FINAL
DRAFT

INTERNATIONAL
STANDARD

ISO/FDIS 18118

ISO/TC **201**/SC **7**

Secretariat: **BSI**

Voting begins on:
2023-11-09

Voting terminates on:
2024-01-04

Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials

Analyse chimique des surfaces — Spectroscopie des électrons Auger et spectroscopie de photoélectrons — Lignes directrices pour l'utilisation de facteurs expérimentaux de sensibilité relative pour l'analyse quantitative de matériaux homogènes

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO/FDIS 18118
https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43c7-afc6-9cda12458907/iso-fdis-18118

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

Reference number
ISO/FDIS 18118:2023(E)

© ISO 2023

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO/FDIS 18118
https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43c7-afc6-9cda12458907/iso-fdis-18118

COPYRIGHT PROTECTED DOCUMENT

© ISO 2023

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

Foreword .... **iv**
Introduction .... **v**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Symbols and abbreviated terms **4**
5 General information **6**
6 Measurement conditions **7**
6.1 General 7
6.2 Excitation source 7
6.3 Energy resolution 7
6.4 Energy step and scan rate 7
6.5 Signal intensity 7
6.6 Gain and time constant (for AES instruments with analogue detection systems) 7
6.7 Modulation to generate a derivative spectrum 7
7 Data-analysis procedures **8**
8 Spectrometer response function **8**
9 Determination of chemical composition using relative sensitivity factors **9**
9.1 Calculation of chemical composition 9
9.1.1 General 9
9.1.2 Composition determined from elemental relative sensitivity factors 9
9.1.3 Composition determined from atomic relative sensitivity factors or average matrix relative sensitivity factors 9
9.2 Uncertainties in calculated compositions 9
Annex A (informative) **Formulae for relative sensitivity factors** **10**
Annex B (informative) **Information on uncertainty of the analytical results** **16**
Bibliography **19**

iTeh Standards (https://standards.iteh.ai) Document Preview
ISO/FDIS 18118
https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43c7-afc6-9cda12458907/iso-fdis-18118

© ISO 2023 – 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.

iTeh Standards
(https://standards.iteh.ai)
Document Preview

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 201, *Surface chemical analysis*, Subcommittee SC 7, *Electron spectroscopies*.

ISO/FDIS 18118
https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43c7-afc6-9cda12458907/iso-fdis-18118

This third edition cancels and replaces the second edition (ISO 18118:2015), which has been technically revised.

The main changes are as follows:

— The main equation for the use of sensitivity factors for analysis have been moved from Annex A to the main text

— Defined symbols and abbreviated terms from the Annexes have been consolidated to Clause 4.

— Several terms have been modified for formatting purposes, and some have been removed due to no longer being required.

— Several formulae have been removed from Annex A and replaced by references and references to databases of parameters that are more accurate. Such databases are now the recommended source for the parameters previously calculated by these formulae.

— Annex A has been redefined as an informative Annex.

— Multiple small additions have been made to provide new and updated sources for information.

— Editorial changes have been made throughout.

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.

 © ISO 2023 – All rights reserved

Introduction

Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) are surface-analytical techniques that are sensitive to the composition in the surface region of a material to depths of, typically, a few nanometres (nm). Both techniques yield a surface-weighted signal, averaged over the analysis volume. Most samples have compositional variations, both laterally and with depth, and quantification is often performed with approximate methods since it can be difficult to determine the magnitude of any compositional variations and the distance scale over which they can occur. The simplest sample for analysis is one that is homogeneous. Although this situation occurs infrequently, it is often assumed, for simplicity in the analysis, that the sample material of interest is homogeneous. This document provides guidance on the measurement and use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials by AES and XPS.

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO/FDIS 18118
https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43c7-afc6-9cda12458907/iso-fdis-18118

© ISO 2023 – All rights reserved

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO/FDIS 18118
https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43c7-afc6-9cda12458907/iso-fdis-18118

Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials

1 Scope

This document gives guidance on the measurement and use of experimentally-determined relative sensitivity factors for the quantitative analysis of homogeneous materials by Auger electron spectroscopy and X-ray photoelectron spectroscopy. The methods described only apply to polycrystalline and amorphous materials, as effects inherent to single-crystal samples are not addressed.

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.

iTeh Standards
(https://standards.iteh.ai)
Document Preview

ISO 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

ISO 21270, *Surface chemical analysis — X-ray photoelectron and Auger electron spectrometers — Linearity of intensity scale*

3 Terms and definitions

ISO/FDIS 18118
https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43c7-afc6-9cda12458907/iso-fdis-18118

For the purposes of this document, the terms and definitions given in ISO 18115-1 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
absolute elemental sensitivity factor
coefficient for an element by which the measured intensity for that element is divided to yield the atomic concentration or atomic fraction of the element present in the sample

Note 1 to entry: See *relative elemental sensitivity factor*.

Note 2 to entry: The choice of atomic concentration or atomic fraction should be made clear.

Note 3 to entry: The type of sensitivity factor utilized should be appropriate for the formulae used in the quantification process and for the type of sample analysed, for example homogeneous samples or segregated layers.

