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Microbeam analysis — Analytical electron microscopy — Calibration procedure of energy scale for elemental analysis by electron energy loss spectroscopy

Analyse par microfaisceaux — Microscope électronique analytique — Procédure d'étalonnage de l'échelle d'énergie pour l'analyse élémentaire par spectroscopie de perte d'énergie des électrons

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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*, Subcommittee SC 3, *Analytical electron microscopy*.

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

The electron energy loss spectroscopy (EELS) is an effective method for elemental analysis with high spatial resolution. In a transmission electron microscope (TEM) or a scanning TEM (STEM), incident electrons irradiating a sufficiently electron transparent sample, such as thin foil samples, are scattered by the atoms in the sample. Inelastically scattered electrons are analysed to identify elements in the sample by a spectrometer. All elements from hydrogen to uranium are identified based upon comparisons of the peak energies and shapes of the core-loss edges, with table of peak energies and spectra in handbooks for the different elements. To identify specific element core-loss peaks, calibration of the EEL spectrometer to an uncertainty of ±3 % is generally adequate for energy range 0 eV to 3 000 eV. This document details the procedure for the energy calibration intended for work at that level of accuracy.

Determination of a sample to be suitably electron transparent can be established by the EELS log ratio technique (t/λ) for specimen thickness. For further details on the determination of relative specimen thickness using the EELS log ratio technique (t/λ), see Reference [1].

Microbeam analysis — Analytical electron microscopy — Calibration procedure of energy scale for elemental analysis by electron energy loss spectroscopy

1 Scope

This document specifies a calibration procedure of the energy step and the energy scale for electron energy loss spectroscopy in (scanning) transmission electron microscopes to an uncertainty of ±3 % for the energy range 0 eV to 3 000 eV.

This document is intended for electron energy loss spectroscopy with transmitted electrons through sufficiently electron transparent samples, such as a thin foil sample, and is not designed for backscattered electrons from a bulk sample.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 15932, *Microbeam analysis — Analytical electron microscopy — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 15932 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
acceptance angle
γ
half of the angle formed by an entrance aperture to the EEL spectrometer

Note 1 to entry: See Figure 1.

3.2
channel
range of one pixel in the *parallel detector* (3.13)

3.3
collection angle
β
half of the angle formed by an objective aperture in a transmission electron microscope

Note 1 to entry: See Figure 1.

3.4
convergence angle
α
half of the angle of an electron beam focused by a condenser lens in a transmission electron microscope

Note 1 to entry: See Figure 1.

3.5
core-loss
energy loss of an electron in the beam caused by excitation of an inner shell electron

[SOURCE: ISO 15932:2013, 2.2.2.4]

3.6
core-loss edge onset
beginning position of a core-loss edge

Note 1 to entry: See Figure 3.

3.7
core-loss peak
peak maximum position of a core-loss edge

Note 1 to entry: See Figure 3.

3.8
EEL spectrometer
equipment of electrons energetically dispersed by electrostatic/electromagnetic elements and detected by:

— direct electron detectors in *serial detection* (3.17),

— scintillator and a photomultiplier in *serial detection* (3.17),

— a scintillator and a *photodiode array* (3.14) in parallel detection, and

— optical lens coupled with scintillator and CCD/CMOS in parallel detection

3.9
elastically scattered electron
electron scattering in which energy and momentum are conserved in the collision system

[SOURCE: ISO 15932:2013, 2.2.1]

3.10
energy step
energy (eV) per one *channel* (3.2) in a spectrometer

3.11
in-column EEL spectrometer
EEL spectrometer (3.8) located in the imaging system of the TEM

3.12
inelastically scattered electron
electron scattering in which energy and/or momentum are not conserved in the collision system

Note 1 to entry: For inelastic scattering, the electron trajectory is modified by plasmon loss, core loss and other multiple inelastic scatterings.

[SOURCE: ISO 15932:2013, 2.2.2]

3.13
parallel detector
simultaneous detector for energy-dispersed electrons using a *photodiode array* (3.14) or CCD or CMOS

Note 1 to entry: See Figure 1.

