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Surface chemical analysis — X-ray photoelectron spectroscopy — Guidelines for analysis

Analyse chimique des surfaces — Spectroscopie de photoélectrons par rayons X — Lignes directrices pour l'analyse

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Foreword

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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 10810 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *X-ray photoelectron spectroscopy*.

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Introduction

X-ray photoelectron spectroscopy (XPS) is used extensively for the surface analysis of materials. Elements in the sample (with the exception of hydrogen and helium) are identified from comparisons of the measured binding energies of their core levels with tabulations of those energies for the different elements. Their chemical states may be determined from shifts in peak positions and other parameters compared with the data for that element in its pure elemental state. Information on the quantities of such elements can be derived from the measured intensities of photoelectron peaks. Calculation of the quantities of the constituent chemical species present in the surface layer studied may then be made using formulae and relative-sensitivity factors provided by the spectrometer manufacturer or locally measured relative-sensitivity factors and appropriate software.

This guidance document is intended to aid the operator of X-ray photoelectron spectrometers to obtain efficient, meaningful analyses from typical samples.

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Surface chemical analysis — X-ray photoelectron spectroscopy — Guidelines for analysis

1 Scope

This International Standard is intended to aid the operators of X-ray photoelectron spectrometers in their analysis of typical samples. It takes the operator through the analysis from the handling of the sample and the calibration and setting-up of the spectrometer to the acquisition of wide and narrow scans and also gives advice on quantification and on preparation of the final report.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references only the cited edition applies. For undated references, the latest edition of the referenced document (together with any amendments) applies.

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

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3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 18115-1 apply.

4 Symbols and abbreviations

AES	Auger electron spectroscopy
ARXPS	angle-resolved X-ray photoelectron spectroscopy
CCQM	consultative committee for amount of substance
CRM	certified reference material
EAL	effective attenuation length
FAT	fixed analyser transmission
FRR	fixed retard ratio
FWHM	full width at half maximum
IERF	intensity/energy response function
NIST	National Institute of Standards and Technology
NPL	National Physical Laboratory
RM	reference material
RSD	residual standard deviation
S/N	signal-to-noise ratio

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XPS X-ray photoelectron spectroscopy

Δ_1 difference between the measured and reference energies for Au $4f_{7/2}$

Δ_4 difference between the measured and reference energies for Cu $2p_{3/2}$

5 Overview of sample analysis

Figure 1 is a flow chart illustrating the analysis of a typical sample by XPS. A preliminary consultation with the supplier of the sample should be used to ensure that the sample is supplied in the form most appropriate for analysis. ISO 18117[2] explains the issues involved with prior handling by the supplier and also gives information on the most suitable container for transportation. In this consideration, the analyst should also identify any particular problems likely to arise. Table 1 provides a list of example problems. Prior to any work, discussions should be held between the analyst and the customer to gain as much information as possible by reviewing what is already known regarding the sample and its history. In addition to the information listed in ISO 18117[2], Table 2 indicates information that will assist in deciding how to conduct the XPS analysis. Following these preliminary discussions, the sample(s) may need to be prepared to allow mounting in the spectrometer and to reduce, where possible, the subsequent analysis time. ISO 18116[1] provides details of how to do this. The analyst will be responsible for the instrument characterization, which will include the calibration state and the overall performance of the XPS instrument. A guide to calibration of the energy scale is given in ISO 15472[14]. Checks for the intensity scale are given in ISO 24237[9] and ISO 21270[18].

Once the specimen has been mounted in the spectrometer and the system pumped down, data acquisition can commence. A wide scan should be obtained first and this then analysed to determine the elements present. ISO 16243[31] provides information on recording and reporting data in XPS. The wide-scan spectrum can provide qualitative and semi-quantitative information regarding composition and the depth distribution of species. This may yield sufficient information to satisfy the customer and the analysis may be terminated. However, in most cases, more data are required and narrow-scan spectra will then be recorded from regions identified in the wide-scan spectrum. Analysis of these narrow-scan spectra will provide chemical-state information, more accurate quantitative information and near-surface depth information. At a later time in the investigation the wide scan should be repeated to determine if there has been degradation (e.g. due to X-ray irradiation or to surface reactions with ambient gases in the vacuum system). Following evaluation of the XPS data, the analyst should produce a report.

