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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

Reference number
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Contents

Page

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 15470 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *X-ray photoelectron spectroscopy*.

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 International Standard provides a basic list of items devised to enable all X-ray photoelectron spectrometers to be described in a common manner. This International Standard 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 International Standard describes the way in which specific aspects of the performance of an X-ray photoelectron spectrometer shall be described.

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 abbreviations

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 ± 1°.

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

or

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, *Surface chemical analysis — X-ray photoelectron spectrometers — Calibration of energy scales*.

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 ± 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 spatial 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 spatial resolution. The measured FWHM of a line trace for a photoelectron signal characteristic of that feature defines the spatial 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 spatial resolution.

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

NOTE 2 If an instrument has a spatial 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 spatial 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 is comprised of 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 spatial 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 spatial resolution in the direction of the scan.

NOTE 1 If an instrument has a spatial 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 spatial 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 spatial 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.