at which two features of an image can be recognized as distinct and separate

Note 1 to entry: Image resolution can be measured as the minimum distance between two separable details displayed by the image under the operating conditions chosen.

4.4.9

line analysis

analysis obtained when the electron probe is stepped across a portion of the specimen in a line

Note 1 to entry: The analytical points along the line analysis should be equidistant from each other and have the same electron probe dwell time.

4.4.10

point analysis

analysis obtained when the electron probe is placed at a single location and held there for the duration of the spectrometric measurement

4.4.11

secondary electron image

scanning electron beam image in which the signal is derived from a detector that selectively measures secondary electrons (electrons having less than 50 eV) and is not directly sensitive to backscattered electrons

4.4.12

absorbed current image

specimen current image

scanning electron beam image prepared with a signal derived from the current absorbed by the specimen and conducted to ground

Note 1 to entry: A specimen current amplifier in series with the specimen is used to measure the specimen current.

4.4.13

X-ray image

scanning electron beam image in which the output X-ray count rate from an energy-dispersive or wavelength-dispersive X-ray spectrometer is used to modulate the brightness of the displayed pixel corresponding to the beam position on the specimen

Note 1 to entry: The X-ray signal typically represents a narrow range of energy ΔE or wavelength range $\Delta\lambda$ that arises from a particular elemental species, along with the associated continuum in that range.

4.5

X-ray detection

measurement of an X-ray event employing an X-ray detection system

4.5.1

coincidence losses

⟨in serial processing of X-ray events⟩ the loss of X-rays incident on the detector due to the restriction of the pulse measurement circuitry to processing one pulse at a time, resulting in dead time

4.5.2
counter energy resolution
width of the distribution of counting pulses produced by monochromatic X-radiation in a proportional counter

4.5.3
counting pulse
charge collected in a proportional counter from the interaction of a single X-ray photon

4.5.4
counting statistics
mathematical measures of the distribution of stochastic events such as electron-excited X-rays

4.5.5
counting system
pulse counting system
electronic system used to detect and measure counting pulses from a gas proportional counter or an energy-dispersive X-ray detector

4.5.6
dead time
τ
time that the system is unable to record a photon measurement because it is busy processing a previous event and frequently expressed as a percentage of the total time

Note 1 to entry: See *live time* (4.5.11).

4.5.7
discriminator
⟨pulse processing circuitry of a wavelength-dispersive X-ray spectrometer⟩ a circuit whose function is to define a minimum or maximum pulse voltage for acceptance

4.5.8
discriminator threshold
minimum voltage value an amplified pulse must reach for acceptance by the pulse counting system

4.5.9
discriminator window
voltage range above the discriminator threshold that a pulse must fall within for acceptance to the pulse counting system

4.5.10
gas multiplication
method of amplification of an X-ray signal in a proportional counter

Note 1 to entry: In a proportional counter, X-rays are detected following photoelectric absorption by a gas atom and subsequent ejection of an electron, which can scatter inelastically with other atoms and ionize them with the ejection of low-energy electrons. These low-energy electrons are accelerated in the applied field, and can themselves cause ionizations, multiplying the original deposited charge.

4.5.11
live time
time that the pulse measurement circuitry is available for the detection of X-ray photons

Note 1 to entry: See *dead time* (4.5.6).

Note 2 to entry: Live time = real time for analysis minus dead time. Real time is the time that would be measured with a conventional clock. For X-ray acquisition, the real time always exceeds the live time.

4.5.12
proportional counter
gas-filled chamber for X-ray detection in which the X-ray energy is transferred to a photoelectron which then scatters inelastically

Note 1 to entry: There is a range of accelerating voltages for which, when the accelerating voltage is applied to a central collection wire, the charge collected will be proportional to the photon energy.

4.5.13
pulse height distribution
the statistical distribution of counting pulses produced from photons of the same energy captured in a proportional counter

4.6
X-ray spectrometry
method in which an X-ray spectrometer is used to measure the energy or wavelength distribution of X-ray photons

4.6.1
characteristic peak
localized feature in an X-ray spectrum with higher intensity than the general level of the background radiation that is associated with a specific atomic species

Note 1 to entry: Characteristic peaks are produced as a result of a sequence of inner shell ionization followed by inter-shell electron transitions and electromagnetic radiation emission.

Note 2 to entry: The natural peak width in energy units is approximately 1 eV to 3 eV.

