# FINAL DRAFT International Standard

# ISO/FDIS 14594

# Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy

*Analyse par microfaisceaux — Analyse par microsonde électronique (Microsonde de Castaing) — Lignes directrices pour la détermination des paramètres expérimentaux pour la spectrométrie à dispersion de longueur d'onde*

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# Foreword

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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 document 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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This document was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 2, *Electron probe microanalysis*.

This third edition cancels and replaces the second edition (ISO 14594:2014), which has been technically revised.

The main changes are as follows:

— Introduction has been added;

— Terms in Clause 3 have been updated;

— Technical terms in Clause 5 have been updated and the clause has been restructured;

— Content of the Test report in Clause 7 has been revised.

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.

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# Introduction

To ensure reliability and reproducibility during electron probe microanalysis (EPMA), the experimental parameters that include beam current, current density, dead time, wavelength resolution, background, analysis area, analysis depth, and analysis volume should be carefully considered. To reliably consider EPMA results, guidelines standardizing the decision procedure of an experimental parameter are important.

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# Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy

## 1 Scope

This document gives general guidelines for the determination of experimental parameters relating to the electron probe, the wavelength spectrometer, and the specimen that need to be taken into account when carrying out electron probe microanalysis. It also defines procedures for the determination of probe current, probe diameter, dead time, wavelength resolution, background, analysis area, analysis depth, and analysis volume.

This document is applicable for the analysis of a well-polished specimen using normal beam incidence.

This document does not apply to energy dispersive X-ray spectroscopy.

## 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.

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ISO/IEC 17025:2017, *General requirements for the competence of testing and testing laboratories*

## 3 Terms and definitions

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

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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**
**analysis area**
area, projected by the interaction volume on the beam entrance surface, from which X-rays are emitted (at a defined fraction, e.g. 95 % of the total) and collected by the spectrometer

[SOURCE: ISO 23833:2013, 5.7.1.1]

**3.2**
**analysis depth**
maximum depth from which a defined fraction (e.g. 95 % of the total) of the X-rays are emitted from the interaction volume after absorption

[SOURCE: ISO 23833:2013, 5.7.1.2]

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**3.3**
**analysis volume**
volume from which a defined fraction (e.g. 95 % of the total) of the X-rays are emitted after generation and absorption

[SOURCE: ISO 23833:2013, 5.7.1.3]

**3.4**
**background**
non-characteristic component of an X-ray spectrum arising (ideally) from the X-ray continuum

[SOURCE: ISO 23833:2013, 5.7.1.2]

**3.5**
**probe current**
electron current contained within the electron probe

[SOURCE: ISO 23833:2013, 4.3.1]

**3.6**
**probe diameter**
diameter of the probe containing a specified fraction of the total current, for example 0,8 (80 %) of the total

[SOURCE: ISO 23833:2013, 4.3.2]

**3.7**
**dead time**
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

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[SOURCE: ISO 23833:2013, 4.5.6]

**3.8**
**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

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[SOURCE: ISO 23833:2013, 4.6.16]

**3.9**
**field of view**
length in the X- and Y-direction of the image or mapping area

**3.10**
**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.

[SOURCE: ISO 23833:2013, 5.1.3]

**3.11**
**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

[SOURCE: ISO 23833:2013, 4.6.14.8]

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**3.12**
**Johann optics**
wavelength-dispersive X-ray spectrometer in which the diffractor is bent to a radius twice that of the Rowland circle, achieving a “semi focussing” situation

[SOURCE: ISO 23833:2013, 4.6.14.7]

# 4 Abbreviated terms

EPMA electron probe microanalysis or electron probe microanalyser

FWHM full width at half maximum

WDS wavelength-dispersive spectrometer

WDX wavelength-dispersive X-ray spectrometry

# 5 Experimental parameters

## 5.1 General

The parameters given in 5.2.1, 5.2.2, and 5.2.3 should be setup properly according to the purpose of the experiment and recorded. Checking the calibration of probe current, and magnification together with counter dead time should be included in the maintenance schedule of the instrument.

