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Surface chemical analysis — Sample handling, preparation and mounting —

Part 2: Documenting and reporting the preparation and mounting of specimens for analysis

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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 TC 201, *Surface Chemical Analysis*, Subcommittee SC 2, *General Procedures*.

This first edition of ISO 20579-2 cancels and replaces ISO 18116:2005, which has been technically revised.

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 General introduction to the ISO 20579 series

Because sample preparation and handling can have a significant impact on the physical and chemical properties of a sample surface, reliable surface analysis depends upon knowing the analysis objective 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 specifies information that is required to be collected and included as part of the sample history (sample provenance information). The ISO 20579 series describes information that anyone seeking surface analysis is required to provide to an analyst[2] and additional information that an analyst is required to include in the sample provenance record regarding sample handling, storage, and processing.[3] ISO 20579-1 and ISO 20579-2 describe the information to be recorded regarding sample selection, handling, and storage. ISO 20579-1 describes information that is necessary for the sample provenance record and an analyst regarding sample selection and preparation when requesting surface analysis. ISO 20579-2 indicates information about sample handling, preparation, mounting and processing to be recorded and reported by the analyst. ISO 20579-3 and ISO 20579-4 focus on specific reporting requirements associated with biomaterials[5] and nanomaterials,[4] respectively. Each part of the ISO 20579 series can be used independently of the other parts, although the general reporting requirements described in ISO 20579-1 and ISO 20579-2 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 (ISS and including low- and medium-energy scattering LEIS, MEIS), scanning probe microscopy (SPM), low-energy electron diffraction (LEED) and electron energy-loss spectroscopy (EELS), 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.[6][7]

0.2 Introduction to ISO 20579-2

This document is intended for the analyst and describes information that is required to be recorded and reported regarding the sample handling, storage, mounting and other aspects of preparing a sample for surface analysis. This information becomes part of sample provenance record to help validate the reliability and usefulness of data obtained from surface-analysis methods.[8]

Although the categories of necessary 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. When the outer surface of a specimen is to be analysed the specimen needs to be handled carefully so that the introduction of spurious contaminants is avoided or minimized. The goal is to preserve the state of the surface during preparation and mounting so that the analysis remains representative of the original specimen. In other cases, sample processing is required to enable access to the surface or interface to be analysed and some aspects of the sample handling might be less stringent. In all cases, the nature of sample handling and preparation for the desired analyses need to be recorded and reported.

Normative annexes to this document describe methods that the surface analyst can use to minimize the effects of specimen preparation when using any surface-sensitive analytical technique. Annexes also describe methods to mount specimens to ensure that the desired analytical information is not compromised. Annex A describes approaches, issues, and good practices regarding sample handling in preparation for analysis. Annex B provides information about sources of contamination, sample handling and storage requirements for differing analysis objectives.

Surface chemical analysis — Sample handling, preparation and mounting —

Part 2: Documenting and reporting the preparation and mounting of specimens for analysis

1 Scope

This document specifies information to be reported by an analyst in a datasheet, certificate of analysis, report or other publication regarding the handling, preparation, processing and mounting of specimens for surface analysis. Appropriate sample handling with adequate documentation is needed to ensure and assess reliability and reproducibility of analyses. Such information is in addition to other details associated with specimen synthesis, processing history and characterization, and should become part of the data record (sometimes identified as provenance information) regarding the source of the material and changes that have taken place since it was originated.

This document also includes normative annexes that summarize important processes and common approaches relevant to sample preparation and mounting for surface analysis. The descriptions of procedures for which records and reporting are required follow the steps that an analyst would follow from receiving the samples, to cleaning or processing outside of the analysis chamber, sample mounting and then treatments in the analysis chamber. The descriptions of the processes and their implications are intended as an aid for the analyst in understanding the reporting requirements for the specialized sample-handling conditions and approaches required for analyses by techniques such as Auger electron spectroscopy (AES), secondary-ion mass spectrometry (SIMS), and X-ray photoelectron spectroscopy (XPS). The methods described are also applicable for other analytical techniques, such as total reflection X-ray fluorescence spectroscopy (TXRF), low energy electron diffraction (LEED), some types of scanning probe microscopy (SPM) including atomic force microscopy (AFM) and scanning tunnelling microscopy (STM), ultra-violet photoelectron spectroscopy (UPS) and medium- and low-energy ion scattering (MEIS and LEIS [also called ion surface scattering, ISS]) that are sensitive to surface composition.