4.6.2
detector
device for detecting an X-ray photon and/or measuring the energy of that photon

Note 1 to entry: Examples of X-ray detectors include gas-filled proportional counters, microcalorimeters, intrinsic germanium (Ge) detectors, lithium-drifted silicon (Si-Li) detectors, and silicon drift detectors (SDD).

4.6.3
detector artefacts
feature introduced into the X-ray spectrum as a result of the detection process (e.g. coincidence peak, escape peak, higher-order peak) and not related to the true X-ray spectrum as emitted from the specimen

4.6.4
energy-dispersive X-ray spectrometer
EDS
device for determining X-ray intensity as a function of the energy of the radiation

4.6.4.1
dead layer
region of the semiconductor crystal immediately below the surface electrode which has a reduced efficiency, relative to the interior of the crystal, for the collection of charge deposited when an X-ray photon is absorbed

4.6.4.2
false peak
artefact peak
peak in an energy-dispersive X-ray spectrum caused by processes such as pulse coincidence (or pulse sum) and radiation loss from the detector (e.g. Si escape peak)

4.6.4.3

internal fluorescence peak

any spectral feature that is produced by fluorescence within the detector, rather than from excitation of the specimen

EXAMPLE The condition in a semiconductor EDS in which a photon is absorbed photoelectrically in the dead layer just below the front surface of the electrode, and the subsequent emission of the characteristic photon of the detector material (e.g. Si) appears as a contribution to the measured spectrum as an equivalent low-intensity source apparently arising in the specimen.

4.6.4.4

microcalorimeter EDS

energy-dispersive spectrometer that operates on the basis of photoelectric absorption and subsequent thermalization of the photon energy followed by thermometry to determine the temperature rise in the absorber

4.6.4.5

semiconductor EDS

energy-dispersive spectrometer that operates on the basis of photoelectric absorption in a semiconductor crystal and inelastic scattering of the photoelectron leading to charge deposition and measurement

4.6.4.5.1

intrinsic Ge EDS

energy-dispersive spectrometer that operates on the basis of photoelectric absorption in an intrinsic Ge crystal

4.6.4.5.2

lithium-drifted silicon detector EDS

Si-Li EDS

energy-dispersive spectrometer that operates on the basis of photoelectric absorption in an Li-doped Si crystal

4.6.4.5.3

silicon drift detector EDS

SDD EDS

energy-dispersive spectrometer that operates on the basis of photoabsorption in a silicon crystal in which electric fields transverse to the detector surface are used to guide electrons to the collection anode

4.6.4.6

escape peak

peak that occurs as a result of the loss of incident photon energy by fluorescence of the detector material such as Si in an Si-Li EDS detector

Note 1 to entry: Escape peaks occur at an energy equal to that of the incident characteristic peak minus the energy of the X-ray line(s) emitted by the element(s) in the detector (1,74 keV for Si).

Note 2 to entry: Escape peaks cannot occur below the critical excitation energy of the material, so in an Si-Li EDS detector an Si K escape peak does not occur for energies below 1,84 keV.

4.6.4.7

sum peak

artefact peak that arises from pulse coincidence effects which occur within the pulse pair resolution of the pileup inspection circuitry

Note 1 to entry: Sum peaks appear at energies corresponding to the sum of the energies of the photons which arrive at the detector essentially simultaneously.

4.6.4.8

system peak

artefact peak in an EDS spectrum caused by excitation of the specimen stage, the collimator, the chamber and the polepiece remote from the specimen due to an unfocussed component of the primary incident beam and/or backscattering from the specimen

4.6.5
energy-dispersive X-ray spectrometry
EDX
EDS
form of X-ray spectrometry in which the energy of the individual photons is measured and is used to build up a digital histogram representing the distribution of X-rays with energy

4.6.6
energy resolution
width of a peak as measured by an energy-dispersive X-ray spectrometer and expressed as the full peak width at half the maximum of the peak height

Note 1 to entry: For EDS, energy resolution is usually specified as the value for Mn Kα (5,890 keV) because this line can be obtained from a radioactive iron-55 isotope source.

Note 2 to entry: 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.

Note 3 to entry: Adapted from ISO 15632:2012.

4.6.7
full peak width at half maximum
FWHM
measure of the breadth of an X-ray peak in which the background is first removed to reveal the complete peak profile

Note 1 to entry: The maximum peak intensity is determined by fitting the full set of peak channels, and the width is determined at half the peak height.

4.6.8
in-hole spectrum
X-ray spectrum measured while the beam is placed within a Faraday cup/cage placed at the position where a specimen would normally undergo analysis

Note 1 to entry: This procedure is used to determine the extent of stray radiation in an electron beam instrument.

