International Standard

ISO 14594

Third edition
2024-06

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

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

Reference number
ISO 14594:2024(en)

© ISO 2024

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

COPYRIGHT PROTECTED DOCUMENT

© ISO 2024

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

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 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 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.

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

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.

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

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.

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.

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]

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]

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

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

[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

[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]

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]

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

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.

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).

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.

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.

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

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.

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.

The measured maximum intensities of peaks which have narrow FWHM values are strongly affected by the errors in peak location. The peak intensity can be changed due to the chemical state and polarization effects.

NOTE 1 If the element in the specimen of interest is in a different chemical state than that of the reference material, then the shape of the characteristic X-ray peak can be different for specimen and standard. In this case, it is possible that the peak maximum does not provide a reliable measure of the total peak intensity and an alternative approach, such as peak area measurements, can be required to obtain reliable results. These chemical state effects are particularly important for X-ray peaks with low energy values.

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

NOTE 2 If a crystalline specimen causes the polarization effects in relation to the position between the specimen and the analysis crystal, the peak shape and location can be changed. This can be checked by rotating the specimen around an axis perpendicular to the electron probe and observing the effect on peak shape and location. The problem can occur in systems with symmetry lower than cubic and higher than triclinic and is worst when the Bragg angle is close to 45°. The phenomenon has been found in graphite[1] and certain borides.[2] The effect can be significantly reduced by using peak area measurements.

The position of the peak maximum varies with deviation of the probe from the focal point of the spectrometer on the specimen. Calibration measurements and quantitative analysis on the specimen should normally be made with the probe in the same position relative to this focal point, and using the same probe defocus or raster setting, if applied. For all quantitative and qualitative analyses carried out using a defocused and scanned probe, the area of the specimen surface irradiated should not be so large as to cause a significant fall in X-ray counts from that obtained with the static focused electron probe.

5.3.6 Background

The characteristic X-ray peaks are superimposed on a background of continuum X-rays.

To properly calculate the intensities of characteristic X-rays, the magnitude of this background needs to be determined and corrected if it is statistically significant.

5.4 Parameters related to the specimen

5.4.1 Specimen stage

High precision X, Y, and Z stages allow the specimen and standards to be accurately positioned under the electron probe by using an attached optical microscope; the user can set the height of the specimen so the axis of the WDX spectrometer and the primary beam position coincide at the surface of the specimen. Orthogonality between the electron probe (the optical axis) and the specimen stage is essential in order to perform a proper quantitative analysis. A check on the adjustment of the optical microscope should be included in the routine instrument maintenance schedule.

In an automatic mode of operation, where the measurements are to be made at preset points on the standards and the specimen, it is important to know the reproducibility with which the stage retrieves preset points and to adopt appropriate strategies to overcome any obvious limitations.

5.4.2 Surface roughness

For best results, the surface roughness of the specimen should be minimized.

5.4.3 X-ray line

When X-ray peaks of both of K and L-lines or L and M-lines of the analysed element are detected, selecting analysed X-ray line can be critical. X-ray line of shorter wavelength is generally not affected by X-ray absorption in the specimen, surface contamination, or coating material. However, the overvoltage ratio should be higher than 1,5 to secure the sensitivity of the analysis. In the case where analysing longer wavelength X-ray is difficult, selecting higher accelerating voltage can be better solution if it does not affect the analysis of X-ray peaks of other elements or spatial resolution of analysis (see 5.4.4).

X-ray lines of analysed elements shall be recorded in the test report.

5.4.4 Analysis volume

Analysis volume is determined by the incident electron probe area, the depth of electron penetration, the spread of the incident electron probe within the specimen, and the energy of the characteristic X-ray line. This analysis volume can be significantly increased by unwanted fluorescence effects which are caused by the characteristic and continuum X-rays.

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

In case of micro-structural specimen, the analysis depth and/or should be estimated. Minimizing the accelerating voltage and selecting longer wavelength X-ray line can be effective to improve the spatial resolution of analysis.

6 Procedures and measurements

6.1 General

The following procedures should be adopted to determine a number of critical parameters.

