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Surface chemical analysis — Sample handling, preparation and mounting —

Part 1: Documenting and reporting the handling of specimens prior to analysis

Analyse chimique des surfaces — Manipulation, préparation et montage des échantillons —
Partie 1: Documentation et notification des données de manipulation des échantillons avant analyse

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Foreword

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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 document 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*.

This first edition of ISO 20579-1 cancels and replaces ISO 18117:2009.

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.

Introduction

0.1 Introduction to the ISO 20579 series

The handling and preparation of samples for surface analysis can physically or chemically alter the surface. Therefore, reliable surface analysis depends upon knowing the analysis objectives and knowledge of the sample history including aspects of how the sample has been prepared, stored, processed, and handled prior to and during analysis. The ISO 20579 series describes the information that needs to be collected and included as part of the sample history (sample provenance information). Both ISO 20579-1 and ISO 20579-2 describe information to be recorded regarding sample handling, and storage. This document describes information needed regarding sample selection, handling, and preparation when requesting surface analysis. ISO 20579-2 provides information about sample handling, preparation, mounting and processing to be reported by an analyst. ISO 20579-3 and ISO 20579-4 focus on specific handling and reporting needs associated with nanomaterials (ISO 20579-4) and biomaterials (ISO 20579-3). Each part of this series can be used independently of the other parts, although the general reporting requirements described in this document (ISO 20579-1) and in ISO 20579-2[1] are applicable to a wide range of materials and are not reproduced in ISO 20579-3 and ISO 20579-4.

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 thick. Such thin layers can be subject to severe perturbations caused by specimen handling or surface treatments that can 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.[2][3]

0.2 Introduction to this document (ISO 20579-1)

This document is intended for the specimen owner or someone requesting surface analytical services. It describes the minimum information regarding the analysis objectives and sample preparation that an analyst needs to know to determine if and how the desired information can be obtained. This information becomes part of sample provenance record to help validate the reliability and usefulness of data obtained from surface-analysis methods.[4]

Surface analysis methods measure the outer atomic layers of a specimen surface which can be inadvertently altered by inappropriate handling or preparation. Therefore, the degree of care and cleanliness required by surface-sensitive analytical techniques is usually much greater than for many other analysis methods. Appropriate careful sample selection, preparation and storage are essential for reliable surface analysis and the documentation and reporting of this information is critical to the ability to assess the validity of surface analysis information.

Although the categories of needed reporting are similar for all specimens, the details of the required sample handling can vary depending on the nature of the sample and analysis objectives. Annexes to this document and references therein provide background information useful to assist in identification of the necessary sample preparation, handling, storage, and transport requirements that maximize the ability for obtaining the desired information.

Annex A identifies three categories of analysis objectives and provides an overview of the challenges associated with sample preparation for surface analysis in the context of each desired objective. Included is a table summarizing relevant sample handling methods and types of specimen containers needed for the three types of analysis objectives and is intended to help those requiring surface analysis. Annex B discusses common sources of contamination and issues along with methods to minimize contamination related to sample handling. Annex C discusses topics related to sample storage and transportation.

Surface chemical analysis — Sample handling, preparation and mounting —

Part 1: Documenting and reporting the handling of specimens prior to analysis

1 Scope

This document identifies the information needed to ensure that a sample has been selected, processed, handled, and stored in a manner consistent with the analysis objectives, and to ensure the reliability and reproducibility of the surface analyses. Such information is also an important component of sample data record books, datasheets, certificates of analysis, reports, and other publications. This information is in addition to other details associated with the specimens to be analysed, such as source/synthesis information, processing history, and other characterizations that naturally become part of the data record (sometimes referred to as provenance information) regarding the origin of the sample and any changes to its original form.

This document also includes normative annexes as an aid to understanding the special sample handling techniques and storage requirements of surface chemical analysis techniques, particularly: Auger electron spectroscopy (AES), secondary ion mass spectrometry (SIMS), and X-ray photoelectron spectroscopy (XPS). The information presented can also be applicable for other analytical techniques, such as total reflection X-ray fluorescence spectroscopy (TXRF), that is sensitive to surface composition, and scanning probe microscopy (SPM), that is sensitive to surface morphology.

