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# Surface chemical analysis — Handling of specimens prior to analysis

*Analyse chimique des surfaces — Manipulation des échantillons avant analyse*

Reference number
ISO 18117:2009(E)

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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.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18117 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 2, *General procedures*.

This corrected version of ISO 18117:2009 incorporates the following corrections:

- an additional paragraph has been inserted at the beginning of Clause 5;
- subclause 6.6 has been divided into two subclauses, 6.6 and 6.7, and the subsequent subclause numbering (and the cross-references elsewhere in the text to the subclauses concerned) corrected accordingly;
- it has been made clear in Table 1 and 9.2 that the PTFE tape used must be fresh;
- a small number of minor editorial changes have been made.

# Introduction

This International Standard instructs those who wish to submit specimens for surface chemical analysis in the handling and delivery of the specimens to the analyst. Although primarily written for auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS) and secondary-ion mass spectrometry (SIMS), these methods can also be applied to other surface-sensitive analytical measurements. AES, XPS and SIMS are sensitive to surface layers that are typically a few nanometres (nm) thick. Such thin layers can be subject to severe perturbations from improper specimen handling [1, 2]. Proper handling and preparation of specimens is particularly critical for analysis. Improper handling of specimens can result in alteration of the surface composition and unreliable data.

This International Standard is intended for the specimen owner or the purchaser of surface analytical services and for the surface analyst. The optimum handling procedures are dependent on the particular specimen and the needed information, and this document provides illustrative examples for each specimen type that a specimen owner and surface analyst will typically encounter. It is recommended that the specimen supplier consult the surface analyst as soon as possible with regard to specimen history, the specific problem to be solved or information needed, and any particular specimen preparation, handling or shipping procedures required.

This International Standard is based on ASTM E 1829-02, *Standard Guide for Handling Specimens Prior to Surface Analysis*, copyright ASTM, used with permission of ASTM.

This International Standard can be used independently of ISO 18116 [4], which gives guidance to the analyst for specimen preparation and mounting for surface analysis.

# Surface chemical analysis — Handling of specimens prior to analysis

## 1 Scope

This International Standard gives guidance on the handling of and the containers for specimens submitted for surface chemical analysis. It is intended for the user of surface analysis services as an aid in understanding the special sample handling requirements of surface chemical analysis techniques, particularly the following: Auger electron spectroscopy (AES), secondary-ion mass spectrometry (SIMS) and X-ray photoelectron spectroscopy (XPS or ESCA). The protocols presented may also be applicable to other analytical techniques, such as TXRF, that are sensitive to surface composition. In particular instances, with particular specimens, further precautions may be necessary.

## 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 18115, *Surface chemical analysis — Vocabulary*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115 apply.

## 4 Symbols and abbreviated terms

| | |
|---|---|
| AES | Auger electron spectroscopy |
| AFM | atomic force microscopy |
| ESCA | electron spectroscopy for chemical analysis |
| PTFE | polytetrafluoroethylene |
| SEM | scanning electron microscopy |
| SIMS | secondary-ion mass spectrometry |
| TXRF | total reflection X-ray fluorescence spectroscopy |
| XPS | X-ray photoelectron spectroscopy |

## 5 Explanation of the structure of this International Standard

Clause 6 provides the specimen owner with general guidance for minimizing specimen surface contamination during specimen handling and transport. In addition, specimen types are grouped into categories according to the depth (analysis location), relative to the specimen surface, from which the analytical information is being sought. Thus increasing cleanliness in specimen handling and transport is required as the analysis location approaches the top monolayer of the specimen surface. Table 1 then specifies, for each specimen category, the handling procedures and specimen containers necessary to minimize contamination and give optimum quality of analysis. Table 1 refers to more detailed descriptions found in subsequent clauses of this International Standard.

Clause 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. Clause 8 provides specific recommendations on specimen handling procedures necessary to minimize contamination of the specimen surface. Moreover, Clause 8 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. Clause 9 describes different specimen containers that may be used in different conditions. Clause 9 also discusses specimen storage with respect to time, humidity, and temperature. Clause 10 and Clause 11 emphasize 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.

