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Microbeam analysis — Electron probe microanalysis — Guidelines for qualitative point analysis by wavelength dispersive X-ray spectrometry

Analyse par microfaisceaux — Analyse par microsonde électronique (Microsonde de Castaing) — Lignes directrices pour l'analyse qualitative ponctuelle par spectrométrie de rayons X à dispersion de longueur d'onde (WDX)

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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 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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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The committee responsible for this document is ISO/TC 202, *Microbeam analysis*, Subcommittee SC 2, *Electron probe microanalysis*.

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This second edition cancels and replaces the first edition (ISO 17470:2004), of which it constitutes a minor revision.

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Introduction

Electron probe microanalysis is used to qualitatively identify the elements present in a specimen on a micrometric scale. It is necessary to specify the measurement conditions and identification method in order to avoid reporting erroneous or inconsistent results.

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Microbeam analysis — Electron probe microanalysis — Guidelines for qualitative point analysis by wavelength dispersive X-ray spectrometry

1 Scope

This International Standard gives guidance for the identification of elements and the investigation of the presence of specific elements within a specific volume (on a μm^3 scale) contained in a specimen, by analysing X-ray spectra obtained using wavelength dispersive X-ray spectrometers on an electron probe microanalyser or on a scanning electron microscope.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14594:2003, *Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy*

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3 Terms and definitions

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

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3.1
higher order reflections
peaks appearing at the diffracted angles corresponding to $n = 2, 3, 4...$

Note 1 to entry: In WDS, X-rays are dispersed according to Bragg's law, $n\lambda = 2d \sin\theta$, where λ is the X-ray wavelength, d is the interplanar spacing of the diffraction crystal, θ is the diffraction angle, and n is an integer. The higher order reflections are the peaks appearing at the diffracted angles corresponding to $n = 2, 3, 4...$

3.2
point analysis
analysis in which the primary beam is fixed, thus irradiating a selected region of a sample surface

Note 1 to entry: The method where the primary beam rapidly scans over a very small region on the sample surface is also included. The maximum size of a static beam or a raster area should be chosen such that relative X-ray intensities do not change when enlarging the analysis area.

3.3
Rowland circle
<in a wavelength dispersive X-ray spectrometer> circle of focus along which the X-ray source, diffractor, and detector must all lie in order to satisfy the Bragg condition and obtain constructive interference

3.4
X-ray line table
table of X-ray lines used for qualitative analysis by EPMA

Note 1 to entry: The X-ray line table for qualitative analysis by EPMA lists the wavelengths of K-, L-, and, M-lines for the elements observed on each diffraction crystal. It can also list their relative intensities, the full width at half maximum (FWHM) of each peak, the interplanar spacings of the diffraction crystals, and the wavelengths of satellite peaks.

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4 Abbreviated terms

EPMA electron probe microanalysis

WDS wavelength dispersive X-ray spectroscopy or spectrometry

5 Apparatus

Care should be taken to ensure the instrument is performing satisfactorily. In particular, that the electron column is correctly aligned, the beam current is stable, the accelerating voltage and beam current are appropriate for the sample, the sample surface is prepared suitably for quantitative analysis, the working distance is correct, and the spectrometer crystals and X-ray counters are calibrated and aligned so that the spectrum exhibits X-ray peaks with appropriate intensities and shapes.

NOTE 1 Operators should be aware that parameters such as peak position, relative peak heights, peak resolutions, FWHM values, etc. can vary slightly from instrument to instrument, and also from sample to sample. This can be largely corrected for by periodically comparing values with an appropriate X-ray line table and data from appropriate laboratory reference materials.

NOTE 2 If the sample surface is not planar or polished or perpendicular to the beam, care should be taken in determining the actual value of the local take-off angle and the ability of the spectrometer to properly analyse this kind of sample.

6 Procedure for identification

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6.1 General

X-ray spectra are obtained by directing the incident electron beam at the point to be analysed on the sample surface and scanning the X-ray spectrometers over a specified wavelength range. Qualitative analysis is performed by identifying each peak in the resulting X-ray spectra.

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It is necessary to verify whether the peak identified interferes with a peak resulting from another element. Particular care is needed for possible higher order reflections originating from other elements in the sample, usually, but not always, at higher concentrations.

6.2 Setting of analysis conditions

6.2.1 Primary beam

The primary beam energy should be higher than the X-ray excitation energies of analysed elements, but low enough to minimize sample damage, contamination of the sample, and saturation of the X-ray detectors.

NOTE 1 The Bethe inner shell ionization cross section has a maximum for an overvoltage ratio equal to Napier's number (about 2,7). Taking into account the energy loss of the primary electrons, optimum excitation occurs at overvoltage ratios slightly greater than Napier's number. However, in the case of ultra-light elements and low energy X-rays from other elements (i.e. low energy L- and M-lines), absorption from surface layers can significantly affect the optimum overvoltage causing it to be substantially higher than 2,7.

NOTE 2 The intensity of a generated characteristic X-ray, I, is given approximately by Formula (1):

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$$I = C \times i\left[\left(E_0 - E_c\right)/E_c\right]^{1,7} = C \times i\left(U - 1\right)^{1,7} \quad (1)$$

where

- C is the constant;
- i is the primary beam current (A);
- E_0 is the primary beam energy (keV);
- E_c is the critical excitation energy (keV);
- U is the overvoltage ratio (E_0/E_c).

Note that as the primary beam energy increases, the intensity of generated X-rays become larger. Since the depth of the generation also increases, absorption of X-rays leaving the sample occurs, so the intensities of the detected X-rays do not necessarily become proportionally larger. In samples where absorption is particularly high, such as with light elements Be to F, it is recommended that a primary beam energy of 15 keV or less be used to reduce the depth of X-ray generation and hence the absorption effect. In the case of a thin film sample or a powder sample, primary beam energy should be determined by referring to 5.2 of ISO 14594:2003.

The primary beam current should be also set in the range in which sample damage, sample contamination, and saturation of the X-ray detectors are minimized.

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6.2.2 X-ray spectrometer

6.2.2.1 Selection of diffraction crystals

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The diffraction crystals selected should be capable of detecting the intended elements, with a maximum peak-to-background ratio and peak resolution, but with minimum interference from peaks of other elements in the sample. Since it is not always possible to optimize all of these parameters simultaneously, the analyst should select the diffraction crystal that will provide the best compromise for the specific analysis requirements.

6.2.2.2 Scanning speed

A scanning speed should be selected that will enable the detection of the anticipated elements at their anticipated concentrations.

NOTE 1 The scanning speed of a digitally scanned spectrometer is defined by the number of steps, the length of a step, and the sampling time per step. The number of steps should be high enough and the length of a step should be short enough to sufficiently resolve the shape and height of the detected peaks. In practice, a measured peak should contain a minimum of five data points.

NOTE 2 When the signal from the analysed element is low relative to the background, a slower scanning speed should be used.

6.2.2.3 Pulse height analyser

As peaks of higher order reflections, originating from other constituents, appear in WDS spectra, some uncertainty can arise about the presence of elements having a peak in the same position as one of these higher order reflections. In this case, the higher order reflection can be suppressed by using the discrimination of a pulse height analyser. Care should be taken in selecting a proper discrimination window size and baseline level to avoid unnecessary loss of peak intensity from the element of interest. The analyst should confirm proper transmission of signal by analysing an appropriate reference material and comparing the peak height and shape both with and without pulse height discrimination.

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