This document does not specify the nature of instrumentation, instrument conditions (e.g., calibration or vacuum quality), or operating procedures required 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
EELS	electric energy-loss spectroscopy
ESCA	electron spectroscopy for chemical analysis (alternate name for XPS)
FIB	focused ion beam
ID	Identification
ISS	ion-scattering spectroscopy
LEED	low-energy electron diffraction
LEIS	low energy ion-scattering
MEIS	medium energy ion-scattering
PTFE	polytetrafluoroethylene
SIMS	secondary ion mass spectrometry
SPM	scanning probe microscopy
STM	scanning tunneling microscopy
TXRF	total reflection X-ray fluorescence spectroscopy
UPS	ultraviolet photoelectron spectroscopy
XPS	X-ray photoelectron spectroscopy

5 Provenance information to be collected or retained

5.1 Information record

Clause 5 deals with a sample information record that includes the relevant sample history, sample handling requirements, and analysis objectives. This information is usually provided by those requesting analysis. If it is not provided with the sample, it will need to be created (see 5.2).

Surface analysis is usually undertaken to collect useful information relevant to a sample for a specific reason at specific stages during the lifetime or history of the material. To assess the reliability and usefulness of the analysis, it is important to retain as many relevant sample history and handling details that are available to maintain the provenance[8][9][10] of the sample and data related to them.

Samples are often provided to an analyst by someone seeking information about one or more samples. Such samples should arrive with a history and the information described in ISO 20579-1 about the nature of the sample, the analysis objective, and any special requirements (ISO 20579-1:2024, 5.2), and with unique sample identifiers (IDs) and information, including dates, about previous handling, storage, and processing as relevant to the analysis objectives (ISO 20579-1:2024, 5.3).[2] Information about different types of

analysis objectives and the implications for sample handling are provided in ISO 20579-1:2024, Annex A and summarized in Annex B of this document. Detailed information records are especially important for nano-objects as described in ISO 20579-4:2018, Clauses 4 and 5.[4]

Information that an analyst shall record and add to the information record regarding the further preparation and handling of samples for surface analysis are described in Clause 6 of this document. This information, along with data collected becomes part of the information record that provides the history of the physical and chemical processes used on a sample that would allow assessment and replication of the measurements. Appropriate information to be retained and passed along with analysis information will vary depending on the nature and history of the sample and the analysis objectives as described in Clause 6. Dates should be provided whenever possible throughout the provenance record.

5.2 Verification or generation of sample information and analysis objectives

When an analyst receives one or more specimens, a necessary step is to examine the sample documentation, or establish it (with the owner) if not provided, including the nature of the sample(s), clear sample IDs, and appropriate analysis objectives. It is also important to determine if the samples have been handled properly to enable appropriate surface analysis and if relevant, that information about specific analysis areas or regions of analysis interest have been identified and documented.

If this information was not provided, the analyst shall assemble as much information as possible to establish a complete information record and analysis plan that will determine the sample handling and preparation necessary to obtain the desired information from the sample(s).

A visual inspection (documented) of each sample is important to verify information, sample condition and identification of any special features or problems such as fingerprints, adhesive, unexpected particles, or contaminants.

6 Information about sample handling and preparation for analysis to be documented and added to the sample information record

6.1 General

Information about the following topics shall be recorded and reported as part of the sample information record.

6.2 Adherence or exceptions to the general sample handling requirements

To maintain the stringent cleanliness required for meaningful surface analysis the general sample handling protocols listed below and in A.1.1 and B.3.2 shall be followed.[7][11] These generic requirements also appear in ISO 20579-1, 2024, B.2.2 and B.2.3. Any exceptions or deviations shall be documented. Justification for these measures and further details are provided in the Annexes of ISO 20579-1 and this document. Annex A of this document gives some additional details about general considerations for sample handling to minimize contamination and is summarized here.

Avoid touching the sample surface to be analysed with any material, including tools, hands, and containers, as well as adventitious contact from gases, liquids, particulates, or outgassing materials near the surface or present in the environment. If possible, air sensitive samples should be introduced using a glove box or a transfer vessel and documented in the reporting.

Thoroughly document all cleaning processes. Be extremely careful with any cleaning processes to make sure they do not alter any aspect of the sample surface important to the analysis objectives (for additional information see A.3.2). Be very careful to use only clean, pure, non-reactive gases (never blow on the sample by mouth) and delivery systems (including lines, nozzles, etc.) if required to dust off particulates. Note that canned air often contains fluorinated propellants which should be avoided.

If smaller samples must be prepared for analysis, thoroughly document any cutting or sectioning procedures, along with any associated cleaning (see A.3.3 for additional information).

Minimizing contamination also requires using cleaned sample handling tools and fixtures involved in sample mounting. It is also relevant to consider if volatile or otherwise mobile contaminants (e.g., Zn, Na, F) from previous samples in the vacuum system or adjacent during sample handling or storage could introduce contamination be detrimental to the desired analysis.

