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Surface chemical analysis — Recording and reporting data in X-ray photoelectron spectroscopy (XPS)

Analyse chimique des surfaces — Enregistrement et notification des données en spectroscopie de photoélectrons par rayons X (XPS)

Reference number
ISO 16243:2011(E)

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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.

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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 16243 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 2, *General procedures*.

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Introduction

X-ray photoelectron spectroscopy (XPS) is used extensively for the surface analysis of materials. Elements in the specimen (with the exception of hydrogen and helium) are identified from the measurement of core-level binding energies in the photoelectron spectra, comparing them against elemental tabulations of those energies. Information on the chemical state of such elements can be derived from the chemical shifts and/or peak shape of the measured photoelectrons with respect to reference states.

This International Standard defines the level of information on the specimen and the experimental parameters that should be included in the analytical record. The results of the analysis should be recorded in a standard format that should include sufficient detail to allow the experiment to be repeated. This material should be available for reporting, as required.

Experimental conditions and data acquisition parameters should be included so that the quality of the data can be assessed.

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Surface chemical analysis — Recording and reporting data in X-ray photoelectron spectroscopy (XPS)

1 Scope

This International Standard specifies the minimum level of information to be reported by the analyst following the analysis of a test specimen using X-ray photoelectron spectroscopy (XPS). It includes information that is to be recorded on or in the analytical record.

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 18115-1, *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 and the following apply.

3.1
ex situ
outside the analytical system

3.2
in situ
inside the analytical system

4 Levels of recording and reporting

4.1 General

This International Standard defines the minimum level of information that shall be recorded and reported by an analyst following the analysis of a test specimen using XPS. The levels of recording and reporting are separated into six main areas:

a) the analyst's record book or electronic log (e.g. computer data storage system);

b) spectra;

c) quantitative analysis of the specimen;

d) compositional depth profiles;

e) maps;

f) chemical-shift data.

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4.2 Analyst's record

4.2.1 Specimen identification and preparation

For each individual specimen, the record book or electronic log shall contain the following information (sufficient information shall be recorded to allow the measurements to be repeated at a later date):

a) the name of the originating laboratory and the person supplying the specimen;

b) a unique specimen number;

c) a description of the specimen before and after analysis (including details of its physical appearance, its roughness, its colour and any other distinguishing features);

d) the date of the measurement(s);

e) the name of the analyst, and the analyst's department and affiliation;

f) all details concerning *ex situ* specimen preparation before analysis (including the method of mounting, the orientation on the specimen holder with respect to any specific surface features, whether the specimen was cut and, if so, how, details of any solvent cleaning, etc.) (see NOTE 1);

g) all details concerning *in situ* specimen preparation before analysis (including argon ion cleaning, specimen heating, fracture, etc.) (see NOTE 2).

NOTE 1 Guidelines for preparation and mounting of specimens are given in ISO 18116.

NOTE 2 Handling of specimens prior to analysis is described in ISO 18117.

4.2.2 Analytical conditions

A detailed list of the analytical conditions shall be recorded in the record book and/or the electronic log (sufficient information shall be recorded to allow the measurements to be repeated at a later date). The information shall include:

a) the name or identification of equipment used;

b) the X-ray source used and the polarization of the beam, if relevant (Al Kα, Mg Kα, monochromated Al Kα, synchrotron, etc.);

c) the X-ray power (record a minimum of two of the following three parameters: power, anode voltage, emission current);

d) the analyser input and exit slit widths, if adjustable, and details of any other resolution settings;

e) the analyser pass energy (in eV) or retardation ratio;

f) the geometry of irradiation (the direction of the X-ray beam relative to the direction of the detected photoelectron), important for quantitative analysis; the specimen-anode distance in the case of non-monochromated X-ray excitation, if this distance is known.

g) the take-off angle used for the measurement;

h) the analysis chamber pressure before and during analysis;

i) the area of analysis (as defined by the aperture setting and lens magnification, or as the beam diameter in systems in which the analysis area is determined by the diameter of the X-ray spot);

j) the start energy (preferably as a binding energy or a kinetic energy);

k) the end energy or scan width;

l) the number of data points, expressed as an integer or as volts/step, and the width of the energy channel;

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m) the acquisition time, expressed as the time/step or as a total time indicating the measurement time and the X-ray exposure time;

n) the charge compensation conditions, if charge compensation is used;

o) the value of the acceptance angle for photoelectrons, if this is an instrumental variable;

p) when an instrument can be operated in a variety of lens modes, the mode used.

All the above information shall be subsequently given to the customer, if requested, along with the analysis of the XPS data by the instrument operator. The customer and the analyst will define the format used to transfer this information. For example, the experimental information may be contained in the appendix or the experimental section of a report.