## 6 General requirements and classes of specimen

**6.1** The degree of cleanliness required by surface-sensitive analytical techniques is much greater than for many other forms of analysis.

**6.2** Specimens shall never be in contact with the bare hand. Contact of the surface area to be analysed with handling tools or other equipment shall be eliminated or minimized whenever possible.

**6.3** Specimens shall be transported to the analyst in a container that does not come into direct contact with the surface of interest.

**6.4** In many cases, the analysis will be performed on the “as received” specimen; surface contamination or atmospheric adsorbates are not then usually removed because they are the item of interest. Care shall be taken in the handling of these specimens 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 cases where these precautions are not feasible due to the size of the specimen, some alternative specimen handling and transporting methods are presented in 9.2 i), 9.2 j), and 9.2 k).

**6.5** In some cases, it may be necessary to take a representative sample from the specimen. Selection of a smaller sample from a larger specimen should be done after considering 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 should be taken to avoid contaminating the surface of interest during the cutting procedure (see ISO 18116).

**6.6** Special caution shall be exercised with specimens containing potential toxins or other hazardous materials. Whenever possible, chemical hazard data sheets should be supplied with the specimen.

**6.7** The severity of the requirement for careful handling varies dramatically with the condition of the surface, the depth from the surface of the information being sought, and the detection level required for the material being analysed. The following list arranges specimens by their decreasing sensitivity to handling. This list is partly recreated in columns 1, 2 and 3 of Table 1.

a) Reactive specimens where the reactive surface is to be analysed.

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

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

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

e) Specimens with atmospheric adsorbates that may 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 in the analytical chamber.

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) Bulk materials where the information sought is on bulk properties.

## 6.8 Information sought

**6.8.1** Surface chemical analysis can be performed on a wide range of specimens and can be used to obtain very different types of information about surfaces or interfaces. The degree of care that shall 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, requiring different types of specimen:

- type A: information requiring integrity of the outermost surface;
- type B: information as a function of depth (depth profile) or at a buried interface; and
- type C: information that will require subsequent specimen preparation by the analyst.

**6.8.2** Type A specimens include those to be investigated for surface contamination, surface organic coatings, biomaterials-except live organism (cells, bacteria, etc.), surface stains, semiconductors, adhesion failures, etc. This category requires the most care in preparation and packaging. Nothing shall 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 A specimens fall in the first two rows in Table 1.

**6.8.3** Type B specimens include those that require the investigation of thick and thin films, single layers, multilayers, metal contact layers on semiconductors, coatings, dopant profiles, and the chemical and physical properties at an interface. For this category, the packaging requirements are not stringent, although care should still be taken not to contaminate the specimen. In this class, the information sought comes from a layer below the outermost surface and identification of superficial surface contamination is not the goal of the analysis. Surface diffusion, however, may play a role in the interpretation of the results. Care should be taken to avoid carbonaceous and particulate contaminations of the surface as these can degrade the quality of depth profiles. Type B specimens are in the third row of Table 1.

**6.8.4** Type C specimens include those that require preparation by the analyst, which includes specimens for *in situ* fracture, metallurgical lapping or polishing, and specimens that are part of a larger assembly. Generally, these specimens must 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 that undergo further sample preparation by the analyst. For specimens in a larger assembly or subassembly, it may be preferable to leave the specimen in place and let the analyst remove it prior to analysis. Nonetheless, care should still be taken not to contaminate the specimen. Type C specimens are in the fourth (last) row of Table 1.