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Table 1 — Problems likely to arise and related ISO standards

Problem	Example	ISO Standard
Outgassing	Water vapour	ISO 18116
Degradation	Polymers and organics	
Charging	Insulators	ISO 19318[28]
Reduction	Oxides	
Contaminant mobility	Chlorine	
Sample containment	Powders	ISO 18116
Surface topography	Fibres	

Table 2 — Sample information and history

Sample information and history
Thermal
Contamination
Possible composition
Segregation
Surface layer
Homogeneous
Islands

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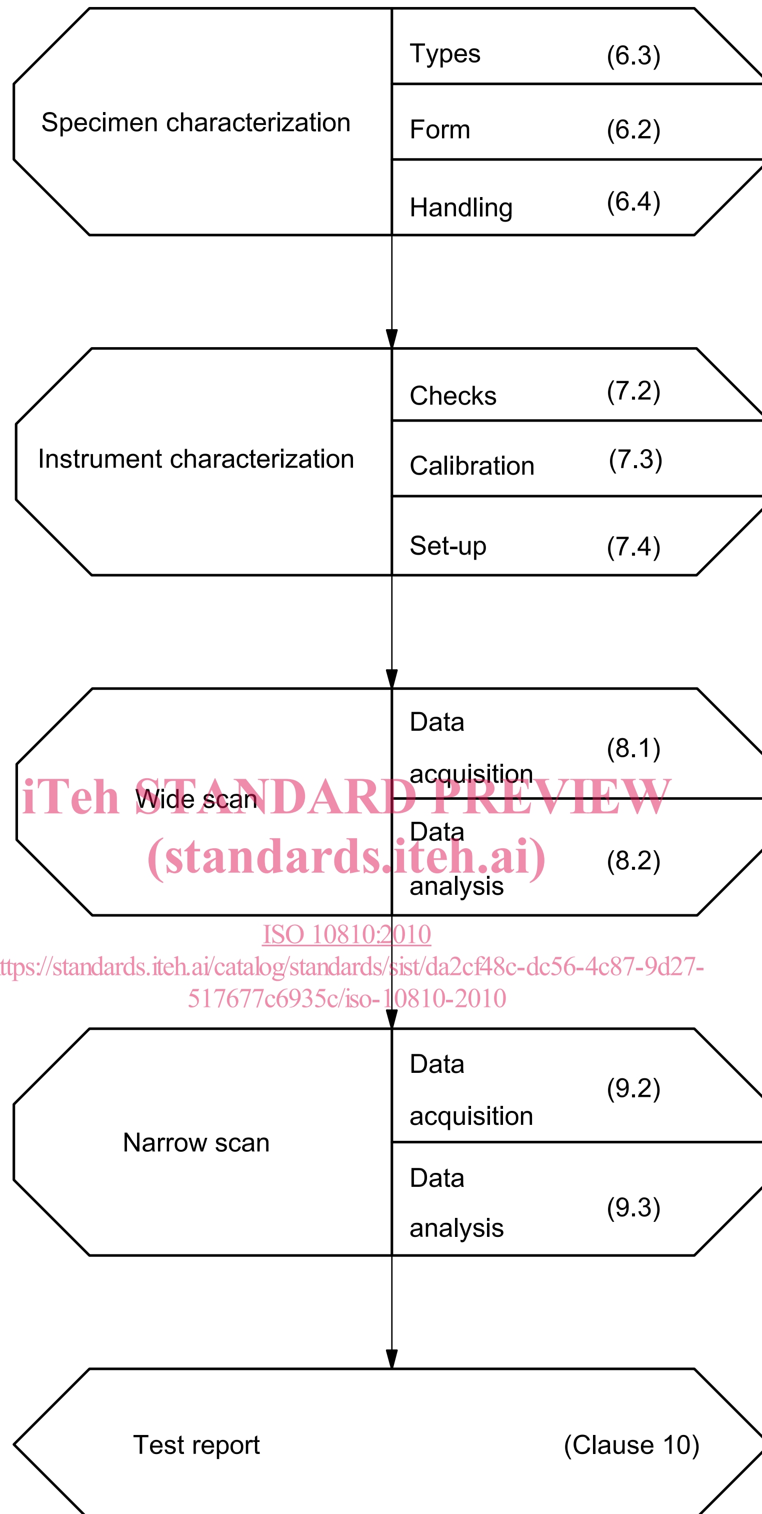