The binding-energy scale of the X-ray photoelectron spectrometer shall be calibrated either in accordance with ISO 15472 or in accordance with the manufacturer's documented calibration procedure.

4.3 Spectra

All XPS spectra supplied to a customer shall include the following minimum information:

a) peak or region labels (e.g. C 1s, Cu 2p3/2);

b) an abscissa label, e.g. binding energy, E_B, or kinetic energy, E;

c) abscissa tic marks showing the energy as width of scan, e.g. 0 eV to 1 200 eV, or as energy/division;,

d) an ordinate label, showing counts, counts/s or simply a scale in arbitrary units;

e) ordinate tic marks showing the intensity as counts/s per division or counts per division (counts per channel or counts/s per channel);

f) the total acquisition time in the displayed region;

g) any energy reference used, such as C 1s (C−H) (= 285 eV) and whether the energy scale has been corrected to this reference;

h) details of all the data-processing functions applied to the raw spectrum, for example smoothing, transmission function correction, spike removal.

Further information may be included at the discretion of the analyst or at the request of the customer. Examples of XPS spectra are shown in Annex A (see Figures A.1 and A.2).

4.4 Quantitative information

When the XPS data are processed and supplied to a customer as quantitative data, the following information on the method of quantification shall also be made available to the customer, if required:

a) the quantification model, e.g. homogeneous solid, homogeneous solid under a contamination layer, layered solid;

b) the name and version of the data-processing software used;

c) the type of background fitted to the data, with start and end points, if appropriate;

d) the sensitivity factors used (including whether by height or by area) and their source, e.g. manufacturer's, in-house standards, theory;

e) any other correction terms used and their justification, e.g. specimen roughness, backscattering, matrix effects;

f) the estimated error, as discussed for example in ISO 20903;

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g) when the atomic fraction of components having particular chemical states are reported, the method used to separate the peak height or peak area from adjacent peaks;

h) if curve fitting (peak synthesis) was used, the function(s) used (e.g. Gaussian), any constraints, and a measure of the goodness of fit;

i) details of any corrections made for deadtime, spectrometer window, etc.;

j) details of any elements specifically excluded from the quantification procedure (such as carbon surface contaminant).

4.5 Compositional depth profiles

Compositional depth profiles may be presented as a montage of spectra or as depth profiles derived from peak area or peak height measurements on spectra. If the data are quantified, the factors listed in 4.4 shall be considered, and noted where appropriate. In addition, the depth of analysis of ion-sputtered depth profiles may be estimated with respect to the sputter rate of a standard material under the same conditions (see Figure A.3 in Annex A).

NOTE The measurement of sputtered depth in depth profiling is described in ISO/TR 15969.

Where depth profiles are acquired from taper sections or dimpled craters, the depth can be estimated by geometrical calculation.

The following information shall be available to be supplied with the compositional depth profiles, if required:

a) an ordinate label, e.g. atomic fraction (together with details of quantification as outlined in 4.4), or nominal counts;

b) ordinate tic marks showing the atomic fraction, counts/s, counts or simply a scale in arbitrary units);

c) an abscissa label, i.e. sputtering time or estimated depth;

d) abscissa tic marks showing time or depth;

e) for ion-sputtered profiles, the sputtering rate and the sputtering-rate calibration material used;

f) details of the ion beam energy and diameter, the ion beam current and the area sputtered or rastered.

4.6 Maps and linescans

When XPS maps or linescans (see Annex A, Figure A.4) are provided, the following information shall be supplied to the customer, as required (if the information is provided as part of a depth profile, also include the details requested in 4.5):

a) the identity of XPS peak and/or area that has been mapped, together with details of any background removal procedure;

b) details of the map field of view (X and Y directions);

c) details of the map field of view calibration in the X and Y directions;

d) details of the property mapped, e.g. atomic percentage, together with a scale indicating the colour, if appropriate;

e) the method used to obtain the map, e.g. parallel acquisition, stage scanning, X-ray beam scanning, input lens scanning.

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4.7 Chemical-state data

If the XPS spectra are analysed for chemical-state information, the following experimental information shall be attached to the collection of spectra:

a) the element and the chemical formula corresponding to each chemical state (if spectra are provided, clearly indicate the binding energy of each component);

b) reference to a database containing the assignments;

c) the FWHM (full width at half maximum) of the peaks, if measured;

d) if curve fitting (peak synthesis) was used, the name and version of the software, the function(s) used (e.g. Gaussian), any constraints, and a measure of the goodness of fit;

e) the kinetic energy of any Auger peaks detected and the value of any Auger parameters calculated.