This document does not define the nature of instrumentation or operating procedures needed to ensure that the analytical measurements described have been appropriately conducted.

2 Normative references

The following documents are referred to in the text in such a way that some 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 terminology 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

AES	Auger electron spectroscopy
AFM	atomic force microscopy
ALD	atomic layer deposition
I.D.	identification
ISS	ion-scattering spectroscopy
PTFE	polytetrafluoroethylene
SEM	scanning electron microscopy
SIMS	secondary ion mass spectrometry
SPM	scanning probe microscopy
TEM	transmission electron microscopy
TXRF	total reflection X-ray fluorescence spectroscopy
XPS	X-ray photoelectron Spectroscopy

5 Information to be documented and accompany a sample for analysis

5.1 General sample handling requirements

The generic sample handling protocols identified in 5.1 shall be followed to maintain the stringent cleanliness required for meaningful surface analysis.[5][6] The protocols are further described and justified in Annex B and shall be carried out in accordance with B.2.2 and B.2.3. Any exceptions or deviations shall be documented and discussed in advance with the analyst.

Specifically, avoid touching the surface to be analysed with any material. This includes tools, hands, and containers. Avoid, to the extent possible, adventitious contact from gases, liquids, particulates, or outgassing materials near the surface or present in the environment.

Thoroughly document all cleaning processes. Be extremely careful of any cleaning processes to make sure they do not alter the sample surface. Be very careful to use only clean, pure, non-reactive gases (never blow on the sample with your mouth!) and delivery systems (including lines, nozzles, etc.) if required to dust off particulates.

If smaller samples need to be prepared for analysis, thoroughly document any cutting or sectioning procedures, along with any associated cleaning. It is best to consult with the analyst in advance on how best to prepare these samples. Consider having the analyst perform any such procedure. Justifications for these measures and guidance for specific analysis objectives are described in Annex A, which also includes a table identifying typical sample handling requirements in relation to the information desired. Information about approaches needed to minimize sample contamination and information damage is provided in Annex B, and information about sample storage and transport requirements in Annex C.

Other sample handling details, based on the guidance in the annexes, shall be reported as indicated in 5.2 and 5.3.

EXAMPLE 1 Because of the small sample size, touching the surface to be analysed with the cleaned mounting tools was unavoidable. However, contact was minimized to the extent possible.

EXAMPLE 2 Sample obtained for failure analysis received unknown handling before it was submitted to the laboratory for detailed study.

EXAMPLE 3 Sample surface might have been contacted by an ungloved hand during collection.

5.2 Nature of sample, analysis objectives and any special requirements

The type of sample and the analysis objectives, considering the hierarchy described in Annex A, shall be recorded, and reported as they provide critical information related to the detailed processes an analyst needs to consider in undertaking surface analysis measurements.

EXAMPLE 1 Thin film of strontium titanate to be examined for surface contamination.

EXAMPLE 2 As received battery electrode to be examined for composition before electrochemical cycling. Needs to be handled in controlled atmosphere and analysed soon after receipt.

EXAMPLE 3 Catalyst particles to be examined to determine compositional and chemical state changes occurring after surface activation using the processing system connected to the analysis chamber.

EXAMPLE 4 Section cut from corroded metal plate. Areas identified in optical photograph are to be examined for compositional abnormalities.

EXAMPLE 5 ALD layers of Alumina to be examined for thickness and purity.

5.3 Sample identification and provenance information

Each sample shall have a unique identifier (sample I.D.), and the area of interest for analysis shall be indicated. In accordance with the guidance in Annexes A, B and C, information related to the history, handling, storage, and processing prior to surface analysis shall be provided.

a) Sample source, identifier/designation, and other useful identifying information.

EXAMPLE 1 Sample 1 was an area of high damage cut from a corroded metal section using cleaned scissors; Sample 2 is a section of uncorroded metal of the same material.