6.2 Electron probe

6.2.1 Probe current

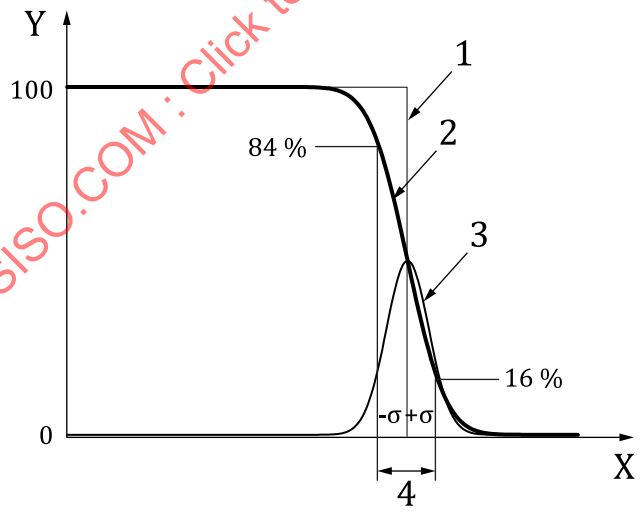
Measure the probe current using a Faraday cup. It should be positioned after the final aperture. If the measurement is carried out at another position, the relationship between the above-mentioned position and this position shall be shown.

6.2.2 Probe diameter

The diameter of the probe shall be defined by one of the following methods.

a) The diameter of electron probe shall be defined as the interval where the emitted secondary electron intensity drops from 84 % to 16 % of the maximum peak intensity, which is equivalent to two standard deviations (2 σ) of the error curve (see Figure 1). This measurement should be done such that the primary electron crosses a knife edge at a right angle.

b) The diameter of area exhibiting optical fluorescence for a material such as aluminium oxide, zirconium oxide, or thorium oxide, whereby that diameter is determined by using an optical microscope. This measurement should be done when the probe diameter is more than 5 µm.



Key

X	distance	2	measured curve
Y	secondary electron intensity	3	error function a diameter
1	true boundary	4	probe diameter

Figure 1 — Method for measuring probe diameter

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

6.3 Parameters related to measured peaks

6.3.1 Dead time correction

6.3.1.1 General Measure the probe current, *i*, in accordance with 6.2.1 and the count rate (*N*).

6.3.1.2 As shown in Figure 2, make a calibration curve by using the count rate, *N*, divided by the probe current, *i*, values as the ordinate values and the count rate, *N*, as the abscissa values.

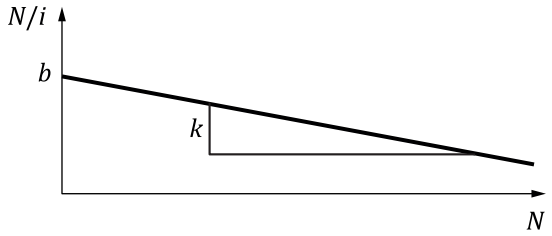


Figure 2 — Counting loss by dead time

In order to confirm the linearity of the current measuring device, it is advisable to monitor the count rate, *n*, of a low intensity line at the same time as the count rate, *N*, of a high intensity line; the ratio *n*:*i* should be constant for the whole range of measurements.

Typically, a Kα line can be used to determine *N* and the corresponding Kβ line for *n*.

NOTE If the probe current cannot be measured accurately, the dead time can be determined by measuring the ratio of two X-ray intensities as a function of count rate on two X-ray spectrometers[2][3].

6.3.1.3 Determine the gradient, *k*, and the intercept value, *b*, at the ordinate axis from the calibration curve.

Calculate the dead time, τ, by using Formula (1):

$$\tau = \frac{-k}{b} \tag{1}$$

where

k is the gradient;

b is the intercept value.

6.3.1.4 Calculate the true count rate, N_o, from the measured count rate, *N*, by using Formula (2):

$$N_o = \frac{N}{1 - N \times \tau} \tag{2}$$

For accurate measurements, the count rate should be restricted so that the correction for dead time does not exceed 5 %.