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Figure 1 — Flow chart of an XPS analysis
(The numbers in brackets indicate the respective subclauses in this International Standard.)

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6 Specimen characterization

6.1 General

The complexity of the interacting factors in XPS analyses arises from the many different forms of specimen materials and the variety of material types that may be encountered as well as from the different XPS experiments that might be required. Table 3 illustrates possible specimen forms, material types, and XPS experiments or issues for further review. The analyst should also be aware that samples can consist of multiple components and phases, and that identification of the components and phases present (and their spatial arrangements) can be an important part of an XPS analysis. A further complication is that non-conducting samples may charge.

Table 3 — Some specimen forms, material types, *in situ* specimen treatments and possible XPS experiments

Specimen forms	Material types	*In situ* specimen treatments	XPS experiments
Adsorbed layers (6.2.3)	Alloy (6.3.2)	Cooling (6.5.2)	Angle-resolved XPS
Amorphous	Biological (6.3.9)	Degradation	Analysis area (small)
Fibres (6.2.8)	Catalyst (6.3.7)	Deposit thin films	Analysis area (large)
Films (6.2.3)	Ceramic (6.3.6)	Expose to high gas pressure (6.5.5)	Depth profile
Interface (6.2.4)	Composite	Fracture (6.5.3)	Imaging
Internal interface (6.2.9)	Glass (6.3.8)	Heating (6.5.2)	Line scan
Liquid	Insulator (6.3.8)	Insert into liquids (6.5.5)	
Multilayered (6.2.4)	Magnetic metal (6.3.5)	Ion bombardment (6.5.4)	
Nano-material	Metal (6.3.2)	Scraping (6.5.3)	
Non-porous (6.2.5)	Non-metal (pure) element		
Pattern system	Polymer (6.3.3)		
Polycrystal	Semiconductor (6.3.4)		
Porous (6.2.6)	Textile		
Powder (6.2.7)			
Residue (6.2.3)			
Segregated layer (6.2.3)			
Single crystal (6.2.2)			
Solid			
Textile (6.2.8)			
Contamination			

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6.2 Specimen forms

6.2.1 General

The form of the specimen to be analysed will strongly dictate the kinds of experimental approach that can and need to be employed.

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6.2.2 Single crystal

This type of sample should have a flat surface. Quantitative analyses will generally be difficult because of anisotropies in the angular distributions of the photoemitted electrons due to electron diffraction or to forward-focussing effects[3][4]. These anisotropies are nevertheless useful in determining the structural properties of the sample.

6.2.3 Adsorbed or segregated layers, films and residues

It should, in general, be possible to obtain a quantitative analysis and chemical-state information for adsorbed or segregated layers, films and residues[5][6]. If the substrate is a single crystal, however, quantitative analyses will generally be difficult, but the angular distributions of the photoemitted electrons can give useful structural information[3]. Angle-resolved XPS (ARXPS), as described in 9.3.3, will enable the layer thickness to be determined, provided the layer thickness does not exceed around three times the effective attenuation length (EAL) of the substrate peak. This will be of progressively lower accuracy for films above one EAL in thickness.

