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Surface chemical analysis — Sample handling, preparation and mounting —

Part 3: Biomaterials

Analyse chimique des surfaces — Lignes directrices pour la manipulation, préparation et montage des échantillons —

Partie 3: Biomatériaux

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Contents

Page

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 on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee 201, *Surface Chemical Analysis*, Subcommittee SC 2, *General procedures*.

The International Standards, ISO 18116 and ISO 18117, are replaced and merged into one new International Standard ISO 20579. Part 1 is a guide to handling, part 2 for preparation and mounting of specimen, part 3 (this document) as a guide for biomaterials handling, preparation and mounting of specimen for surface analysis, and part 4 as a guide for nano-objects history, preparation, handling and mounting.

Introduction

Common Introduction

This International Standard is intended to assist analysts and those seeking surface chemical analysis in the handling, storage, mounting and treatment of specimens. This is a multipart document, with the first two parts being general requirements for (Part 1) sample handling and storage, and (Part 2) mounting and treatment of samples. The ensuing parts combine any new requirements of sample handling/storage and/or sample mounting/preparation for new materials classes. Part 3 focuses on biomaterials analysis and handling, Part 4 focuses on reporting and handling needs for nano-objects. Each part of this International Standard can be used independently of the other parts, although, the general procedures described in Parts 1 and 2 are applicable to a wide range of materials and are not reproduced in detail in materials 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 International Standard 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.

ISO 20579:— **Part 3 Introduction**

This part of ISO 20579 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 Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS) and secondary-ion mass spectrometry (SIMS) were originally developed for the analysis of inorganic materials, the methods described in this International Standard are 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 biomaterials applications are artificial hip and knee joints, bone plates for fracture fixation, dental in plants, optical devices (intraocular lenses), heart valves and stents for cardiovascular systems as well as 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[1-4] or surface treatments that may be necessary prior to introduction into the analytical chamber. Proper handling and preparation of specimens is particularly critical for biomaterials analysis. Improper handling of specimens can result in alteration of the surface composition and unreliable data.

Proper preparation and mounting of specimens is particularly critical for surface chemical analysis of biomaterials. Improper preparation may result in the alteration of the surface composition and in unreliable analyses. Specimens have to be 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 International Standard describes methods that the surface analyst may need to use in order to minimize the effects of specimen preparation when using any.

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 but also soft surfaces, such as self-assembled monolayers (SAMs), hydrogels, scaffolds, and some polymers.

All the treatment and handling of biomaterials should be followed the local laws such as Federal Food, Drug, and Cosmetic Act (USA), Medicines Act 1968 (UK), Arzneimittelgesetz (Germeny) Pharmaceutical Affairs Act (Japan).

Surface chemical analysis — Sample handling, preparation and mounting —

Part 3: Biomaterials

1 Scope

This part 3 of the International Standard 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 techniques as follows,

— X-ray Photoelectron Spectroscopy (XPS or ESCA)

— Secondary Ion Mass Spectrometry (SIMS)

— Auger Electron Spectroscopy (AES).

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

— Attenuated Total Reflectance FTIR (ATR- FTIR),

— Total Reflection X-ray Fluorescence (TXRF),

— Ultraviolet Photoelectron Spectroscopy (UPS).

In particular instances, with particular specimens, further precautions for handling should be necessary. Biomaterials are hard and soft materials as metals or bones on one hand or polymers, hydrogels or scaffolds on the other hand.

The influence of vacuum conditions applied as well as the issue of contamination before and after analysis and implantation as well as issues related to contamination during analysis will be addressed. Biomaterials covered here are hard and soft specimens such as metals, ceramics, scaffolds or polymers. Soft materials need a different handling or sample preparation than hard materials.

Preparing whole mounts of biological specimens for imaging macromolecular structures by light and electron microscopy is reported in Mathieu et al[5]. A novel technique for preparation and analysis of the implant surface and its interface to bone is described in Gilmore et al[6]. Focused ion beam and transmission electron microscopy for the characterization in cross section of Titanium implant surfaces is reported by Jarmar et al[7]. However, this international Standard does not cover such viable biological materials such as cells tissues and living organisms.