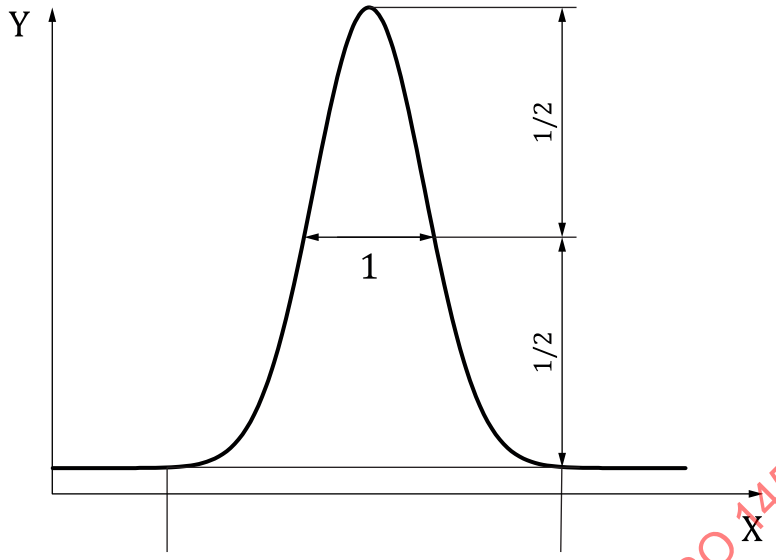
6.3.2 Wavelength resolution of detected characteristic X-ray peaks

6.3.2.1 Measure a characteristic X-ray intensity versus wavelength spectrum for the elements of interest by measuring the intensity of the X-ray signals while scanning over the wavelengths of interest. Calculate

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

the wavelength resolution of the detected characteristic X-rays by using the following definition. After subtracting the background, (see 6.3.3) wavelength resolution is equal to FWHM as shown in Figure 3.



Key

X wavelength

Y X-ray intensity

1 FWHM (Full Width at Half Maximum)

Figure 3 — Definition of FWHM

6.3.3 Background subtraction

6.3.3.1 The net intensity of a characteristic X-ray peak is obtained by subtracting the intensity of the background from the intensity of the observed X-ray peak because the observed X-ray peak is the sum of the true intensity of the characteristic X-ray peak and the background intensity.

6.3.3.2 Measure X-ray intensity versus wavelength spectrum in accordance with 6.3.2.1.

6.3.3.3 Select wavelength positions on both sides of the peak of interest as near as possible to the peak, but where any residual intensity from the peak is less than 1 % of the maximum peak intensity, avoiding any subsidiary or interfering peak. To obtain the net characteristic peak intensity, the background intensity at the peak position should be estimated by making a linear interpolation between the two selected background positions, and by subtracting that background intensity from the intensity of the measured peak.

The procedure in 6.3.3.3 can be difficult to apply when the intensity of the background is changing rapidly with wavelength, or when some interference prevents the selection of positions that are reasonably close to the peak. In these cases of difficulty, it will be necessary to estimate background by applying a curve fitting procedure or by measuring a material whose average atomic number is as close as possible to the specimen of interest and which does not contain the element that produces the characteristic peak being analysed.

The time used to count the background intensity should be enough for the desired precision to be maintained.

The nature of the measurement being made, e.g. major component or trace element, is of prime importance in this consideration and should be reported along with details of the individual and overall precision which have been achieved.

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

6.4 Parameters related to the specimen

6.4.1 General

For many routine analyses, an individual calculation of the dimensions of the analysed volume is not necessary and an estimate can be given with reference to previous similar analyses. There are, however, special cases such as inclusions, fine-grained material, or coatings where knowledge of the spatial resolution of the analysis provides an important justification that the analysis is valid. In these cases, the depth, area, or volume of the analysis can be calculated using the information given in 6.4.2, 6.4.3, and 6.4.4. When this is done, the method chosen and the result should be included in the test report.

Methods for the determination of analysis area, analysis depth, and analysis volume are given in Annex A, Annex B, and Annex C, respectively.

6.4.2 Analysis area

Estimate the analysis area by applying an appropriate method, detailing all parameters used.

6.4.3 Analysis depth

Estimate the analysis depth by applying an appropriate method, detailing all parameters used.

6.4.4 Analysis volume

The first estimation of analysis volume is equal to the product of the analysis area (6.4.2) times the analysis depth (6.4.3).

The more precise value of the analysis volume should be calculated using one of the methods given in Annex C.