**Table 1 — Minimum handling methods and specimen containers for different popular categories of information sought**

| Information sought | | | Handling method | | Specimen container | |
|---|---|---|---|---|---|---|
| **Sub-clause 6.7, item** | **Specimen type (See Sub-clause 6.8)** | **Specimen category/ depth of information** | **Sub-clause 8.5, item** | **Method** | **Subclause 9.2, item** | **Container** |
| a) | A | Specimens at analysis are to be as close to original condition as possible. | a) | Clean non-magnetic, uncoated stainless steel or specialty tools only, these tools being handled using polyethylene gloves. | a), b) | Argon or nitrogen glove box or vacuum transfer vessel.<br>Two flat specimens, face-to-face, sealed with fresh PTFE tape. |
| b), c), d) | A | Specimens requiring hydrocarbon, molecular, contaminant, or surface layer analysis (e.g. static SIMS and XPS analyses). | b) | Clean non-magnetic, uncoated stainless steel tweezers or grippers only, these tools being handled using polyethylene gloves. | c), d), e), f), g), h) | Clean glass with glass, fresh PTFE tape, or clean Al foil stopper.<br>High quality polypropylene wafer holder (note: avoid polyethylene spider inserts). |
| e), f), g) | B | Specimens where the surface of interest is obscured by a surface contamination layer from handling or environmental exposure | c), d) | Powder-free, polyethylene disposable gloves holding specimen by edge.<br>Powder-free, silicone-free, latex gloves holding specimen by edge. | i), j) | Clean Al foil.<br>Polyethylene box or bag. |
| h), i), j), k), l) | C | Specimens with buried interfaces, fracture specimens, bulk analysis. | e), f) | Clean tools, hand held (no gloves).<br>Acid-free, lint-free paper to hold specimen by edge. | k) | Acid-free, lint-free paper. |

# 7 Specimen influences

## 7.1 Specimen information needed by analyst

The analyst should be told of the specimen history, special storage or transport requirements, the specimen's prior exposure to possible contaminants, and the information being sought.

## 7.2 History

The history of a specimen may affect the choice of handling of the surface before analysis. For example, a specimen that has been exposed to a contaminating environment may reduce the need for exceptional care if the surface becomes less reactive. Alternatively, the need for care may increase if the surface becomes toxic.

## 7.3 Specimens previously examined by other analytical techniques

It is best if surface chemical analysis measurements are made before the specimen is analysed by other analytical techniques because such specimens may become damaged or may be exposed to surface contamination. For example, insulating specimens analysed by electron microscopy may have been coated, for example, by Au, Pt, C or other conductive material, to reduce charging. Such coatings will render the specimen unsuitable for subsequent surface analysis.

Furthermore, exposure to an electron beam in a scanning electron microscope (SEM) can also induce damage or cause the adsorption of surface species from the residual vacuum. Contamination build-up in the SEM is almost always carbonaceous in nature, and this may complicate the interpretation of subsequent surface analysis measurements where carbon is detected. If it is not possible to perform the surface chemical analysis work first, then the SEM analysis should be done on a different, but nominally identical, specimen or area of the specimen. If SEM analysis is done on a different, but identical area of the specimen, the remainder of the specimen can be wrapped with clean aluminium foil to prevent exposure of the remainder of the specimen to the SEM electron beam.

The prior use of atomic force microscopy can alter a surface and, even a low dose of static SIMS, in which the SIMS shows no effect, can affect some samples over a significant depth. [5]

### 7.4 Identification of specimens

Clearly identify all specimens with a unique name or identifier. Clearly indicate the area of analysis by marking up a drawing or a photograph. If necessary, a scribe or a pencil can be used on an area adjacent to the areas of interest. Care should be taken that any particles generated during scribing or marking do not adhere to the specimen, particularly the area of interest. If there is any doubt as to which side of the specimen is to be analysed, clearly identify the back of the specimen.

### 7.5 Precautions

Do not touch the surface of interest, either by hand or with a tool. Do not “protect” the surface of interest by covering it with tape, contaminated foil or porous wrapping material. Do not use a diamond scribe to mark semiconductors. Fragile specimens should not be mounted onto double-sided tape since they may fracture during removal from the tape.

## 8 Sources of specimen contamination in handling

**8.1** An unprotected hand must never handle specimens, even when the skin will not touch the surface of interest. Fingerprints and hand creams contain mobile species which may migrate and contaminate the surface of interest.

**8.2** Specimens should only be handled with clean tools to ensure that the specimen surface is not altered. Such tools should be made of materials that will not transfer to the specimen. Moreover, clean tools should be used to handle the edges of the specimen, but analysis must be in a region where the tools have not made contact. All tools should be regularly cleaned in high purity solvents and dried prior to use.

