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Surface chemical analysis — X-ray photoelectron spectroscopy — Reporting of methods used for charge control and charge correction

*Analyse chimique des surfaces — Spectroscopie de photoélectrons
— Indication des méthodes mises en oeuvre pour le contrôle et la
correction de la charge*

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Contents

	Page
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Symbols and abbreviated terms	1
5 Apparatus	2
6 Calibration of binding-energy scale	2
7 Reporting of information related to charge control	2
7.1 General.....	2
7.2 Information about specimen.....	2
7.2.1 Specimen form.....	2
7.2.2 Specimen dimensions.....	2
7.2.3 Specimen mounting methods.....	2
7.2.4 Specimen treatment prior to or during analysis.....	3
7.3 Instrument and operating conditions.....	3
7.4 General method for charge control.....	3
7.5 Reasons for needing charge control and for choosing the particular method for charge control.....	3
7.6 Values of experimental parameters.....	4
7.7 Information on the effectiveness of the method of charge control.....	4
7.7.1 Adequacy of charge control.....	4
7.7.2 Damage assessment.....	4
8 Reporting of method(s) used for charge correction and the value of that correction	4
8.1 General.....	4
8.2 Approach.....	4
8.3 Value of correction energy.....	5
Annex A (informative) Description of methods of charge control and charge correction	6
Bibliography	12

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 19318:2004), which has been technically revised.

The main changes compared to the previous edition are as follows:

- [Clause 7](#) has been reorganized and [7.7](#) (effectiveness of charge control) has been updated;
- [Annex A](#) has been updated, in particular [A.2.1](#) (specimen damage), [A.2.5.2](#) (specimen isolation) and [A.3.3](#) (adventitious-hydrocarbon referencing);
- up-to-date bibliographical references have been added throughout the document.

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.

Introduction

X-ray photoelectron spectroscopy (XPS) is widely used for the characterization of surfaces of materials. Elements in the test specimen (with the exception of hydrogen and helium) are identified from comparisons of the binding energies of their core levels, determined from measured photoelectron spectra, with tabulations of these binding energies for the various elements. Information on the chemical state of the detected elements can frequently be obtained from small variations (typically between 0,1 eV and 10 eV) of the core-level binding energies from the corresponding values for the pure elements. Reliable determination of chemical shifts often requires that the binding-energy scale of the XPS instrument be calibrated with an uncertainty that could be as small as 0,1 eV.

The surface potential of an insulating specimen will generally change during an XPS measurement due to surface charging, and it is then difficult to determine binding energies with the accuracy needed for elemental identification or chemical-state determination. There are two steps in dealing with this problem:

- a) experimental steps can be taken to minimize the amount of surface charging (charge-control methods);
- b) corrections for the effects of surface charging can be made after acquisition of the XPS data (charge-correction methods).

Although the build-up of surface charge can complicate analysis in some circumstances, it can be creatively used as a tool to gain information about a specimen.

The amount of induced charge near the surface, its distribution across the specimen surface, and its dependence on experimental conditions are determined by many factors including those associated with the specimen and characteristics of the spectrometer. Charge build-up is a well-studied,^[6,7] three-dimensional phenomenon that occurs along the specimen surface and into the material. Charge build-up can also occur at phase boundaries or interface regions within the depth of the specimen that is irradiated by X-rays. Some specimens undergo time-dependent changes in the level of charging because of chemical changes or volatilization induced by photoelectrons and secondary electrons, X-rays, or heating. It is possible that such specimens will never achieve steady-state potentials.

There is no universally applicable method or set of methods for charge control or for charge correction.^[8-10] This document specifies the information to be provided to document the method of charge control during data acquisition or the method of charge correction during data analysis, or both. [Annex A](#) describes common methods for charge control and charge correction that can be useful for many applications. The particular charge-control method that is chosen in practice depends on the type of specimen (e.g. powder, thin film or thick specimen), the nature of the instrumentation, the size of the specimen, and the extent to which the specimen surface might be modified by a particular procedure.

