



**International  
Standard**

**ISO 20579-1**

**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

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 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](http://www.iso.org/directives)).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at [www.iso.org/patents](http://www.iso.org/patents). ISO shall not be held responsible for identifying any or all such patent rights.

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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](http://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<sup>[1]</sup> 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.<sup>[2][3]</sup>

## 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.<sup>[4]</sup>

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.

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# 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.<sup>[5][6]</sup> 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  $\text{TiO}_2$  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,<sup>[7]</sup> in a paper by Stevie et al.<sup>[6]</sup> and book chapters by Lindfors<sup>[5]</sup> and Geller.<sup>[2]</sup>

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.

- d) Specimens that have been exposed to the atmosphere and are to be analysed as received.

### A.1.3 Objective type 2

Objective 2 specimens include those that require the investigation of thick or thin films, single layers, multilayers, metal contact layers on semiconductors, coatings, dopant profiles, and the chemical and physical properties at an interface. For such samples the information sought comes from a layer below the outermost surface and identification of superficial surface contamination is not the primary goal of the analysis. Consequently, the sample handling and packaging requirements are usually not as stringent for those where the information is on the outer surface. Care is still required not to introduce contamination to the specimen since surface diffusion of contaminants could compromise interpretation of the results. Because the objective concerns material below the outer surface, these samples often require some treatment (such as depth profiling) to expose the region of interest. Care is necessary to avoid carbonaceous and particulate contamination of the surface as these can degrade the quality of depth profiles and other processes. Type 2 specimens are in the third row of [Table A.1](#).

Types of specimens that fit analysis Objective Type 2.

- e) Specimens with atmospheric adsorbates that might interfere with analysis.
- f) Specimens with a contamination layer (or other topmost layer) that is of no interest and that will be removed just prior to insertion in the analytical chamber (e.g., treatment by solutions, abrasion, plasma, exposure to radiation, etc.).
- g) Specimens with a contamination layer (or other topmost layer) that is of no interest and that will be removed inside the analytical chamber.

### A.1.4 Objective type 3

Objective 3 specimens include those that require preparation by the analyst or other special handling to get at the desired information. Examples include specimens for *in situ* fracture, metallurgical lapping or polishing, and specimens that are part of a larger assembly. Generally, these specimens need to be shaped (e.g., for fracture), chemically or mechanically altered (as happens with lapping) or disassembled. Few special precautions are needed for samples that are to be fractured or undergo further sample preparation by the analyst. For specimens in a larger assembly or subassembly, it might be preferable to leave the specimen in place and let the analyst remove it prior to analysis. Nonetheless, care must still be taken not to contaminate the specimen. Type 3 specimens are in the fourth (last) row of [Table A.1](#).

Examples of specimens that fit analysis Objective Type 3.

- h) Thin films that will be delaminated by the analyst prior to insertion into the analysis chamber.
- i) Specimens that will be fractured or freshly prepared outside the analysis chamber, including materials prepared in a controlled atmosphere.
- j) Uniform thin films that are to be removed by ion etching or scraping in the analysis chamber to expose a layer or interface of interest.
- k) Samples that will be fractured *in situ*.
- l) Materials where the information on the bulk properties is desired (any surface layers might need to be removed).

## A.2 Overview of relationships between objectives and sample handling and storage requirements

The minimum handling and specimen container requirements for different categories of information needs are summarized in [Table A.1](#).

Table A.1 — Minimum handling methods and specimen containers for different analysis objectives

Sample analysis objective type	Information sought		Handling method		Specimen container	
	A.1.2 through A.1.4 Specimen type	Specimen category/depth of Information	B.2.3 Sample handling method	Method	C.3.2 Container type	Container
1	a	Reactive specimens for which analyses are to be as close to original condition as possible.	a	Often handled in a specialized environment using clean non-magnetic, uncoated stainless steel or specialty tools only, handled using polyethylene gloves.	a, b	Argon or nitrogen glove box or vacuum transfer vessel. Two flat specimens, face-to-face, sealed with PTFE tape.
1	b, c, d	Specimens requiring surface hydrocarbon, molecular, contaminant, or ambient surface layer analysis (e.g., static SIMS and XPS analyses).	b	Clean non-magnetic, uncoated stainless-steel tweezers or grippers only, handled using polyethylene gloves.	c, d, e	Clean glass with glass, PTFE tape, or clean Al foil stopper. High quality polypropylene wafer holder
2	e, f, g	Specimens where the surface of interest is obscured by a surface contamination layer from handling or environmental exposure	c	Powder-free, polyethylene disposable gloves holding specimen by edge. Powder-free, silicone-free, latex gloves holding specimen by edge.	f, g	Any of the above Clean Al foil. Polyethylene box or bag.
3	h, i, j, k, l	Specimens with buried interfaces or layers are of interest, fracture specimens, bulk analysis.	d, e	Clean tools or handheld by edges with gloves. Acid-free, lint-free paper to hold specimen by edge.	h	Any of the above Acid-free, lint-free, paper