In the specific case of silicon, low Ni stainless steel tools are suggested, since Ni containing tools have been reported to contaminate silicon.

Non-magnetic tools should always be used to handle the specimen.

**8.3** Gloves and wiping materials are sometimes used to handle specimens; it is likely that their use may result in some contamination. Care should be taken to avoid gloves with talc, silicone release agents, and other materials. “Powder free” gloves have no talc and may be better suited. Unnecessary contact shall be avoided.

**8.4** Blowing on the specimen with the mouth is not recommended for the removal of unwanted particulate debris from the surface. Blowing on the specimen using a compressed gas source may be necessary to remove particulates. Since some gas sources may introduce contaminates such as oil or other particulates from the delivery line, it is preferable for this step to be conducted by the analyst rather than the specimen owner during specimen handling and packaging. Most surface analysis facilities utilize clean, dry nitrogen or other inert gas passing through clean stainless steel tubing to minimize contamination during particulate removal. Cleanliness of the analytical facility gas shall have been verified by surface analysis of clean specimens, such as oxidized silicon or quartz, that have been blown off by the compressed gas.

**8.5** To minimize the potential for contamination of the analysis area during handling, select one of the methods in the list a) to f) below. The list is in order from most severe to least severe and columns 4 and 5 of Table 1 show the relationship to the types of specimen. The selection of the specific method should be made in careful consideration of the type of specimen (see 6.8) and the information being sought.

a) Use a clean specialty tool (e.g. wafer tweezers or non-magnetic grippers) to hold the specimen. The tool should be washed in high purity isopropyl alcohol and dried between uses. The hand should be covered by a polyethylene glove.

b) Use clean, dry tweezers or grippers to hold the specimen. The tool should be washed in high purity isopropyl alcohol regularly. A polyethylene glove should cover the hand.

c) The tool should be washed in high purity isopropyl alcohol and dried between uses. An ungloved hand may be used to manipulate the cleaned tool: the hand must never touch the end of the tool that grips the specimen.

d) The tool should be washed in high purity isopropyl alcohol and dried regularly. An ungloved hand may be used to manipulate the cleaned tool: the hand must never touch the end of the tool that grips the specimen.

e) The hand in a polyethylene glove may be used to manipulate a clean specialty tool, tweezers or grippers to hold the specimen.

f) Acid-free, lint-free paper (e.g. chemical wipes) may be used to grip specimens by their edges. The paper must not come into contact with the analytical surface.

## 9 Specimen storage and transfer

### 9.1 Storage

#### 9.1.1 Time

The longer a specimen is in storage, the more care must be taken to ensure that the surface to be analysed has not been contaminated. Even in clean laboratory environments, surfaces can quickly become contaminated to the depth analysed by AES, XPS, SIMS, and other surface sensitive analytical techniques.

#### 9.1.2 Containers

**9.1.2.1** Storage containers should not transfer contaminants to the specimen via particles, liquids, gases, or surface diffusion. Preferably, the surface to be analysed should not come into contact with the container or any other object. Keep in mind that volatile species (e.g. plasticizers) may be emitted from containers and could contaminate the surface. Glass jars with an inside diameter slightly larger than the width of a specimen can hold a specimen without contact with the surface. When contact is unavoidable, wrapping in clean, pre-analysed aluminium foil may be satisfactory. For semiconductor samples, standard wafer carriers are generally adequate.

**9.1.2.2** Containers such as glove boxes, vacuum chambers, and desiccators may be excellent choices for temporary storage of specimens. A vacuum dessicator may be preferable to a standard unit and should have been maintained free of grease and mechanical pump oil. Cross-contamination between specimens may also occur if multiple specimens are stored together.

#### 9.1.3 Temperature and humidity

Possible temperature and humidity effects should be considered when storing or shipping specimens. Most detrimental effects result from elevated temperatures. Additionally, low specimen temperatures can lead to moisture condensation on the surface.