
**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**

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

*Partie 4: Exigences de rapport sur les nanomatériaux, défis en matière
d'analyse et méthodes d'extraction des solutions*

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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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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.

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Introduction

Introduction to the ISO 20579 series

This 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 document, with the first two parts being general requirements for sample handling and storage in ISO 20579-1, and mounting and treatment of samples in ISO 20579-2. The ensuing parts combine new requirements of sample handling/storage and/or sample mounting/preparation for new materials classes. ISO 20579-3 focuses on biomaterials and ISO 20579-4 focuses on reporting needs for nano-objects. Each part of this document 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 the materials-specific documents.

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 document are also applicable to many other surface-sensitive analytical techniques such as ion-scattering spectrometry, scanning probe microscopy, 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 might be subject to severe perturbations caused by specimen handling or surface treatments that could 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.

Introduction to this document

Although all types of samples requiring surface analysis need thoughtful preparation, as noted in ISO 20579-1 and ISO 20579-2^[1], nano-objects present additional challenges in order to avoid artefacts due to the handling and preparation of materials prior to analysis^[2]. The types of procedures described in ISO 20579-1 and ISO 20579-2 apply generally to nanomaterials, but because of the nature of nano-objects it is important to carefully document how these and other procedures are implemented. This document indicates the minimum information regarding sample preparation that needs to be reported about the handling and preparation for surface analysis that should become part of sample provenance information to help assure the reliability and usefulness of data obtained from surface-analysis methods^[3]. Informative [Annex A](#) provides a background to some unique aspects of nano-objects that amplify the reporting needs. Informative [Annex B](#) provides an overview of practices used by research groups around the world to extract particles from solution in preparation for surface chemical analysis, and [Annex C](#) shows an example of a sample data form. Although focused on surface chemical analysis of nano-objects, many issues apply to nanomaterials more generally.

Nanomaterials include both materials with their internal or surface structures in the nanoscale, i.e. nanostructured materials, and objects with one or more external dimensions in the nanoscale, i.e. nano-objects. Nano-objects, in particular, present a range of characterization challenges that have the potential to inhibit or delay the scientific and technological impacts of nanoscience and nanotechnology^[4-10]. The standardization of these characterization methods is led by ISO TC 229 (nanotechnologies) with many standards on particle size measurement produced by ISO TC 24/SC 4. Because nano-objects are comprised to a large degree of surfaces and interfaces, the importance of adequate characterization of their surfaces and interfaces has been highlighted by many^{[4][11]} and the roles of surface chemical analysis methods for nanomaterial characterization are discussed in ISO/TR 14187^[12].

Many nano-objects are produced and stored in conditions far from a state of equilibrium, making them particularly susceptible to change as a function of time, upon exposure to different environments, during handling and when subjected to different measurements^[4]. Seemingly minor variations in synthesis, age or source of precursor chemicals, processing or storage have been found to produce materials with significantly different properties or lifetimes^[13-15]. The large impact of such minor changes complicates the ability of experiments to be reproduced and emphasizes the importance of sample history in providing complete information about a sample and the impacts of analysis. These types of issues have led the Organisation for Economic Co-operation and Development (OECD) to prepare a