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Surface chemical analysis — Handling of specimens prior to analysis

Analyse chimique des surfaces — 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.