2 Normative references

The following referenced documents are indispensable for the application 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 20579-1, *Surface chemical analysis —Guidelines to sample handling, preparation and mounting — Part1: Guide to handling of specimens prior to analysis*

ISO 20579-2, *Surface chemical analysis —Guidelines to sample handling, preparation and mounting — Part2: Guidelines to preparation and mounting of specimens prior to analysis*

ISO 20579-4, *Surface chemical analysis — Guidelines to sample handling, preparation and mounting — Part 4: Reporting information related to the history, preparation, handling and mounting of nano-objects prior to surface analysis*

3 Terms and definitions

For the purposes of this international standard, the terms and definitions given in ISO 18115 Parts 1and 2 regarding surface analysis apply along with the specific terms from the ISO/TS 80004- series of standards and standards 13527 and 27687 listed below:

3.1
Biomaterial
A Biomaterial is a material intended to interface with biological systems to evaluate, treat, augment or replace any tissue, organ or function of the body. A biomaterial is a nonviable material used in a medical device, intended to interact with biological systems[8]. A biomaterial is a material that is specially prepared and/or presented to exhibit bioacceptability and/or biocompatibility. (ISO 10993:2007, ISO 22803:2004).

3.2
Biological material
Biological materials are organic and/or mineral substances produced by living organisms (e.g. bone, skin, seashell, woods, and cultured cells) (ISO 18458:2015).

4 Symbols and abbreviated terms

AES	Auger electron spectroscopy
AFM	Atomic force microscopy
ATR	-FTIR Attenuated Total Reflection - Fourier Transform Infrared Spectroscopy
ESCA	electron spectroscopy for chemical analysis
SEM	scanning electron microscopy
SIMS	secondary-ion mass spectrometry
TXRF	total reflection X-ray fluorescence spectroscopy
UPS	ultra-violet photoelectron spectroscopy
XPS	X-ray photoelectron spectroscopy

5 Explanation of the structure of this Committee Draft (International Standard)

— Section 6 provides specific recommendations on specimen handling procedures necessary to minimize contamination of the biomaterial specimen surface. Moreover, section 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.

— Section 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.

— Section 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.

— Section 9 discusses specimen storage with respect to time, humidity, and temperature. Clause 10 also describes different specimen containers for biomaterials that may be used in different conditions.

— Section 10 emphasizes that specimen handling has an effect on the information derived from surface analytical measurements, and that specimen owners as well as analysts will benefit from improved analyses when prescribed specimen handling protocols are followed.

— Section 11 discusses specimen mounting procedures for various types of solid samples, i.e. powders, wires, and filaments. It provides information on pedestal mounting and reduction of thermal heating during analysis.

— Section 12 provides methods for reducing specimen charging, and in particular for using electron and ion beams for the analysis of biomaterials.

— Section 13 highlights specimen preparation techniques of biomaterials, such as sectioning techniques, using solvents and chemical etching, Ion sputtering is discussed in particular as well as the influence of UV radiation.

— Section 14 discusses fracturing, cleaving and scribing of samples.

— Finally, Section 15 on specimen handling techniques refers to part 2 where prepumping of gassy specimens, viscous liquids, and solute residues is discussed.

Part 1 and 2 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. The growth of overlayers, solvents, chemical etching as well as ion sputtering, and plasma etching followed by sample heating and ultraviolet radiation is emphasized.

6 General requirements and classes of specimens

6.1 General Information

General information on specimen handling of solid biomaterials is available in references[1-5, 8-13]. Biomaterials analysis requires special methods to control many of the biological reactions occurring in response to a biomaterial. Functionality, biocompatibility and durability are of concern. Some information for solid surfaces is found in[2]. Contamination is a key question. The degree of cleanliness in particular for biomaterials required by surface-sensitive analytical techniques is much higher than for many other forms of analysis[1]. Specimens and mounts must never be in contact with the bare hand. Handling of the surface to be analysed should be eliminated or minimized whenever possible. Fingerprints contain mobile species that may contaminate the surface of interest. Hand creams, skin oils and other skin materials are not suitable for high vacuum.

6.2 Handling

Care shall be taken in the handling of biomaterials to ensure that nothing, apart from air or clean inert gases, comes in contact with the surface to be investigated. In particular, avoid contacting the specimen surface with solvents or cleaning solutions, gases such as compressed air or solvent vapours, metals, tissue or other wrapping materials, tape, cloth, tools, packing materials, or the walls of containers. In response to hydrophobic environment components may migrate to the surface of the specimen, thus reducing interfacial energy. Responding to an aqueous environment, the surface may reverse its structure and point polar (hydrophilic) groups outward to interact with the polar water molecules. Many materials can undergo a reversal of surface structure when transferred from air into a water environment. A hydroxylated polymer, as a pHEMA contact lens exhibits a surface rich in methyl groups