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Surface chemical analysis — Sample handling, preparation and mounting —

Part 3: Biomaterials

Analyse chimique des surfaces — Manipulation, préparation et montage des échantillons —

Partie 3: Biomatériaux

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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).

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 2, *General procedures*.

A list of all parts in the ISO 20579 series can be found on the ISO website.

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.

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Introduction

0.1 Common introduction

The ISO 20579 series is intended to assist analysts and those seeking surface chemical analysis in the handling, storage, mounting and treatment of specimens. This is a multipart series, with the first two parts specifying general requirements for reporting of sample handling and storage (ISO 20579-1[1)]), and reporting of mounting and treatment of samples (ISO 20579-2[1)]). The ensuing parts combine any new requirements of sample handling/storage and/or sample mounting/preparation for classes of new materials. This document focuses on biomaterials analysis and handling, and ISO 20579-4 focuses on reporting and handling needs for nano-objects. Each part of this series can be used independently of the other parts, although the general procedures described in ISO 20579-1 and ISO 20579-2 are applicable to a wide range of materials and are not reproduced in detail in material-specific sections.

Although primarily prepared for the surface-analysis techniques of Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS) and secondary ion mass spectrometry (SIMS), the methods described in this series will also be applicable to many other surface-sensitive analytical techniques such as ion-scattering spectrometry, low-energy electron diffraction and electron energy-loss spectroscopy, where specimen handling can influence surface-sensitive measurements. AES, XPS and SIMS are sensitive to surface layers that are typically a few nanometers in thickness. Such thin layers may be subject to severe perturbations caused by specimen handling or surface treatments that may be necessary prior to introduction into the analytical chamber. Proper handling and preparation of specimens is particularly critical for dependable analysis. Improper handling of specimens can result in alteration of the surface composition and unreliable data.

0.2 ISO 20579-3 introduction

This document is specifically intended to assist analysts in the handling, preparation and mounting of specimens submitted for surface chemical analysis of biomaterials. Applications of synthetic materials in a body includes metals, ceramics, polymers, glasses, carbons and composite materials. Surface-analysis techniques such as AES, XPS and SIMS were originally developed for the analysis of inorganic materials, but the methods described in this document may also be applicable to biomaterials. Many other surface-sensitive analytical techniques such as ion-scattering spectrometry, low-energy electron diffraction and electron energy-loss spectroscopy can be applied for specimen analysis. A few examples of biomaterial applications are artificial hip and knee joints, bone plates for fracture fixation, dental implants, optical devices (intraocular lenses), heart valves and stents for cardiovascular systems, and membrane materials for guided tissue regeneration. More examples are discussed elsewhere[1],[2].

Specimen handling can influence surface-sensitive measurements. Surface methods for chemical analysis are sensitive to surface layers that are typically only a few nanometers in thickness. Such thin layers may be subject to severe perturbations caused by improper specimen handling[4],[7] or surface treatments that may be necessary prior to introduction into the analytical chamber. Proper handling and preparation of specimens is particularly critical for biomaterial analysis. Improper handling of specimens can result in alteration of the surface composition and unreliable data.

Proper preparation and mounting of specimens are particularly critical for surface chemical analysis of biomaterials. Improper preparation may result in the alteration of the surface composition and in unreliable analyses. Specimens are handled carefully so that the introduction of spurious contaminants is avoided or minimized. The goal prior to analysis is to preserve the state of the surface during preparation and mounting so that the analysis remains representative of the original specimen. This document describes methods that the surface analyst may need to use in order to minimize the effects of specimen preparation when using any analytical method.

In addition, the change of composition of the surface of a biomaterial before and after implantation may be an issue related to contamination. It is intended to highlight general ideas about surface chemical analysis, in particular solid surfaces but also soft surfaces, such as self-assembled monolayers (SAMs), hydrogels, scaffolds and some polymers.

1) To be developed.

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Surface chemical analysis — Sample handling, preparation and mounting —

Part 3: Biomaterials

1 Scope

This document gives guidance on methods of handling, mounting and surface treatment for a biomaterial specimen prior to surface chemical analysis. It is intended for the analyst as an aid in understanding the specialized specimen-handling conditions required for analyses by the following techniques:

— X-ray photoelectron spectroscopy (XPS or ESCA);

— secondary ion mass spectrometry (SIMS);

— Auger electron spectroscopy (AES).

The protocols presented are also applicable to other analytical techniques that are sensitive to surface composition, such as:

— attenuated total reflectance -Fourier transform infrared spectroscopy (ATR-FTIR);

— total reflection X-ray fluorescence (TXRF);

— ultraviolet photoelectron spectroscopy (UPS).

The influence of vacuum conditions applied and the issue of contamination before and after analysis and implantation, as well as issues related to contamination during analysis, are addressed. Biomaterials covered here are hard and soft specimens such as metals, ceramics, scaffolds and polymers.

This document does not cover such viable biological materials as cells, tissues and living organisms. Other related topics not covered in this document include: preparation of specimens for electron or light microscopy.

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 and the following 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

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— IEC Electropedia: available at http://www.electropedia.org/

3.1
biomaterial
material intended to interface with biological systems to evaluate, treat, augment or replace any tissue, organ or function of the body

3.2
biological material
organic and/or mineral substances produced by living organisms (e.g. bone, skin, seashell, woods, and cultured cells)

4 Abbreviated terms

AES	Auger electron spectroscopy
ATR-FTIR	attenuated total reflection - Fourier transform infrared spectroscopy
ESCA	electron spectroscopy for chemical analysis
HDPE	high density polyethylene
LDPE	low density polyethylene
pHEMA	poly (2-hydroxyethyl methacrylate)
PTFE	polytetrafluoroethylene
SIMS	secondary-ion mass spectrometry
SEM	scanning electron microscopy
TXRF	total reflection X-ray fluorescence spectroscopy
UHV	ultra-high vacuum
UPS	ultra-violet photoelectron spectroscopy
XPS	X-ray photoelectron spectroscopy

5 Explanation of the structure of this document

ASTM E1829 - 14 and ASTM E1078 – 14 provide specific information on specimen preparation techniques prior to surface analysis of materials, covering general considerations, mechanical separation, thinning versus removal of layers, removal from the substrate, and sectioning techniques[1],[2]. The growth of overlayers, solvents, chemical etching as well as ion sputtering, and plasma etching followed by sample heating and ultraviolet radiation is emphasized.

— Clause 6 provides specific recommendations on specimen handling procedures necessary to minimize contamination of the biomaterial specimen surface. Moreover, Clause 6 gives a series of alternative specimen handling procedures based on maintaining increasing degrees of specimen cleanliness during handling and transfer of the specimen to storage containers.

— Clause 7 discusses additional considerations, such as specimen history and previous analyses of the specimen that affect the composition of the surface. Documentation of these influences should accompany the carefully handled and packaged specimen when submitted for analysis.

— Clause 8 provides recommendations for sample handling and preparation with emphasis on tools, gloves and wiping materials. Exposure to gases, instrumental vacuum, electrons, ions and X-rays as well as the contamination of the analytical chamber is discussed.

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