TECHNICAL REPORT

ISO/TR 23173

First edition
2021-06

Surface chemical analysis — Electron spectroscopies — Measurement of the thickness and composition of nanoparticle coatings

Analyse chimique des surfaces — Spectroscopies d'électrons — Mesurage de l'épaisseur et de la composition des revêtements de nanoparticules

Reference number
ISO/TR 23173:2021(E)

© ISO 2021



COPYRIGHT PROTECTED DOCUMENT

© ISO 2021

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 ... **1**
5 Overview ... **3**
6 X-ray photoelectron spectroscopy ... **4**
6.1 General ... 4
6.2 Coating thickness measurement ... 4
6.3 Nanoparticle coating thickness ... 5
6.4 Numerical methods ... 6
6.5 Descriptive formulae ... 9
6.6 Modelling and simulation software ... 11
6.7 Method comparisons ... 12
6.8 Inelastic background analysis ... 15
6.9 Elemental composition ... 16
6.10 Variable excitation energy XPS ... 18
6.10.1 General ... 18
6.10.2 Qualitative depth-profiling ... 19
6.10.3 Quantitative depth-profiling ... 22
6.11 Near-ambient-pressure XPS (NAP-XPS) ... 23
6.11.1 General ... 23
6.11.2 Internal structure of bimetallic NP catalysts ... 24
6.11.3 Measurement of NP's in liquid suspension ... 24
7 Auger electron spectroscopy ... **26**
7.1 General ... 26
7.2 Coating thickness measurement ... 26
7.2.1 General ... 26
7.2.2 Destructive depth-profiling ... 27
7.2.3 Non-destructive depth-profiling ... 27
7.2.4 Elemental composition ... 27
7.2.5 Imaging and line scans ... 28
8 Complementary analysis ... **30**
9 Deviations from ideality ... **33**
9.1 General ... 33
9.2 Multilayered coatings ... 33
9.3 Other non-ideal cases ... 35
Annex A (informative) **Example script for modelling of XPS data from nanoparticles** ... **40**
Bibliography ... **42**

© ISO 2021 – 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 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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

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 2021 – All rights reserved

Introduction

Recently, there has been increasing development and use of nanoparticles in a wide range of application areas, including catalysis, medicine, energy, optoelectronics and cosmetics[1]–[7]. In particular, nanoparticles having some form of coating layer, which is present either by design or due to incidental processes such as contamination or oxidation, are among the most commonly studied and utilised[8]–[11]. An essential part of the characterisation of nanoparticles is the measurement of the surface properties because a large proportion of the material is at a surface or interface. In the case of coated nanoparticles, the thickness and composition of the coating has a significant role determining its functional properties and defines the interaction of the particle with its environment. Many applications require nanoparticles to have coatings that are specifically designed in order to achieve a desired level of performance. Measurement of surface composition and coating thickness of nanoparticles is a challenge to which electron spectroscopies are well suited, due to high surface sensitivity, well-understood physical principles and accessibility. Such measurements can have a significant dependence on sample format and condition; sample handling and provenance of nanoparticle samples for surface chemical analysis are addressed in ISO 20579[12]. A general introduction to the challenges of surface chemical analysis of nanostructured materials is provided in ISO/TR 14187[13].

© ISO 2021 – All rights reserved

Surface chemical analysis — Electron spectroscopies — Measurement of the thickness and composition of nanoparticle coatings

1 Scope

This document provides a description of methods by which the coating thickness and chemical composition of "core-shell" nanoparticles (including some variant and non-ideal morphologies) can be determined using electron spectroscopy techniques. It identifies the assumptions, challenges, and uncertainties associated with each method. It also describes protocols and issues for the general analysis of nanoparticle samples using electron spectroscopies, specifically in relation to their importance for measurements of coating thicknesses.

This document focuses on the use of electron spectroscopy techniques, specifically X-ray photoelectron spectroscopy, Auger electron spectroscopy, and synchrotron-based methods. These cannot provide all of the information necessary for accurate analysis and therefore some additional analytical methods are outlined in the context of their ability to aid in the interpretation of electron spectroscopy data.

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

ISO 18115-2, *Surface chemical analysis — Vocabulary — Part 2: Terms used in scanning-probe microscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1 and ISO 18115-2 apply.

ISO and IEC maintain terminological 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/

4 Symbols and abbreviated terms

X subscripts	denote the material of the overlayer
Y subscripts	denote the material of the core
x subscripts	denote a specific photoelectron peak from material *X*
y subscripts	denote a specific photoelectron peak from material *Y*
I_i	intensity of electrons arising from a peak, *i*

© ISO 2021 – All rights reserved