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Surface chemical analysis — X-ray photoelectron spectroscopy — Description of selected instrumental performance parameters

National foreword

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**Surface chemical analysis — X-ray
photoelectron spectroscopy —
Description of selected instrumental
performance parameters**

*Analyse chimique des surfaces — Spectroscopie de photoélectrons
X — Description de certains paramètres relatifs à la performance
instrumentale*





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Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Abbreviated terms	1
5 Description of selected instrumental performance parameters	1
5.1 Method of analysis.....	1
5.2 Samples.....	1
5.3 System configuration.....	2
5.4 X-ray source.....	2
5.4.1 Anode type.....	2
5.4.2 Anode power.....	2
5.4.3 Expected anode lifetime.....	2
5.5 Spectrometer intensity performance and energy resolution.....	2
5.6 Spectrometer energy scale.....	2
5.7 Spectrometer intensity linearity.....	3
5.8 Spectrometer response function.....	3
5.9 Imaging and selected area resolution.....	3
5.9.1 General.....	3
5.9.2 Method 1.....	3
5.9.3 Method 2.....	3
5.9.4 Method 3.....	4
5.10 Charge neutralization.....	4
5.11 Angle-resolved XPS.....	4
5.12 Vacuum environment.....	4
Bibliography	5

Foreword

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

This second edition cancels and replaces the first edition (ISO 15470:2004), of which it constitutes a minor revision.

The changes compared to the previous edition are as follows:

- a typo has been corrected in [5.10](#);
- a Bibliography has been added;
- the text has been editorially revised to comply with the most recent drafting rules.

Introduction

X-ray photoelectron spectrometers are produced by many manufacturers throughout the world. While the basic principle of the XPS analytical method in each instrument is the same, the specific designs of the instruments and the way that performance specifications are provided differ widely. As a result, it is often difficult to compare the performance of instruments from one manufacturer with those from another. This document provides a basic list of items devised to enable all X-ray photoelectron spectrometers to be described in a common manner. This document is not intended to replace the manufacturer's specification, which may extend to 30 or more pages. It is intended that, where certain items are defined in that specification, there is an agreed and defined meaning to that item.

Surface chemical analysis — X-ray photoelectron spectroscopy — Description of selected instrumental performance parameters

1 Scope

This document describes the way in which specific aspects of the performance of an X-ray photoelectron spectrometer are described.

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 (all parts), *Surface chemical analysis — Vocabulary*

3 Terms and definitions

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

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

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Abbreviated terms

FWHM full width at half maximum

XPS X-ray photoelectron spectroscopy

5 Description of selected instrumental performance parameters

5.1 Method of analysis

A short description of the methods used to obtain information from the sample shall be given, and the availability (as an option) of other analytical techniques in the system under consideration shall be stated.

5.2 Samples

The size and shape of samples that may be analysed with the instrument performing to specification shall be given. If the size or shape is restricted for particular modes of analysis, e.g. angle-resolved measurements, measurements for insulators, etc., this shall be specified.

5.3 System configuration

The designed geometric configuration of significant analytical components of the system and their tolerances shall be described.

EXAMPLE Tolerances for angles are often given as $\pm 1^\circ$.

5.4 X-ray source

5.4.1 Anode type

The X-ray anode material shall be specified. The energies and relative intensities of unwanted X-rays shall be specified.

5.4.2 Anode power

The maximum X-ray anode power shall be specified by stating the potential between the electron source and the X-ray anode and the maximum filament emission current to the anode at that potential. These data shall be given for each of the instrumental operating modes specified, and the analytical areas shall be defined, such as:

- a) large area;
- b) defined areas for monochromators;
- c) high-energy resolution or other relevant modes of operation, as appropriate.

5.4.3 Expected anode lifetime

The expected lifetime of the X-ray anode under the instrumental operating conditions specified in [5.4.2](#) shall be stated. This is normally a guaranteed lifetime operation but may, alternatively, be a mean historical lifetime or a graphical description of performance versus operating hours at the instrumental operating modes specified in [5.4.2](#). The type of lifetime shall be specified.

5.5 Spectrometer intensity performance and energy resolution

The energy resolution of the system shall be specified by use of the full width at half maximum (FWHM) of the silver Ag 3d_{5/2} peak from a foil of silver cleaned by ion sputtering or scraping such that the intensities of contaminant peaks are less than 5 % of the Ag 3d_{5/2}. Both peak height intensity and the FWHM of the Ag 3d_{5/2} peak shall be given for all of the instrumental operating modes specified in [5.4.2](#). Both the peak height intensity and the FWHM are measured after removal of a straight-line background drawn tangentially at energy points 3 eV above and below the peak position. The energy resolution and intensity data may be provided for any other conditions, as required. It is desirable, but not essential, to specify the performance drift over a period of 10 min and 1 h after any required warm-up time.