4.6.9
interfering peak
composite peak containing intensity contributions from more than one characteristic X-ray peak

4.6.10
natural line width
natural peak width
finite width of a characteristic X-ray peak which is a consequence, through the Heisenberg uncertainty principle, of the finite lifetime $\mathrm{d}t$ of the excited state of a photo-ionized atom resulting in an uncertainty $\mathrm{d}E$ in the photon energy

4.6.11
peak shift
change in the position of an X-ray peak, typically caused by chemical bonding effects

4.6.12
pulse height analysis
technique for separating X-rays of different energies that diffract at the same WDS setting

Note 1 to entry: In diffraction-based wavelength dispersive spectrometers, peak interference can occur when photons of two different energies but which have the same value of the product $n\lambda$ diffract at the same spectrometer setting ($n\lambda = 2d\sin\theta$). By operating the high-voltage gas ionization detector in the voltage regime where charge collected on the central wire is proportional to photon energy, the distribution of pulses coming from the detector will have a separate peak for each of the two photon energies entering the detector. Discrimination of the pulse peaks is achieved by placing a voltage acceptance window in the pulse processing amplification chain.

4.6.13
spectrometer efficiency
fraction of photons incident upon an X-ray spectrometer that are actually detected

4.6.14
wavelength-dispersive spectrometer
WDS
device for determining X-ray intensity as a function of the wavelength of the radiation, where separation is based upon Bragg's law, $n\lambda = 2d\sin\theta$, where n is an integer, λ is the X-ray wavelength, d is the spacing of the atom planes of the crystal or the repeated layers of a synthetic diffractor and θ is the angle at which constructive interference takes place

Note 1 to entry: X-rays diffracted at a particular angle are directed to a gas counter operated in the proportional response regime where the charge produced is proportional to the photon energy.

4.6.14.1
***d*-spacing**
spacing of the atomic planes in a crystal or the repeat distance in a synthetic multilayer structure used as a diffractor

4.6.14.2
defocussing
deviations of the X-ray source from the Rowland circle, due for example to the beam displacement caused by scanning

Note 1 to entry: The wavelength-dispersive spectrometer with curved diffracting crystals has optical focussing properties such that the position of the X-ray source, the diffractor and the detector must all lie on the Rowland circle. Defocussing may cause a loss in spectrometer transmission, especially if the motion is in the direction perpendicular to the diffractor thickness or out of the diffractor plane.

4.6.14.3
diffracting crystal
X-ray scattering element in a wavelength-dispersive X-ray spectrometer, consisting of a periodic array of atoms obtained either in a natural crystal or in a synthetic multilayer

4.6.14.4
high-order diffraction
peak observed with a wavelength-dispersive X-ray spectrometer for which the order n of the diffraction (in the Bragg equation $n\lambda = 2d\sin\theta$) is greater than 1

4.6.14.5
horizontal geometry
arrangement in WDS where the plane of the spectrometer (or Rowland circle) is perpendicular to the electron beam axis

4.6.14.6
inclined geometry
arrangement in WDS in which a significant fraction of the plane of the spectrometer (or Rowland circle) is projected onto the electron beam axis

4.6.14.7
Johann optics
wavelength-dispersive X-ray spectrometer in which the diffractor is bent to a radius twice that of the Rowland circle, achieving a "semifocussing" situation

4.6.14.8
Johansson optics
wavelength-dispersive X-ray spectrometer in which the diffractor is bent to a radius twice that of the Rowland circle and then its surface ground to the radius, achieving a fully focussing situation

4.6.14.9
layered synthetic microstructure
LSM
diffractor created artificially by alternating layers of a material (e.g. tungsten) with strong X-ray scattering power and a weak scattering material (e.g. carbon) to create large grating spacings for diffracting low-energy (long-wavelength) X-rays

4.6.14.10
LDE crystal
layered synthetic microstructure diffractor having a wide spacing to permit diffraction of long-wavelength (low-energy) X-rays

4.6.14.11
Rowland circle
circle of focus along which the X-ray source, the diffractor and the detector must all lie to satisfy the Bragg condition and obtain constructive interference

4.6.14.12
vertical geometry
arrangement in WDS where the plane of the spectrometer (or Rowland circle) is parallel to the electron beam axis z

4.6.15
wavelength-dispersive spectrometry
wavelength-dispersive X-ray spectrometry
WDX
method for examining the intensity of X-rays as a function of the photon wavelength

