TECHNICAL REPORT

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

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





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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).

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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.

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]-[2]. 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, wellunderstood 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 a ISC red man 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].

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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 intensity of electrons arising from a peak, I