Note 4 to entry: The source of sensitivity factors should be given to ensure that the correct *matrix factors* or other parameters are used.

© ISO 2023 – All rights reserved

Note 5 to entry: Sensitivity factors depend on parameters of the excitation source, the spectrometer, and the orientation of the sample to these parts of the instrument. Sensitivity factors also depend on the matrix being analysed, and in *SIMS* this has a dominating influence.

[SOURCE: ISO 18115-1]

3.2
elemental relative sensitivity factor

<AES, XPS> coefficient proportional to the *absolute elemental sensitivity factor* (3.1), where the constant of proportionality is chosen such that the value for a selected element and transition is unity

Note 1 to entry: Elements and transitions commonly used are C 1s or F 1s for *XPS* and Ag $M_{4,5}$VV for *AES*.

Note 2 to entry: The type of sensitivity factor used should be appropriate for the type of sample analysed, for example homogeneous samples or segregated layers.

Note 3 to entry: The source of sensitivity factors should be given to ensure that the correct *matrix factors* or other parameters are used.

Note 4 to entry: Sensitivity factors depend on parameters of the excitation source, the spectrometer, and the orientation of the sample to these parts of the instrument. Sensitivity factors also depend on the matrix being analysed, and in *SIMS* this has a dominating influence.

[SOURCE: ISO 18115-1]

3.3
average matrix relative sensitivity factor
AMRSF

<AES, XPS> coefficient, proportional to the intensity, calculated for an element in an average matrix, by which the measured intensity for that element is divided in calculations to yield the atomic concentration or atomic fraction of the element present in the sample

iTeh Standards (https://standards.iteh.ai) Document Preview

Note 1 to entry: See sensitivity factor, *elemental relative sensitivity factor* (3.2) and *pure element relative sensitivity factor* (3.4).

ISO/FDIS 18118

Note 2 to entry: The choice of atomic concentration or atomic fraction should be made clear.

https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43c7-afc6-9cda12458907/iso-fdis-18118

Note 3 to entry: The type of sensitivity factor utilized should be appropriate for the formulae used in the quantification process and for the type of sample analysed, for example homogeneous samples or segregated layers.

Note 4 to entry: The source of sensitivity factors should be given. *Matrix factors* are taken to be unity for average matrix relative sensitivity factors.

Note 5 to entry: Sensitivity factors depend on parameters of the excitation source, the spectrometer, and the orientation of the sample to these parts of the instrument. The numerical values of the sensitivity factors can also depend on the method used to measure the peak intensities.

[SOURCE: ISO 18115-1]

3.4
pure-element relative sensitivity factor
PERSF

<AES, XPS> coefficient, proportional to the intensity measured for a pure sample of an element, by which the measured intensity for that element is divided in calculations to yield the atomic concentration or atomic fraction of the element present in the sample

Note 1 to entry: See sensitivity factor, *elemental relative sensitivity factor* (3.2), and *average matrix relative sensitivity factor* (3.3).

Note 2 to entry: The choice of atomic concentration or atomic fraction should be made clear.

© ISO 2023 – All rights reserved

Note 3 to entry: The type of sensitivity factor used should be appropriate for the formulae used in the quantification process and for the type of sample analysed, for example homogeneous samples or segregated layers.

Note 4 to entry: The source of sensitivity factors should be given to ensure that the correct *matrix factors* or other parameters are used. Matrix factors are significant and should be used with pure-element relative sensitivity factors.

Note 5 to entry: Sensitivity factors depend on parameters of the excitation source, the spectrometer, and the orientation of the sample to these parts of the instrument. The numerical values of the sensitivity factors can also depend on the method used to measure the peak intensities.

[SOURCE: ISO 18115-1]

3.5
spectrometer response function
quotient of the number of particles detected with a spectrometer by the number of such particles per solid angle and per interval of the dispersing parameter available for measurement as a function of the dispersing parameter

Note 1 to entry: See *spectrometer étendue*.

Note 2 to entry: The dispersing parameter is commonly energy, mass, or wavelength.

Note 3 to entry: The units of the spectrometer response function can be sr·eV, sr·amu, or sr·m.

Note 4 to entry: The spectrometer response function is similar to the *spectrometer transmission function* (3.6) or étendue but includes the efficiencies of all other components of the measurement chain, such as detectors and the electronic processing and recording equipment.

iTeh Standards
(https://standards.iteh.ai)
Document Preview

Note 5 to entry: For some methods of *quantitative analysis*, the energy dependence of the response function is needed in order to use *relative sensitivity factors*. For these cases, a function is determined which is proportional to the absolute response function, where the proportionality constant is not necessarily important.

[SOURCE: ISO 18115-1]

ISO/FDIS 18118
https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43c7-afc6-9cda12458907/iso-fdis-18118

3.6
spectrometer transmission function
analyser transmission function
quotient of the number of particles transmitted by the analyser by the number of such particles per solid angle and per interval of the dispersing parameter (e.g. energy, mass, or wavelength) available for measurement as a function of the dispersing parameter

Note 1 to entry: See *spectrometer response function* (3.5).