5 Release of data to the customer

This will be covered by the relevant International Standard on reporting results or suitable in-house procedures, but shall include, as a minimum, the name and signature (or other identifying mark or stamp) of the analyst and the date of issue.

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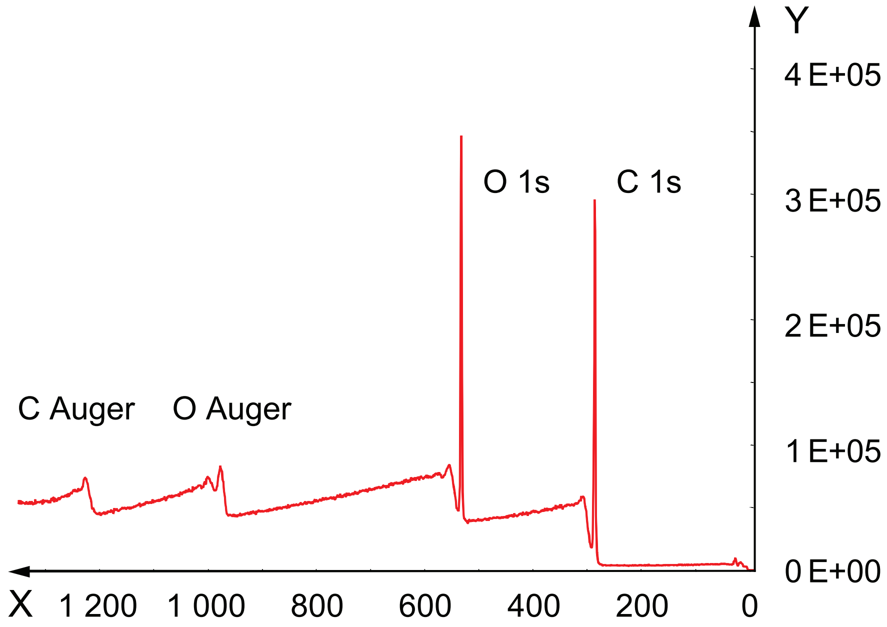
Annex A
(informative)

Examples of spectra

Figures A.1 and A.2 are examples of, respectively, a survey and a high-resolution spectrum of a photoresist poplypropylene coating (dry-film side). Figure A.3 is an example of a depth profile and Figure A.4 shows examples of chemical maps.



Key

X binding energy (eV)

Y counts/s

Further details, such as acquisition time, energy scale calibration and charge correction, are recorded in the record book or electronic log. (Data supplied by Thermo Fisher Scientific.)

Figure A.1 — Photoresist polypropylene coating — Survey spectrum with minimum information

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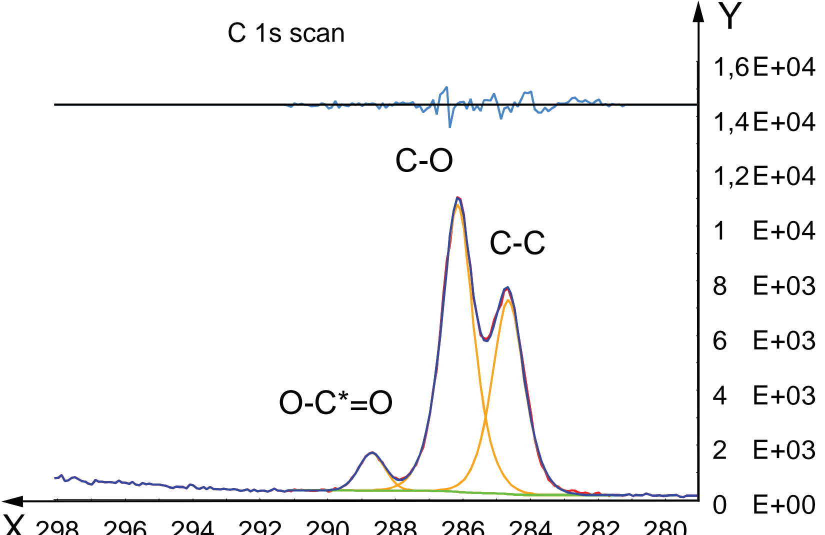

Key

X binding energy (eV)

Y counts/s (residual × 2)

Details of fitting parameters are recorded in the record book or electronic log. (Data supplied by Thermo Fisher Scientific.)