4.6.16
wavelength resolution
full peak width at half maximum of a peak in terms of wavelength ($\Delta\lambda$) obtained from a single X-ray transition by a WDS

4.6.17
wavelength resolving power
$\lambda/\Delta\lambda$
reciprocal of the ratio of the full peak width at half maximum of a peak in terms of wavelength ($\Delta\lambda$) obtained from a single X-ray transition by a WDS to the wavelength (λ) of that peak

5 Definitions of terms used in EPMA methodology

5.1
accelerating voltage
potential difference applied between the filament and the anode to accelerate the electrons emitted from the source

5.1.1
incident electron energy
kinetic energy of beam electrons as they enter the specimen

5.1.2
optimum accelerating potential
applied accelerating voltage providing the ideal beam energy for the measurement of a particular X-ray peak

Note 1 to entry: The optimum accelerating potential is based upon the need to establish sufficient overvoltage for excitation while avoiding too great a depth of production and subsequent loss of intensity due to self-absorption within the specimen.

5.1.3

overvoltage ratio

ratio of the incident beam energy to the critical excitation energy for a particular atomic shell

Note 1 to entry: This factor must be greater than unity for characteristic X-ray production to occur from that atomic shell.

5.2

detection limit

smallest amount of an element or compound that can be measured under specific analysis conditions

Note 1 to entry: By convention, the detection limit is often taken to correspond to the amount of material for which the total signal for that material minus the background signal is three times the standard deviation of the signal above the background signal. This convention may not be applicable to all measurements and, for a fuller discussion of detection limits, Reference [7] should be consulted.

Note 2 to entry: The detection limit may be expressed in many ways depending on the purpose. Examples of expressions are mass or weight fraction, atomic fraction, concentration, number of atoms and mass or weight.

Note 3 to entry: The detection limit will generally be different for different materials.

[ISO 18115-1:2010]

5.2.1

absolute detection limit

detection limit expressed as the equivalent mass of the element of interest within the interaction volume

5.2.2

analytical sensitivity

for the particular measurement conditions in use, ability to distinguish, for a given element, between two concentrations C and C’ that are nearly equal

Note 1 to entry: The sensitivity is limited by the background statistical fluctuations (concentration limit of detection) and described by an appropriate statistical expression.

5.2.3

background

non-characteristic component of an X-ray spectrum arising (ideally) from the X-ray continuum

5.2.3.1

background filtering

mathematical technique for spectrum processing, based upon a frequency filtering approach in which low-frequency components of the spectrum, which include the background, are selectively removed so that only the characteristic peaks remain

5.2.3.2

background modelling

mathematical technique for spectrum processing to extract characteristic peaks from background based upon a mathematical description of the background (e.g. Kramers’ law) in terms of beam energy, photon energy and specimen composition

5.2.3.3

linear interpolation

method of background subtraction in which a background channel is selected on each side of a peak, and a linear fit is performed between these channels to estimate the background under the peak

5.2.4

background statistical fluctuation

deviations with time in the production of continuum X-ray photons

5.2.5

relative detection limit

detection limit expressed as the concentration (mass fraction) of the element of interest

5.3

experimental data correction

correction applied to the experimental data in order to obtain a quantitative analysis

5.3.1

background correction

separation of the characteristic intensity from the underlying background intensity

Note 1 to entry: Background intensity includes X-ray continuum as well as background associated with artefacts.

5.3.2

beam current correction

correction of the measured X-ray intensity for deviations in the incident beam current during the counting period

Note 1 to entry: This correction typically assumes that the beam current drift is linear with time.

5.3.3

dead time correction

correction of the measured X-ray intensity for the loss of X-ray photons due to coincidence effects arising from the finite pulse processing time of the counting system

5.3.4

integrated beam current correction

correction of the measured X-ray intensity by the use of a periodic beam current monitoring system that records a measurement of the beam current throughout the period of an X-ray measurement

5.4

quantitative analysis

determination of the quantities of elemental constituents in an analysis volume of micrometer scale by electron-excited X-ray spectrometry

Note 1 to entry: The analyte may be elemental or compound in nature.

Note 2 to entry: The amount may be expressed, for example, as atomic or mass percent, atomic or mass fraction, moles or mass per unit volume, number (of atoms) per unit area or number (of atoms) per unit volume, as appropriate or as desired.

Note 3 to entry: The specimen material may be inhomogeneous so that a particular model structure may be assumed in the interpretation. Details of that model need to be stated.

