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

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National foreword

This British Standard is the UK implementation of ISO 10810:2019. It supersedes BS ISO 10810:2010, which is withdrawn.

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

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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 documents 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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *Electron spectroscopies*.

This second edition cancels and replaces the first edition (ISO 10810:2010), which has been technically revised. The main changes to the previous edition are as follows:

— Table 3: semiconductor wafer added as a specimen form;

— 6.2.7: paragraph replaced to reflect modern practice;

— 6.3.10: nanoparticles added as a material type;

— Clause 8 and the flow chart in Figure 6 have been thoroughly revised to improve clarity. The cells in the flow chart now contain references to the appropriate subclause within Clause 8;

— 8.2.1: it is now pointed out that the use of the C 1s peak provides only an approximate binding energy reference;

— 9.3.3.3: mention has been made of the use of ionised clusters of inert gas atoms for depth profiling.

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.

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Introduction

X-ray photoelectron spectroscopy (XPS) is used extensively for the surface (1 nm to 10 nm) 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 (outer 1 nm to 10 nm) 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 operators of X-ray photoelectron spectrometers in their analysis of the surfaces (outer 1 nm to 10 nm) of typical samples.

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

1 Scope

This document 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 documents are referred to in the text in such a way that some or all 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*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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

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RM reference material

S/N signal-to-noise ratio

XPS X-ray photoelectron spectroscopy

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

Δ_{Cu} 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 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 semiquantitative 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[1]
Degradation	Polymers and organics	ISO 18554
Charging	Insulators	ISO 19318[28]
Reduction	Oxides	
Contaminant mobility	Chlorine	
Sample containment	Powders	ISO 18116[1]
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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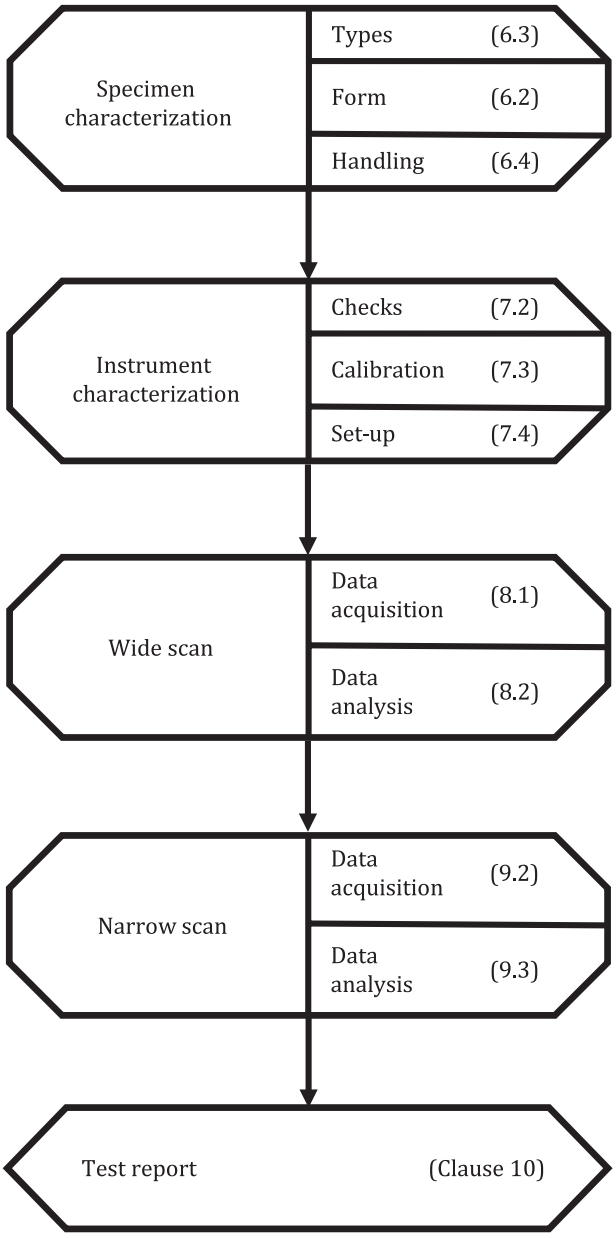

NOTE The numbers in brackets indicate the respective subclauses in this document.

Figure 1 — Flow chart of an XPS analysis

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.

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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	Small area analysis
Fibres (6.2.8)	Catalyst (6.3.7)	Deposit thin films	Large area analysis
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)			
Powder (6.2.7)			
Residue (6.2.3)			
Segregated layer (6.2.3)			
Single crystal (6.2.2)			
Solid			
Semiconductor wafer	Semiconductor	Ozone and UV/ozone cleaning	
Textile (6.2.8)			
Contamination			

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.

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-focusing 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.

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