Note 2 to entry: The units of transmission can be sr·eV, sr·amu, or sr·m.

Note 3 to entry: Often, an incomplete use of the term occurs where just the solid angle of acceptance of the spectrometer, in sr, or a fraction of the 2π solid angle of available space is given. This usage is deprecated, cf. *solid angle of analyser*.

Note 4 to entry: This term is often used incorrectly instead of spectrometer response function, which includes contributions from the detector and the signal-processing system.

[SOURCE: ISO 18115-1]

3.7
atomic relative sensitivity factor
ARSF
coefficient, proportional to the intensity measured for a single atom of an element, by which the measured intensity for that element is divided in calculations to yield the atomic concentration or atomic fraction of the element present in the sample.

Note 1 to entry: See *elemental relative sensitivity factor* (3.2).

© ISO 2023 – All rights reserved

Note 2 to entry: The choice of atomic concentration or atomic fraction should be made clear.

Note 3 to entry: The type of sensitivity factor utilized should be appropriate for the formulae used in the quantification process and for the type of sample analysed, for example homogeneous samples or segregated layers.

Note 4 to entry: The source of sensitivity factors should be given to ensure that the correct *matrix factors* or other parameters are used.

Note 5 to entry: Sensitivity factors depend on parameters of the excitation source, the spectrometer, and the orientation of the sample to these parts of the instrument. Sensitivity factors also depend on the matrix being analysed, and in *SIMS* this has a dominating influence.

4 Symbols and abbreviated terms

AES Auger electron spectroscopy

A_i atomic mass of element i

$E_{b,i}$ binding energy of core level for element i

E_g band-gap energy

E_i kinetic energy of an auger electron or photoelectron from element i

E_p free-electron plasmon energy

E_{pr} primary electron energy

F_i matrix correction factor for element i

F_j matrix correction factor for element j

$H(\cos\alpha, \omega_i)$ Chandrasekhar function for parameters $\cos\alpha$ and ω_i

I_{key} measured intensity of the chosen peak in the key material

C_i number of atoms of element i in the molecular formula of the compound

$I_{i,ref}$ measured intensity of element i in the reference sample

$I_{i,unk}$ measured intensity of element i in the unknown sample

$I_{j,ref}$ measured intensity of element j in the reference sample

$I_{j,unk}$ measured intensity of element j in the unknown sample

J excitation beam intensity

M_i molecular mass of the compound containing element i

n number of identified elements in the unknown sample

N_A Avogadro constant

N_{av} atomic density for the average matrix sample

N_i atomic density of element i

$N_{i,ref}$ atomic density of the reference sample of element i

iTeh Standards
(https://standards.iteh.ai)
Document Preview
ISO/FDIS 18118
https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43c7-afc6-9cda12458907/iso-fdis-18118

© ISO 2023 – All rights reserved

N_{key}	atomic density of the key element
N_{unk}	atomic density of the unknown sample
N_v	number of valence electrons per atom or molecule
Q_{av}	elastic-scattering correction factor for the average matrix sample at the electron energy E_i for the particular Auger-electron or photoelectron peak of interest
Q_i	elastic-scattering correction factor for element *I* at the electron energy E_i for the particular Auger-electron or photoelectron peak of interest
$Q_i(0)$	elastic-scattering correction factor for element *i* at emission angle $\alpha = 0$ with respect to the surface normal
$Q_{i,ref}$	elastic-scattering correction factor for element *i* in the reference sample
$Q_{i,unk}$	elastic-scattering correction factor for element *i* in the unknown sample
R_{av}	backscattering correction factor for the average matrix sample
R_i	backscattering correction factor for element *i*
$R_{i,ref}$	backscattering correction factor for element *i* in the reference sample (these terms are unity for XPS)
$R_{i,unk}$	backscattering correction factor for element *i* in the unknown sample (these terms are unity for XPS)
RSF	relative sensitivity factor
$S_{i,At}$	atomic relative sensitivity factor for element *i*
$S_{i,Av}$	average matrix relative sensitivity factor for element *i*
$S_{i,Ec}$	elemental relative sensitivity factor for element *i* in a specified compound
$S_{i,Ep}$	pure-element relative sensitivity factor for element *i*
$S_{i,RSF}$	relative sensitivity factor for element *i*
$S_{i,E}$	elemental relative sensitivity factor for element *i*
$S_{j,RSF}$	relative sensitivity factor for element *j*
T	spectrometer response function (SRF)
XPS	X-ray photoelectron spectroscopy
Z	atomic number
Z_{av}	atomic number of the average matrix sample
α	emission angle with respect to the surface normal
θ	angle of incidence of electron beam
$X_{i,ref}$	atomic fraction of element *i* in the reference sample
$X_{i,unk}$	atomic fraction of element *i* in the unknown sample

iTeh Standards
(https://standards.iteh.ai)
Document Preview
ISO/FDIS 18118
https://standards.iteh.ai/catalog/standards/sist/957adc92-9b98-43e7-afc6-9cda12458907/iso-fdis-18118

© ISO 2023 – All rights reserved