5.4.1

empirical method

method of quantitative X-ray microanalysis, based upon the use of the hyperbolic relationship between the k-value and the mass concentration c

Note 1 to entry: See *intensity ratio* (5.4.4).

5.4.1.1

alpha factor

alpha coefficient

α

constant in the hyperbolic equation between k and c in the empirical method

5.4.1.2

calibration curve

graphical representation of the analytical signal as a function of the concentration of the analyte, generally determined at two or more fixed points corresponding to independently known reference materials

5.4.2

error

natural deviation from the true value in a measured quantity arising from (1) random counting fluctuations in a time-distributed phenomenon (e.g. X-ray photons) and (2) systematic deviations from the true value introduced during application of calculated correction factors (e.g. ZAF matrix correction factors) to convert the measured quantity (e.g. X-ray photons) to a different dimension (e.g. concentration)

5.4.2.1

confidence level

range of analytical error expected to contain the true value with a stated uncertainty as estimated from a statistical model of the measurement process

5.4.2.2

error distribution

range of errors associated with a measured quantity

5.4.2.3

error estimation

application of statistical models to a measured quantity to develop an estimate of the error associated with the measurement

5.4.2.4

error histogram

distribution of errors in a measured quantity, plotted as a histogram of frequency (on the vertical axis) versus the magnitude of the error value (on the horizontal axis)

5.4.2.5

error propagation

mathematical technique of estimating the impact of uncertainty in one or more parameters of a correction scheme to determine the error impact on the final calculated value

5.4.2.6

relative error

accuracy

%RE

method of expressing measurement error in which

$$\%RE = [(\text{measured value} - \text{true value})/\text{true value}] \times 100\ \%$$

Note 1 to entry: In this definition, overestimates are positive-going errors, and underestimates are negative-going errors.

5.4.3

Hall method

method of quantitative X-ray microanalysis typically applied to biological specimens which attempts to compensate for electron-beam-induced mass loss through the measurement of the X-ray continuum at a high photon energy (> 10 keV) as an internal standard

5.4.4

intensity ratio

k-value

k-ratio

K-value

ratio of the X-ray photon intensity I measured for a particular characteristic peak from the unknown specimen to the value measured for the same X-ray peak from a reference material of known composition under identical conditions of beam energy, spectrometer efficiency and electron dose and given by the equation

$$k = I_{\text{unknown}}/I_{\text{reference material}}$$

Note 1 to entry: Any changes in instrumental conditions between specimen and reference material, such as electron dose, must be taken into account when calculating the intensity ratio for quantitative determination of a given element.

5.4.5

normalization

mathematical procedure of dividing each concentration by the sum of the concentrations, so as to force the modified values to a sum of unity in quantitative X-ray microanalysis

5.4.6

standardless analysis

procedure for quantitative X-ray microanalysis in which the standard intensity in the *k*-value expression is supplied from purely physical calculations, referred to as "first principles" standardless analysis, or from a partial suite of reference materials, with empirical fitting or physical calculations being used to provide missing reference materials or to adjust the reference materials suite to a different set of experimental conditions

5.4.7

ZAF matrix correction

method of quantitative electron probe X-ray microanalysis in which separate multiplicative factors are used to correct for differences between the specimen and the reference material in electron scattering and retardation (Z), X-ray absorption (A) and secondary X-ray fluorescence (F)

Note 1 to entry: See *X-ray fluorescence effect* (3.4.8).

Note 2 to entry: The Z, A and F factors are derived from a combination of experimental measurements, empirical fits and theoretical calculations.

5.4.8

$\phi(\rho z)$ matrix correction

method of quantitative electron probe X-ray microanalysis in which correction factors are calculated from empirical equations developed from fits to experimental data of X-ray production as a function of depth, the so-called $\phi(\rho z)$ function

Note 1 to entry: The term is pronounced "phi rho zed".

5.5

quantitative analysis correction

correction applied to the elemental intensity ratios in order to obtain a quantitative elemental analysis

5.5.1

absorption correction

matrix correction arising from the loss of X-ray intensity for element A while the X-rays are propagating through the specimen in the direction of the X-ray spectrometer due to photoelectric absorption by all elements within the specimen

5.5.2

atomic number correction

matrix correction for the modification of the X-ray intensity for element A due to electron backscattering and stopping power, which are influenced by all elements in the analytical volume

5.5.3

backscattering correction

matrix correction which accounts for the loss of X-ray production due to backscattering of beam electrons (fraction and energy distribution)