## Annex B (normative)

### Critical information about sample handling and order of analyses to minimize contamination

#### B.1 Potential Sources of contamination during handling

Because of the sensitivity of surface analysis methods, there are many different actions or processes that can contaminate a sample during selection or handling including the following:

- a) Handling a sample with bare hands, even without touching the surface to be analysed, is to be avoided. Fingerprints and hand creams have molecules that can migrate and contaminate a surface of interest.
- b) Handling a sample with tools that have not been solvent cleaned will transfer and spread contamination to a surface. Tools with high Ni-content have been found to contaminate silicon. Use of nonmagnetic tools is also important to both avoid magnetizing a sample and having it move in unintended ways during mounting for analysis.
- c) Any unnecessary contact with a sample is to be avoided if possible. Powder-free and silicone-free gloves are useful in handling clean tools but are not to be used to contact the sample unless absolutely essential.
- d) Blowing on a sample by mouth is totally unacceptable for removal of unwanted particulates. Many sources of compressed gas also contain unwanted contaminants. High purity non-reactive gas sources are sometimes used to remove dust from a sample, but verification that the source, and delivery hardware (such a tubing or nozzles) do not contaminate a clean sample should be conducted.
- e) Storing samples alongside materials which outgas (including storage containers) will contaminate the sample (this is discussed further in [Annex C](#)).

#### B.2 Suggested handling procedures to minimize contamination

##### B.2.1 Generic and special sample handling:

The extent/level of special handling depends on several factors including the condition of the surface, depth from the surface of the information being sought, and the detection level needed to get the desired information. Generic sample handling recommendations that are useful for most samples are given below, followed by more specific recommendations.

Special precautions are needed for samples that can contain toxins or other hazardous materials, and MSDS data sheets should be provided to the analyst for this type of sample.

##### B.2.2 Important generic sample handling recommendations:

Specimens should never be in contact with the bare hand. Eliminate or minimize contact with the sample surface to be analysed with handling tools or other equipment.

Specimens need to be transported to the analyst in a container that does not come into direct contact with the surface of interest (see [Annex C](#) for more details on sample storage).

In some cases, it is necessary to take a representative sample from the specimen. Selection of a smaller sample from a larger specimen requires consideration of the information being sought because inhomogeneities are often present. It is recommended that this choice be made in consultation with an experienced analyst. Specific care is needed to avoid contaminating the surface of interest during the cutting procedure. It is

often better to request that the analyst perform any cutting or sectioning of the sample to ensure that it will fit inside the analysis chamber and be suitable for the analysis objectives.

### B.2.3 Specific sample handling requirements

In cases where the analysis will be performed on the “as received” specimen; surface contamination or atmospheric adsorbates are not usually removed because they are the item of interest. Special care shall be taken in the handling of these specimens to ensure that nothing, apart from air or clean inert gas, contacts the surface to be investigated. In such cases it is often appropriate to 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 cases where these precautions are not feasible due to the size of the specimen, some alternative specimen handling and transporting methods are presented in [Annex C](#).

To minimize the potential for contamination of the analysis area during handling, select one of the methods in the list a) to e) below. The list is in approximate order from most severe to least severe requirements. Suggested handling methods for different categories of specimens and analysis objectives are indicated in column 5 in [Table A.1](#). The selection of the specific method requires careful consideration of the type of sample and the information being sought, and again consultation with the analyst prior to handling can be helpful.

- a) Use a clean specialty tool (e.g., wafer tweezers or non-magnetic grippers) to hold the specimen. The tool needs to be washed in high purity isopropyl alcohol and dried between uses. The hand needs to be covered by a polyethylene glove.
- b) Use clean, dry stainless-steel tweezers or grippers to hold the specimen. The tool needs to be washed in high purity isopropyl alcohol regularly. Covering the hand with a polyethylene or a silicone-free latex glove is also needed to further limit contamination.
- c) A powder-free polyethylene or silicone-free latex gloved hand can be used to manipulate the sample providing that the hand only comes in contact with the edges of the sample. Even a gloved hand will contaminate a surface to be analysed and any such contact is to be avoided.
- d) In unique cases (e.g., analysts with skin sensitivity to gloves or lack of appropriate non-contaminating gloves) an un-gloved hand might be used to manipulate a cleaned tool; however, if the hand touches the end of the tool that contacts the specimen unwanted contamination will occur. Tools need to be washed in high purity isopropyl alcohol and dried between uses. In this circumstance some type of check for contamination spread is appropriate.
- e) Acid-free, lint-free paper can be used to grip specimens by their edges. However, contamination can occur if the paper comes into contact with the analytical surface.