Example descriptions of exceptions or issues related to general sample handling requirements are given in EXAMPLES 1 to 3.

EXAMPLE 1 Because of the small sample size, touching the surface to be analysed with a clean mounting tool was unavoidable.

EXAMPLE 2 Although the analysis chamber and entry system were processed/cleaned between samples, we note that the most recent samples contained fluorine. Therefore, any fluorine identified on the current sample should be viewed with caution.

EXAMPLE 3 Carbon tape was used to mask the sample for charge control. The tape was within the sputter area, so during sputtering, it was redeposited onto the sample surface, resulting in carbon contamination.

6.3 Description of ex situ sample handling

Based on sample information and analysis objectives, the analyst shall determine and report the steps followed to store or prepare the samples for analysis, including any storage, cutting, sectioning, polishing, cleaning, or other preparation before insertion into the analysis chamber in accordance with A.3 (for ex situ handling) and B.3 and B.4 (for handling and storage).

Example descriptions of sample storage and ex situ treatments are given in EXAMPLES 1 to 6.

EXAMPLE 1 As received samples were placed in a desiccator where they were stored for one week before analysis.

EXAMPLE 2 To facilitate AES analysis of the layered structure the sample was polished by angle lapping.

EXAMPLE 3 To minimize sample charge buildup during AES and XPS analysis, the sample was thinned by focused ion beam milling.

EXAMPLE 4 Section was cut from corroded metal plate using a cleaned hack saw blade. Areas to be analysed were identified in an optical photograph.

EXAMPLE 5 Metal samples were machined so that they could be fractured in the analysis chamber to determine grain boundary composition after fracture.

EXAMPLE 6 MgO sample was heated to 800 C in air to remove organic contamination and moisture, and then was inserted into the intro chamber within 30 seconds, before the surface temperature reached 200 C.

6.4 Method of mounting samples for analysis

The analyst shall report details of the approach to sample mounting, which depend on the sample type, the instrument, analysis objectives, and the need for any special environmental control or in situ processing in accordance with A.4.

Example descriptions of sample mounting are given in EXAMPLES 1 to 5.

EXAMPLE 1 Sample was mounted directly onto a specimen holder using a spring clip to ensure good connectivity to spectrometer ground.

EXAMPLE 2 Potentially insulating sample was mounted for XPS analysis using double sticky tape, making sure the sample was isolated from the specimen holder so that surface potential could be controlled by the charge neutralization system.

EXAMPLE 3 A portion of the particles of this sample were pressed into indium foil to enable AES analysis of individual particles.

EXAMPLE 4 A solution of nanoparticles was deposited on a silicon wafer. Multiple deposits were made until the substrate was covered. It will be tested to determine that no signal arises from the substrate during XPS analysis.

EXAMPLE 5 Liquid sample was deposited on a LN_2 cooled substrate.

6.5 In situ sample cleaning or other sample preparation or processing

The analyst reports details of any in situ cleaning, and other sample preparation and processing of the sample prior to analysis, including any methods used to expose the region of analysis, in accordance with A.5.

Example descriptions of in situ cleaning and processing are given in EXAMPLES 1 to 6.

EXAMPLE 1 The high vapor pressure sample was degassed by (pre)pumping in the entry chamber (or an auxiliary vacuum system) before insertion into the main analysis chamber.

EXAMPLE 2 The sample was sputter cleaned to remove the thin surface oxide layer using low energy Ar^+ sputtering (0,5 kV). The sputter time for the sputter conditions would have removed approximately 10 nm of SiO_2.

EXAMPLE 3 Using a 20 kV focused Ga ion beam in the AES system, a cross section of the sample was created to enable analysis of the layered structure.

EXAMPLE 4 The sample was scribed inside the system to expose fresh surface for analysis.

EXAMPLE 5 The machined sample was fractured in the liquid nitrogen cooled impact fracture unit to expose grain boundaries for analysis.

EXAMPLE 6 An argon cluster ion source was used to remove organic contamination from the sample surface before analysis (Ar_{1000} at 4 kV). An ion current density of X was applied for 30 seconds (or specify the equivalent sputter removal of X nm of a reference material such as irganox).

6.6 Post analysis handling and storage

The analyst reports the disposition of samples after analysis, calling attention to any specific handling or storage of samples potentially relevant to later analysis or use.

Example descriptions of post analysis sample handling or storage are given in EXAMPLES 1 to 4.

EXAMPLE 1 Samples were discarded after analysis.

EXAMPLE 2 Samples were returned to dry box storage.

EXAMPLE 3 Samples were archived at location ABC.

EXAMPLE 4 Samples were transported to lab XYZ for further characterization.