3.14
photodiode array
group connected by light tubes to the scintillator positioned at the energy dispersion plane, which detects the energy of electrons simultaneously

3.15
plasmon-loss
type of energy loss in EEL in which the incident electron is affected by the collective oscillations of free electrons in the specimen and loses kinetic energy as a result

[SOURCE: ISO 15932:2013, 2.2.2.2]

3.16
post-column EEL spectrometer
EEL spectrometer (3.8) located behind the imaging/detecting system of the TEM

3.17
serial detection
single channel detection in serial times along the energy axis

3.18
zero-loss
unscattered and *elastically scattered electrons* (3.9) (with only minimal loss of energy due to phonon excitation), giving rise to an intensity peak or the position of which defines zero in the EEL spectrum

[SOURCE: ISO 15932:2013, 2.2.1.1]

4 Symbols and abbreviated terms

CCD	charge coupled device
D	measured energy step of the spectrometer, in eV / channel
dV	change of voltage applied to the drift tube in the magnetic prism
EEL	electron energy loss
EELS	electron energy loss spectroscope/spectroscopy
Ei	measured core-loss edges of the elements to be analysed, *i*, in eV
STEM	scanning transmission electron microscope/spectroscopy
TEM	transmission electron microscope/spectroscopy
Xi	measured positions of zero-loss peak and core-loss edges, *i*, in channel

5 Energy calibration and its determination methods

5.1 Equipment

This document is intended for in-column EEL spectrometer, post-column EEL spectrometer and alternative EEL spectrometer. EEL spectrometers basically work under the same principle as the post-

column EEL spectrometer. Since the post-column EEL spectrometer is widely used, it is adopted as the typical EEL spectrometer. A serial detection system in the EEL spectrometer acquires a spectrum by changing the energy in serial times, where the energy loss spectra are recorded sequentially. Since serial times along the energy axis of serial detection can be replaced with multiple channels of parallel detection, parallel detection is adopted as the model detection in this document.

For the electron energy loss spectroscopy, the apparatus should consist of a spectrometer dispersing the electron energy and a TEM or a STEM with an electron gun and electromagnetic lens system for focusing the electron beam and imaging (see Figure 1).

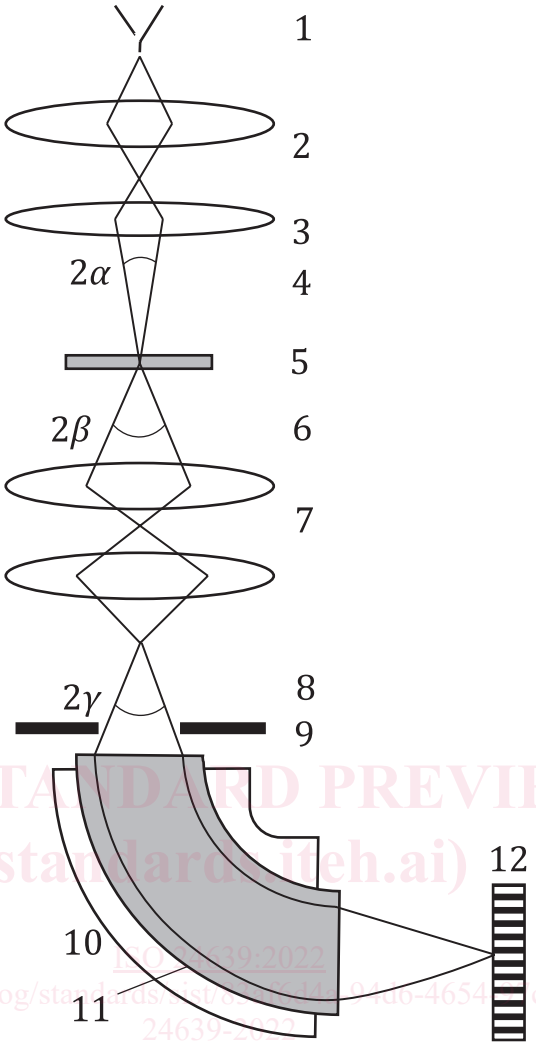
The spectrometer consists of the following (see Figure 1):

— the entrance aperture which allows transmitted electrons within the acceptance angle to enter the magnetic prism;

— the magnetic prism which disperses transmitted probe electrons into a spectrum based on their energy;

— the parallel detector which is a simultaneous detection system of electron energies using a photodiode array or CCD or CMOS;

— the drift tube which accelerates velocities of electrons within a magnetic prism.



Key

1	electron source	7	lenses
2	condenser lens	8	acceptance angle
3	probe-forming lens	9	entrance aperture
4	convergence angle	10	magnetic prism
5	specimen	11	drift tube
6	collection angle	12	parallel detector

Figure 1 — Schematic diagram of incident beam, sample, scattered electrons and spectrometer, which consists of an entrance aperture, a magnetic prism, a drift tube and a parallel detector

5.2 Calibration procedure for energy step and energy scale

5.2.1 General

When detecting electron energy loss spectra by a parallel detector, both the energy step and the energy scale should be calibrated. The energy step should be calibrated before the energy scale calibration. The energy scale needs to be calibrated for every run of EELS analysis, but the energy step calibration does not need for every run. The absolute energy scale needs an accuracy of ±3 % to identify elements of core-loss edges.