7 Test report

Records of the instrument and individual investigations shall be kept so that a test report conforming to ISO/IEC 17025:2017, 7.8.2 can be issued. The test report shall contain the following:

a) title of ISO method used;

b) the method used (if the standard includes several);

c) name and address of laboratory that performed the calibration(s) or test(s);

d) name and address of client, where relevant;

e) date of receipt of calibration item or test item and the date(s) that the calibration(s) or test(s) were performed, where relevant;

f) the sample

g) the result(s), including a reference to the clause which explains how the results were calculated;

h) instrument type and reference number;

i) reference to sampling procedure, where relevant;

j) Accelerating voltage;

k) probe current;

l) probe diameter

m) X-ray peak(s) detected (element, type, order);

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

n) take-off-angle of the instrument;

o) estimation method of analysis volume, if relevant; note value of the constant for Formula (A.1) in Annex A;

p) estimate of the uncertainty of the calibration or test result (where relevant);

q) any deviations from, additions to, or exclusions from the calibration method or test method, and any other information relevant to a specific calibration or test, including, e.g. environmental conditions;

r) any unusual features observed;

s) signature and title of person giving the signature, or an equivalent identification of the person(s) accepting responsibility for the content of the certificate or report, and date when the report was issued.

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

Annex A
(informative)

Methods of estimating analysis area

Although not always an issue in wavelength dispersive X-ray analysis, determination of the analysis area is often useful, particularly when analysing particles, fine grained materials, or layered specimens. A variety of methods exists in the literature for estimating the approximate area of electrons having energies sufficient to excite an X-ray line of interest. Any of these procedures can be used as a first estimate of the analysis area Type text.

The analysis area, A, is given by Formula (A.1):

$$A = \pi \left(\frac{d}{2} \right)^2 \tag{A.1}$$

where d is the diameter computed or estimated by one of the following methods:

— Calculate the diameter of the analysis area using the Monte Carlo method (see Annex C).

— Calculate the analysis area after determining the diameter of the analysis area by applying Formula (A.2), which is based on the diffusion model for electron penetration, and Formula (B.1) with a constant equal to 0,025.

$$d = D_e + \frac{2,2\gamma}{1+\gamma} \times Z_m \tag{A.2}$$

where

$$\gamma = 0,187 Z^{2/3}$$

$$Z_m = 0,025\left(E_i^{1,7} - E_k^{1,7} \right) \times \frac{m_a}{\rho Z}$$

where

d is the diameter of analysis area, in micrometres;

D_e is the diameter of primary electron probe, in micrometres;

Z_m is the X-ray generation depth, in micrometres;

Z is the mean atomic number of the bombarded point;

E_i is the energy of the incident electron, in kilo-electron volts;

E_k is the critical excitation energy, in kilo-electron volts;

m_a is the mean atomic mass of the bombarded point;

ρ is the mass density of the bombarded point, in grams per cubic centimetre.

— The following experimental method which estimates the diameter of the analysis area by using a bi-metal system.

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

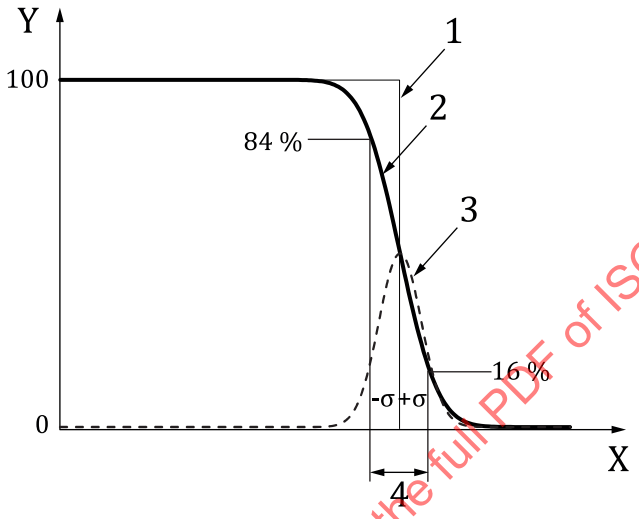
© ISO 2024 – All rights reserved

Prepare a specimen, formed by plating, which shall be made of two joined materials that have similar mass density, do not suffer inter-diffusion, and have only a small fluorescence effect on each other, e.g. Ni/Cu.

Measure the characteristic X-ray intensity as a function of distance by moving an electron probe perpendicular to the interface between the two different materials or by moving the specimen stage.

Determine the interval distance over which the intensity of one characteristic X-ray signal drops from 84 % to 16 % of the maximum signal intensity and use that distance as diameter of the detected X-ray signal. That interval distance is equivalent to two standard deviations (2 σ) of the error curve as shown in Figure A.1.

NOTE This approach is only applicable to the particular couple used, unless the probe diameter is much greater than the electron spread.