EXAMPLE 2 Samples a, b, and c are three types of catalyst powders before activation, samples d, e and f are after activation and need to be handled in a controlled environment.

EXAMPLE 3 Single crystal of $TiO2_2$ purchased from ACX crystal sources, serial number 12584, month/day/year.

b) Other information regarding the selection and handling prior to submission for surface analysis.

EXAMPLE 1 Selected samples with and without damage were selected for analysis.

EXAMPLE 2 The sample was rinsed with solvent isopropyl alcohol to remove organic surface contamination.

EXAMPLE 3 Sample was heated to 400 °C in oxygen to form a corrosion layer.

EXAMPLE 4 Coupon was selected from others synthesized by ALD and handled using cleaned metal tools.

c) Information regarding any analyses prior to surface analysis (recognizing that some other types of analysis can alter the surface, see B.3).

EXAMPLE 1 Optical images of sample were collected to identify areas for surface analysis.

d) Information regarding storage time, containers, and transportation, in accordance with Annexes A and C.

EXAMPLE 1 Section of sample extracted from field corroded specimen on July 10, 2020, handled by cleaned tools with gloved hands and placed in a cleaned glass tube that made no contact with the surface to be analysed.

EXAMPLE 2 Powders were packed in an argon filled sealed container.

EXAMPLE 3 Two sections of same sample were packed face to face in a clean glass container.

Annex A
(normative)

Overview of issues and methods related to sample handling

A.1 Analysis objectives

A.1.1 Types of analysis objectives and related sample handling requirements

Surface chemical analysis can be performed on a wide range of specimens and multiple approaches can be used to obtain very different types of information about surfaces or interfaces. The degree of care that needs to be taken depends upon the type of analysis that is required and the nature of the problem. The information being sought usually falls into three general categories.

a) Analysis objective type 1 - information requiring integrity of the outermost surface.

b) Analysis objective type 2 - information as a function of depth (depth profile) or at a buried interface.

c) Analysis objective type 3 - information that will require subsequent specimen preparation by the analyst, including bulk analysis or results from some type of sample processing.

Independent of the specific analysis objective, minimizing contamination of the surface of any sample to undergo surface analysis is an essential requirement which places important requirements on sample selection, handling, storage, transport, and related documentation. Some of the specific requirements related to analysis objectives are indicated in this annex. General sample handling requirements are briefly discussed in Annex B, while storage and transport issues are discussed in Annex C. Additional information related to the specific analysis objectives and useful methods can be found in the clauses indicated, in ASTM E 1829,[7] in a paper by Stevie et al.[6] and book chapters by Lindfors[5] and Geller.[2]

Sometimes very special sample requirements are needed to obtain the desired information. Examples include the analysis of catalysts after activation and analysis of soils or other environmentally relevant materials from non-ambient environments. It is useful to discuss needs and opportunities with the relevant surface analysts for their input and guidance before submitting such samples for surface analysis.

A.1.2 Objective type 1

Objective 1 specimens include those to be investigated for surface contamination, surface organic coatings, biomaterials - except live organisms (cells, bacteria, etc.), surface stains, semiconductors, adhesion failures, etc. Two types of samples can fit into this category, those with highly reactive surfaces that need to be handled in controlled environments, and those for which the ambient-exposed surface needs to be analysed in the as received condition. This category requires the most care in preparation and packaging. Nothing should be allowed to contact the surface of interest. If certain elements are to be analysed at low levels, ensure that, as far as possible, those elements are not contained in any handling tools, gloves, or container materials. Type 1 specimens are described in the first two rows in Table A.1.

Types of specimens that fit Objective Type 1.

a) Reactive specimens where the reactive surface is to be analysed, without special processing by the analyst or in the instrument, although a reactive surface might be handled in a protective or anaerobic environment to minimize additional reaction.

b) Specimens with hydrocarbons, molecular films, or biomaterials on the surface that are the objective of the analysis.

c) Specimens with a contamination layer that is the object of the analysis.