This document identifies information on methods of charge control or charge correction, or both, to be included in reports of XPS measurements (e.g. from an analyst to a customer or in publications) in order to evaluate, assess and reproduce data on insulating materials and to ensure that measurements on similar materials can be meaningfully compared. It enables published binding energies to be used with confidence by other analysts and will lead to the inclusion of more reliable data in XPS databases.

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Surface chemical analysis — X-ray photoelectron spectroscopy — Reporting of methods used for charge control and charge correction

1 Scope

This document specifies the minimum amount of information spectroscopy to be reported with the analytical results to describe the methods of charge control and charge correction in measurements of core-level binding energies for insulating specimens by X-ray photoelectron. It also provides methods for charge control and for charge correction in the measurement of binding energies.

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*

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3 Terms and definitions (standards.iteh.ai)

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 abbreviated terms

BE	binding energy, in eV
BE_{corr}	corrected binding energy, in eV
BE_{meas}	measured binding energy, in eV
$BE_{\text{ref,meas}}$	measured binding energy of a reference material, in eV
BE_{ref}	reference binding energy, in eV
FWHM	full width at half maximum amplitude of a peak in the photoelectron spectrum above the background, in eV
XPS	X-ray photoelectron spectroscopy
Δ_{corr}	correction energy to be added to measured binding energies for charge correction, in eV

5 Apparatus

One or more of the charge-control techniques, also called charge-neutralization techniques, mentioned in [Clause A.2](#) can be employed in most XPS spectrometers. The XPS instrument shall be operated in accordance with the manufacturer's instructions or other documented procedures.

Some of the techniques outlined in [Clause A.2](#) require special apparatus, such as an electron flood gun or a source for evaporative deposition of gold.

Certain specimen-mounting procedures, such as mounting the specimen under a fine metal mesh^[11], can enhance electrical contact of the specimen with the specimen holder, or reduce the amount of surface charge build-up. This and other methods of specimen mounting to reduce static charge are described in References [\[4\]](#) and [\[5\]](#).

6 Calibration of binding-energy scale

The binding-energy scale of the X-ray photoelectron spectrometer shall be calibrated using ISO 15472 or another documented method before application of this document.

7 Reporting of information related to charge control

7.1 General

Methods commonly used to control the surface potential and to minimize surface charging are summarized in [Clause A.2](#). Information on the critical specimen and experimental conditions, as specified in [7.2](#) through [7.7](#), shall be reported for individual specimens or collections of similar specimens.

7.2 Information about specimen

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7.2.1 Specimen form

The form of the specimen shall be reported. The physical nature, source, preparation method and specimen structure can influence charging behaviour.^[2]

EXAMPLE 1 Powder.

EXAMPLE 2 Thin film spin-cast on silicon.

EXAMPLE 3 Macroscopic mineral specimen.

7.2.2 Specimen dimensions

The size and shape of a specimen can have a significant effect on the extent of specimen charging. The shape of the specimen shall be reported together with approximate values of the dimensions of the specimen or of any relevant specimen features (e.g. particle diameters).

7.2.3 Specimen mounting methods

Specimen mounting and contact with the specimen holder can significantly impact charging.^[10,4,5,2,3] The method by which a specimen is mounted, including information about special methods used to increase conductivity or isolate a specimen from ground, shall be reported.

EXAMPLE 1 Powder specimen pressed into foil, which was attached to a specimen holder using tape.

EXAMPLE 2 1 ml of contaminated liquid deposited on a silicon substrate and dried prior to analysis.

EXAMPLE 3 Specimen held to specimen holder using conductive adhesive tape of a specified type.

EXAMPLE 4 Corroded specimen held on specimen holder by metal screw.

7.2.4 Specimen treatment prior to or during analysis

The specimen treatment prior to or during analysis shall be reported, including any physical or chemical treatment that can affect charging of the specimen during XPS measurements.