5.6 Spectrometer energy scale

The repeatability standard deviation of the Cu 2p_{3/2} peak binding energy for samples re-positioned by the positioning procedure shall be given for all sources specified. This procedure should be described in the instrument manual. The binding energy error at the Ag 3d_{5/2} peak position shall be specified when the instrument has been calibrated using the Cu 2p_{3/2} and the Au 4f_{7/2} peaks. The accuracy of the binding energy scale calibration as a function of time shall also be specified at the energy for the Cu 2p_{3/2} or Au 4f_{7/2} peaks.

NOTE A method for calibrating the binding energy scale of X-ray photoelectron spectrometers is given in ISO 15472.

5.7 Spectrometer intensity linearity

The maximum useful count rate, and the maximum count rate for a defined limit of count rate linearity, such as $\pm 2\%$, shall be stated.

5.8 Spectrometer response function

Either the spectrometer response function or the energy dependence of that function shall be provided for the relevant instrumental operating modes specified in [5.4.2](#). The extent to which these functions remain constant with time shall be stated.

5.9 Imaging and selected area resolution

5.9.1 General

Imaging systems and selected area systems are treated equivalently. The measured value of the lateral resolution shall be obtained by one of the methods described in [5.9.2](#), [5.9.3](#) or [5.9.4](#).

5.9.2 Method 1

For imaging systems, a sample shall be analysed which has an isolated feature smaller than 30 % of the instrument's stated lateral resolution. The measured FWHM of a line trace for a photoelectron signal characteristic of that feature defines the lateral resolution. For selected area systems, a sample with a small feature of this kind may be scanned across the analysed area using a micrometer sample stage. The distance for the feature signal to rise from 50 % of the maximum to 100 % and then fall again to 50 % gives the lateral resolution.

NOTE 1 If the width of the isolated feature is greater than 30 % of the lateral resolution, the measured lateral resolution will be greater than the true lateral resolution.

NOTE 2 If an instrument has a lateral resolution function that can be represented by a Gaussian function, then the FWHM of such a function corresponds to the distance over which the measured signal changes from 12 % to 88 % of its maximum value. The use of a small sample allows easy confirmation of the system astigmatism.

NOTE 3 The lateral resolution may be energy-dependent. A sample of an appropriately sized bulk dot of copper on gold and gold on copper allows the resolution to be specified at both extremes of the energy scale.

5.9.3 Method 2

For imaging systems, a sample shall be analysed which comprises two materials with their surfaces in the same plane and joined along a common straight edge. A line trace for a photoelectron intensity, characteristic of one of the two materials, measured at 90° to the edge, is used to define lateral resolution. For selected area systems, the sample may be scanned across the analysed area using a micrometer sample stage by movement at 90° to the above edge. For both systems, the distance for the photoelectron intensity to change from 12 % to 88 % of the difference in the intensities in the plateau regions away from the edge defines the lateral resolution in the direction of the scan.

NOTE 1 If an instrument has a lateral resolution function that can be represented by a Gaussian function, then the FWHM of such a function corresponds to the distance over which the measured signal changes from 12 % to 88 % of its maximum value.

NOTE 2 Close to the limit of resolution, astigmatism may be observed, and so the lateral resolution may need determination in more than one azimuth.

NOTE 3 Where the analysis area is defined by an electron-optical method using an image aperture, the resolution is defined by the aperture, and the intensity:distance distribution is equivalent to 64 % of the aperture size.

NOTE 4 The lateral resolution may be energy-dependent. A sample comprised of copper and gold areas allows the resolution to be specified at both extremes of the energy scale.

5.9.4 Method 3

A sample shall be analysed which is composed of a knife edge of one material, in the sample plane, over a hole with a depth more than five times its diameter. A line trace for a photoelectron intensity, characteristic of the knife edge material, measured at 90° to the edge, is used to define lateral resolution. The distance for the photoelectron intensity to change from 12 % to 88 % of the difference in the intensities in the plateau regions away from the edge defines the lateral resolution in the direction of the scan.

NOTE 1 If an instrument has a lateral resolution function that can be represented by a Gaussian function, then the FWHM of such a function corresponds to the distance over which the measured signal changes from 12 % to 88 % of its maximum value.

NOTE 2 Close to the limit of resolution, astigmatism may be observed, and so the lateral resolution may need determination in more than one azimuth.

5.10 Charge neutralization

A description shall be given of available methods for charge neutralization on insulating samples during XPS measurements. As an indication of the effectiveness of charge neutralization, data shall be given for the peak energies and peak widths (FWHM) of the three C 1s peaks in poly(ethylene terephthalate) under specified conditions of operation of the X-ray source and the electron spectrometer as given in [5.4.2](#).

5.11 Angle-resolved XPS

A sample manipulator will usually be arranged so that the sample normal can be aligned along the spectrometer axis and then be set at some defined angle to this. The range and precision with which these angles may be defined shall be specified together with the angular acceptance angle of the spectrometer in the azimuth of the rotation of the sample normal.

5.12 Vacuum environment

The base pressure attainable 24 h after a 12 h bake at a specified temperature or the time to reach the specified base pressure shall be given. The sample introduction time for analysis at a specified pressure shall also be given.

Bibliography

- [1] ISO 15472, *Surface chemical analysis — X-ray photoelectron spectrometers — Calibration of energy scales*

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