Key

X distance
Y X-ray intensity
1 true boundary
2 measured curve
3 error function
4 diameter of the X-ray signal

Figure A.1 — Estimation for the analysis area

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

Annex B
(informative)

Methods of estimating analysis depth

Although not always an issue in wavelength dispersive X-ray analysis, determination of the analysis depth is often useful, particularly when analysing particles, fine grained materials, or layered specimens. A variety of methods exists in the literature for estimating the approximate depth of penetration of electrons having energies sufficient to excite an X-ray line of interest. Any of these procedures can be used as a first estimate of the analysis depth. Three such methods are outlined below. However, the exact analysis depth (defined as the depth above which *n* % of the characteristic X-rays detected by the spectrometer are generated, where "*n*" is greater than 90) can be significantly affected by the depth distribution of absorbed X-rays, particularly for low energy radiations or high-*Z* materials, and by X-rays generated by characteristic and continuum fluorescence. Other methods can also be used.

— $\Phi(\rho Z)$ expression[4]

— Monte Carlo simulation (see Annex C)

— Formula (B.1) which is based on many experimental results where

$$Z_m = const. \times \left(E_i^{1,7} - E_k^{1,7} \right) \times \frac{m_a}{\rho Z} \tag{B.1}$$

where

Z_m is the X-ray generation depth, in micrometres;

E_i is the energy of the incident electron, in kilo-electron volts;

E_k is the critical excitation energy, in kilo-electron volts;

m_a is the mean atomic mass of the bombarded point;

ρ is the mass density of the bombarded point, in grams per cubic centimetre;

Z is the mean atomic number of the bombarded point.

$$m_a = \sum_i C_i m_{a,i} \tag{B.2}$$

$$Z = \sum_i C_i Z_i \tag{B.3}$$

where

C_i is the mass fraction for element *i*;

$m_{a,i}$ is the atomic mass of the element *i*;

Z_i is the atomic number of the element *i*.

NOTE The constant in Formula (B.1) is normally either 0,033[5] or 0,025[6].

If 0,025 and 0,033 are used as the constant, approximately 95 % and 99 % respectively of the directly produced characteristic X-rays are produced within the depth Z_m

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved

Annex C
(informative)

Method of estimating X-ray analysis volume by applying the Monte Carlo (MC) simulation

C.1 General

When an electron probe is incident on a specimen, X-rays are generated from a large volume due to electron penetration. In X-ray microanalysis, it is important to know this volume in order to know the resolution of this analysis. The volume is dependent upon beam energy, the kind of characteristic X-ray, and so on. A variety of methods such as an experimental method, an analytical method based on the transport formula, and a Monte Carlo method, have so far been proposed.[7] The Monte Carlo method outlined below simulates the electron behaviour based on the electron scattering theory, which is the statistical method due to random sampling through use of pseudo-random numbers. The method can be applied to any solid materials, can handle any type of experimental conditions, and is relatively easy to set in a simulation programme. Here, the simplest model, the single scattering model, is described. Other methods can be also used.

C.2 By applying the following theories and estimations, it is possible to use the Monte Carlo method to simulate the electron trajectory in solid materials.

a) Principal formula

Differential scattering cross section (based on screened Rutherford theory) as shown in Formula (C.1)[8]

$$\frac{d\sigma_i}{d\Omega} = \frac{e^4 Z_i (Z_i + 1)}{4E^2 (1 + 2\beta_i - \cos\theta)^2} \tag{C.1}$$

where

Ω is the scattering solid angle;

E is the elementary electron charge;

Z_i is the atomic number of the element i;

E is the kinetic energy of an electron, in electron volts.

Screening parameter, as shown in Formula (C.2)[9]

$$\beta_i = \frac{5{,}44 Z_i^{2/3}}{E} \tag{C.2}$$

Stopping power, as shown in Formula (C.3)[10]

$$\frac{dE}{dS} = -2\pi e^4 \rho \times \frac{N}{E} \times \sum \frac{C_i Z_i}{A_i} \times \ln \frac{1{,}166E}{J_i} \tag{C.3}$$

Mean ionization potential, as shown in Formula (C.4)[11]

$$J_i = 9{,}76 Z_i + 58{,}5 Z_i^{-0{,}19} \tag{C.4}$$

b) Scattering process, as shown in Formula (C.5)

STANDARDSISO.COM : Click to view the full PDF of ISO 14594:2024

© ISO 2024 – All rights reserved