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## 5.2 Parameters related to the electron probe

### 5.2.1 Accelerating voltage

The beam energy accelerating voltage typically ranges from 2 keV to 30 keV. Since the sensitivity and spatial resolution (or analysis volume) of analysis depend on the accelerating voltage, the optimization of accelerating voltage can be critical in some cases. However, these performances are also depending on the material of the specimen and the measured X-ray lines. The optimization of accelerating voltage is specified in the guidance for the element and line (see 5.4.3) and the analysis volume (See 5.4.4).

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### 5.2.2 Probe current

Because X-ray peak intensity is directly proportional to the probe current, the precision of the measurement of the probe current should be better than the precision required for quantitative analysis.

Probe current stability over long periods of time is essential for consistent quantitative analysis. The probe current stability should be tested periodically, especially prior to quantitative calibration and analysis. It is possible to compensate for small changes in probe current if this is recorded prior to and following each measurement. Then all X-ray peak and background measurements should be scaled appropriately by $I_i/I_m$, where $I_i$ is the initial probe current and $I_m$ is the probe current at the time of the measurement.

### 5.2.3 Magnification and field of view

To properly define the field of view for line-scans and images acquired by deflecting the electron probe, it is essential to calibrate the magnification scale while operating in the scanning electron mode.

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## 5.3 Parameters related to wavelength dispersive X-ray spectrometers

### 5.3.1 General

An instrument may be fitted with one or more WDX spectrometers, each with a number of diffracting crystals-depending on the line of the analysed element. The following parameters are important for the proper operation of WDX spectrometers.

### 5.3.2 Take-off angle

The take-off angle affects quantitative analysis. Any comparison of measurements from instruments with different take-off angles should be taken into account and the procedures used be noted in the analysis report.

NOTE The value of this angle, which is normally fixed, is provided by the instrument manufacturer.

### 5.3.3 Wavelength resolution

The spectral resolution depends on the following parameters:

— crystal material (and Miller indices of the crystal planes);

— the radius of curvature of the diffracting crystal (Johansson optics vs. Johann optics);

— the size and position of the counter entrance window or of the entrance slit if present.

All these settings determine the wavelength resolution of the measured X-ray spectrum and the observed line-width (FWHM) of the characteristic X-ray peaks. X-ray lines of analysed elements and X-ray order shall be recorded in the test report.

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Resolution can also influence the ability of the system to discriminate against overlapping peaks, background signals, and the sensitivity of measurements to specimen height and beam position on the specimen.

### 5.3.4 X-ray detector and Pulse height analyser

Many spectrometers use a gas-filled proportional counter to detect X-rays. The magnitude of the output pulses from these detectors is determined by the incident X-ray energy and/or the voltage applied to the counters. Two discriminators are used to select the pulse of interest. A low discriminator setting is used to eliminate pulses due to noise, while a high discriminator setting excludes pulses from high order reflections of more energetic X-rays. Optimum settings depend on the X-ray lines of interest.

It is important to set the discriminator to ensure that any unintended shift in pulse amplitude, for example, due to high count rates or changes in atmospheric temperature and pressure (flow counter), has no significant effect on the measured count rate.

Because X-ray counting efficiency decreases with increasing count rate, it is important to correct the measured count rate for the effect of the dead time. In an automated system, the discriminator settings can be set automatically. These settings should be routinely checked to ensure proper automatic operation.

### 5.3.5 Peak location (wavelength)

Under normal circumstances, the wavelength which has the maximum peak intensity is used to define the location of an X-ray peak. It is necessary, using suitable reference materials, to periodically check and correct for the difference in a peak's theoretical position and its actual measured position on a given spectrometer and diffraction crystal. The time between checks will depend on the stability of the instrument spectrometers.

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