6.2.4 Interfaces and multilayered samples

Ion sputter depth profiling should permit the depth distribution and thickness of the layers to be determined, together with a semi-quantitative analysis of the layers, as described in 9.3.3.

6.2.5 Non-porous

A quantitative analysis together with chemical-state information can be obtained.

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6.2.6 Porous

Only a semi-quantitative analysis may be possible since the sample will have a rough surface.

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6.2.7 Powder

Mount the sample by embedding in a suitable matrix. In many cases, indium proves sufficiently soft to be able to accept the powder without particles falling off. Then treat the composite sample as a porous sample but be sure to remember to subtract the indium peaks. Double-sided, carbon-loaded, conductive adhesive tape is also very suitable as a mounting material.

6.2.8 Fibres and textiles

For fibre analysis, the alignment of the fibres relative to the X-ray source may be an important factor. The diameter of the fibre relative to the diameter of the analysis area will also affect the ability to quantify the data. If possible, mount several fibres in a bundle to increase the surface area. However, a quantitative analysis will generally not be possible with many manufacturers' software systems, although some chemical-state information can be obtained. Under certain conditions, it is possible to analyse one monofibre, using a coaxial ion gun to conduct a sputter depth profile or, if there is sufficient spatial resolution in relation to the fibre diameter, ARXPS may be conducted around the circumference.

6.2.9 Internal interface

An internal interface can be analysed using ARXPS, as described in 9.3.3, bearing in mind the depth limit of around three times the EAL discussed in 6.2.3. To analyse a weak or brittle internal interface that occurs at greater depths, it is generally necessary to first expose the interface in the ultra-high vacuum by use of fracture stages, etc. For other internal interfaces, one of the forms of depth profiling described in ISO/TR 15969[21] may prove effective.

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6.3 Material types

6.3.1 General

For different materials, there are various consequences for an XPS experiment that may need to be considered. For example, problems may arise when analysing magnetic, radioactive and outgassing samples.

6.3.2 Metals and alloys

With specimens in this category, there should be minimal surface charging, but there may be a surface oxide film together with a high level of carbon contamination. In general, there should be no need for surface treatment prior to analysis. However, in many cases *in situ* ion sputtering is carried out prior to analysis to remove any oxide/contaminant overlayer.

6.3.3 Polymers

It may be difficult to achieve the desired vacuum with this category of sample due to outgassing. During analysis, adventitious carbon and possibly sample charging and sample degradation may occur. The spectra should contain intense peaks from C, O and N, possibly also from F, Cl and S.

6.3.4 Semiconductors

There should be minimal surface charging with these specimens and there should be low levels of carbon contamination. However, expect to see a surface oxide.

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6.3.5 Magnetic materials

Take care when handling magnetic materials. First demagnetize them, if possible, and analyse with any magnetic immersion lens switched off. A magnetized sample will affect the performance of a magnetic lens in a way which will depend on the kinetic energy of the electrons being analysed. A magnetized sample may also lead to changes in a measured spectrum that depend on the electron energy. Expect the analysis to be similar to that for metals and alloys.

6.3.6 Ceramics

Sintered or porous ceramics may outgas and it may be difficult to evacuate the chamber to a pressure sufficiently low for XPS analysis. A threshold pressure may be set by the manufacturer to protect the X-ray source or other instrumental items. There may be significant surface charging and one should expect moderate levels of surface carbon contamination.

6.3.7 Catalysts

These samples may behave in a similar way to ceramics, and there may be health and safety considerations when handling.

6.3.8 Glass and insulators

These samples may be analysed but will charge, and the use of an electron flood gun with or without a low-energy positive-ion flood may be necessary to reduce the effect of charging.

6.3.9 Biological

These samples may outgas in the spectrometer and suffer degradation due to either the vacuum environment or the X-ray flux or both.

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