### B.3 Considerations when multiple analysis techniques are required

Some samples require analysis with multiple techniques. Careful consideration must be taken in determining the order in which the different techniques are to be performed. Where possible, employ surface analytical techniques before employing bulk techniques.<sup>[5]</sup> There are distinct advantages to performing the surface analytical techniques first, both to minimize contamination from additional sample handling and to eliminate or reduce effects of the other analytical techniques on the surface analysis data. Many techniques do not require such careful attention to avoid surface contamination, so it is possible that the analysts are not aware of the stringent requirements needed to obtain reliable surface analysis results.

Another consideration is that the sample can be altered or damaged by other techniques (including other surface analytical techniques).<sup>[5]</sup> Specimens analysed using electron spectroscopy, especially insulating samples, are often coated with graphite or conducting materials to minimize charging. These coatings will obscure the surface for surface analysis. Specimens analysed using techniques requiring extensive sample modification (e.g., TEM) are likewise most likely unsuitable for subsequent surface analysis. Even when the sample is not mechanically modified, exposure to the probe beams (electron, photon, etc.) alone can modify the surface. For example, exposure to the electron beam in SEM or even AES can induce damage or react with residuals in the vacuum chamber to deposit material (often carbon) onto the sample surface beneath the

beam.<sup>[8]</sup> It can be possible to wrap the sample with clean aluminum foil, leaving only a small area exposed to the SEM or other probe beam, which area can be avoided during subsequent analysis. Sometimes, but not always, changes to the sample (often discoloration) can be observed visually, but morphological changes are also possible (e.g., from AFM). Obviously, any technique that etches (sputters) the sample will irreversibly alter the surface, but even static SIMS can affect some samples over a significant depth.<sup>[9]</sup> It is to be noted that even different surface analytical techniques (XPS, AES, etc.) can affect the sample, especially for small area analysis, during which portions of the sample can be exposed to much higher beam flux densities. Among surface analytical techniques, XPS is best done before AES, which in turn needs to be done before static SIMS and ISS.<sup>[5]</sup>

If possible, it can be advisable to prepare different, but equivalent samples for surface analytical techniques. If this is not possible, and the damage is localized and can be identified, different (undamaged) regions of the sample can be used for the different techniques, provided that the sample is homogeneous.

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## Annex C (normative)

# Sample Storage and Transport

### C.1 Storage requirements and needs

It is generally best to prepare the sample just before analysis to avoid any contamination or other alteration of the surface over time. However, samples frequently require storage before the initial analysis, or need to be transported for analysis. In all cases, the same care must be taken to avoid contamination during storage and transport of the samples, both before analysis and perhaps even after analysis (e.g., for archival samples, or samples that will undergo further or repeated analyses at a later time.) Although care is always required, the longer the time between sample collection or preparation and analysis, the greater the care needed. All the details discussed in [Annexes A](#) and [B](#) should be considered, along with the additional points in this annex.

### C.2 Storage time, temperature and humidity

Many processes that can alter sample surfaces will continue over time, with increased effects to the sample. Exposure to the atmosphere and nearby materials or incidental handling can expose the samples to a greater contaminant load with time. Unstable samples can continue to degrade or react over time as well. In these cases, the ideal situation is to prepare the sample as close to the analysis time as possible, but sometimes this is simply not practical. Consideration of the best way to store and transfer the samples to minimize contamination is given in the next two clauses.

Storage time needs to be minimized, when possible, to decrease possibilities of sample changes or contamination, and documentation of the time in storage is often important information. Temperatures, both high and low, can be problematic, with some types of chemical reactivity and outgassing increasing with temperature. There is the possibility of condensation onto the sample with too cold of temperatures. Humidity can be an issue, particularly for hygroscopic samples, but also due to condensation of material onto the sample that can occur with changes in humidity. Highly sensitive samples can benefit from storage in a controlled-atmosphere glove box or similar container.<sup>[5]</sup> At the minimum, these environmental parameters are important parts of the sample provenance record.

### C.3 Containers

#### C.3.1 Avoid contamination by the container

Some type of container is often used to protect (and store) the sample. Care must be taken to ensure that the material of the container itself does not react with the sample, and to prevent the transfer of contaminants to the sample from the container. If necessary, clean the container using the guidance given in [Annexes A](#) and [B](#). In addition to particles (dust), residual material on the container walls and volatile species within the container material (e.g., plasticizers) can diffuse onto the sample. Lids can be of a different material from the container and can have a liner that must be evaluated for contamination potential.<sup>[6]</sup> Seah and Spencer compiled information about contamination rates of clean samples in glass, polypropylene, and polyethylene containers and laboratory air.<sup>[10]</sup>

#### C.3.2 Container types and minimizing contact/contamination during storage

A wide variety of containers and strategies are available for sample storage. The list below gives some common options and is loosely ordered from most protective to least protective. Suggested sample