Figure A.2 — Photoresist polypropylene coating — C 1s spectrum fitted using standard commercial software

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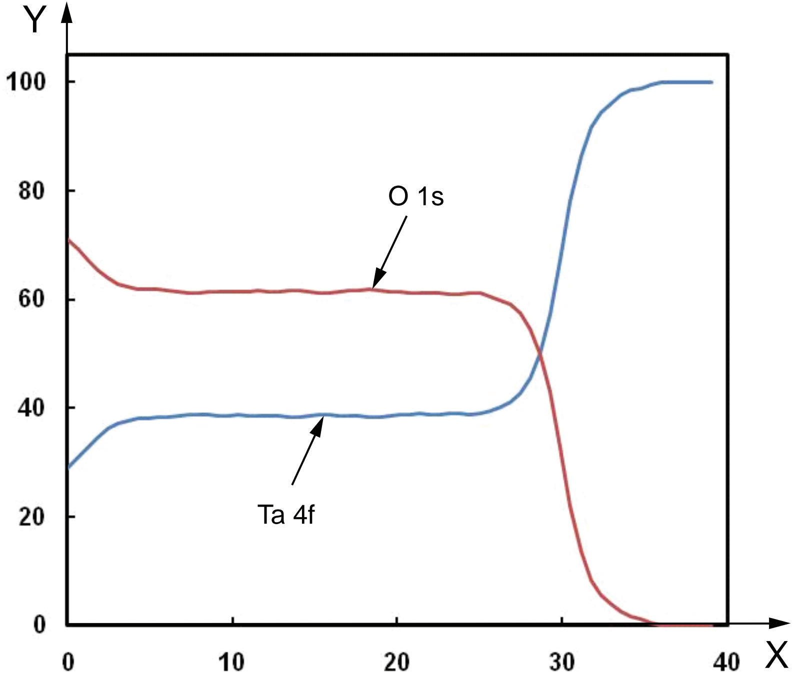


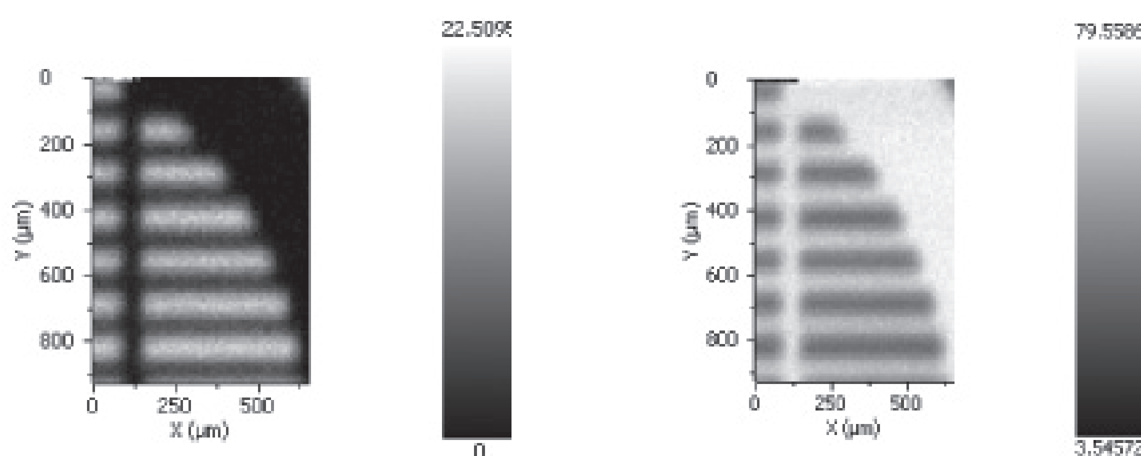
Key

X depth (nm)

Y atomic concentration (%)

A Ta_2O_5 layer on Ta was analysed by XPS sputter profiling. Conditions: Argon ion beam energy 1 keV, beam current 3 µA, scanned area 4 mm × 2 mm, angle 58° from specimen normal, no rotation, total etch time 900 s. Etch rate calibrated against a Ta_2O_5 standard. The profiles were then generated using a linear least-squares fitting software. (Data supplied by Thermo Fisher Scientific.)

Figure A.3 — Example of depth profiling using XPS



a) Atomic percent profile for C 1s CF_2 **b) Atomic percent profile for C 1s C–C**

Conditions: Stage mapping, Al Kα monochromated X-ray spot size 30 µm, step size 10 µm, analyser pass energy 150 eV, 64-channel snapshot spectrum at each pixel, linear background subtracted from C 1s envelope, peak-fitting applied, quantification from fitted peaks. (Data supplied by Thermo Fisher Scientific.)

Figure A.4 — Examples of XPS maps

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[2] ISO/TR 15969, *Surface chemical analysis — Depth profiling — Measurement of sputtered depth*

[3] ISO 18116, *Surface chemical analysis — Guidelines for preparation and mounting of specimens for analysis*

[4] ISO 18117, *Surface chemical analysis — Handling of specimens prior to analysis*

[5] ISO 20903, *Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Methods used to determine peak intensities and information required when reporting results*

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