EXAMPLE 1 Gold deposition.

EXAMPLE 2 Ar gas implantation from sputter ion source.

NOTE Such treatment of the specimen can modify the surface composition as well as the electrical conductivity, and hence charging, of the surface region.

7.3 Instrument and operating conditions

The instrument operating conditions shall be reported, including details of the:

- particular XPS instrument;
- nature of the X-ray source;
- approximate size of the X-ray beam on the specimen surface;
- analyser pass energy;
- measure of energy resolution such as the FWHM of the silver $3d_{5/2}$ photoelectron line for the selected operating conditions;
- angle between the specimen normal and the X-ray source;
- use or not of a magnetic lens.

7.4 General method for charge control

The particular instrumental component(s) used for charge control shall be identified.

EXAMPLE 1 Electron flood gun.

EXAMPLE 2 Electron flood gun in combination with an ion gun.

EXAMPLE 3 Specimen heating.

EXAMPLE 4 Irradiation with ultraviolet light.

EXAMPLE 5 Vendor XYZ charge neutralization system.^[10]

If the components used are not standard for the XPS instrument, information shall be provided on the manufacturer or on the relevant design characteristics.

7.5 Reasons for needing charge control and for choosing the particular method for charge control

The reasons for needing charge control and for choosing a particular method shall be reported.

EXAMPLE 1 The portion of the specimen of interest was isolated from ground. Flood gun electrons were supplied for charge compensation using the standard flood gun for this instrument.

EXAMPLE 2 Experience with similar specimens indicated that differential charging was likely. To obtain good spectra, these specimens were totally isolated from ground. The application of the combined fluxes of a low-energy electron flood gun and a low-energy ion flux produced well-resolved peaks.

EXAMPLE 3 Initial spectra without any charge control showed peak shifting and broadening. Placing a grounded fine grid above the specimen solved these problems without leading to a significant signal due to the grid material. This method is easy to apply and is used routinely in measurements with similar specimens.

7.6 Values of experimental parameters

Values of parameters used to control charge, such as flood gun settings, shall be reported. Information about typical parameters for some charge neutralization systems on modern instruments is provided in Reference [10].

EXAMPLE For the flood gun, the cathode voltage was -5 V (with respect to instrumental ground), the emission current was 20 mA, and the gun cathode was 5 cm from the specimen.

7.7 Information on the effectiveness of the method of charge control

7.7.1 Adequacy of charge control

The adequacy of the charge-control methods for the type of analysis being conducted shall be established. FWHMs and the binding energies (BE_{meas}) of peaks in the measured spectra, after charging effects have been minimized, but before any charge correction has been made, provide one useful method for determining adequacy of the charge-control method. To document the effectiveness of the procedure(s) used to produce appropriate BE and FWHM measurements, it can be useful to have as a comparison a measurement of the FWHM of at least one photoelectron peak of similar chemistry in another specimen that is known to be conductive or for which the method of charge control is believed to be effective.

EXAMPLE 1 The FWHM of the oxidized Si 2p photoline was reduced from 2,4 eV to 1,6 eV by application of a flood gun. The 1,6 eV width is consistent with measurements made on a thin SiO₂ layer on Si.

EXAMPLE 2 The ability to control charge compensation over a wide energy range can be documented by measuring the energy separation between different photoelectron peaks from the same element. The adequacy of such a measurement assumes that there are no complications due to chemical state changes with depth or the presence of second phases.

7.7.2 Damage assessment

It is recommended that specimens be examined for the presence or absence of specimen damage due to sample charging or the impact of the charge neutralization method (see A.2.1) and that the results be recorded. If damage is observed, changes to the charge neutralization parameters can need to be adjusted and the changes recorded.

EXAMPLE Survey scans at the start and end of data collection showed no changes suggestive of intensity of peak structure changes due to damage.

8 Reporting of method(s) used for charge correction and the value of that correction

8.1 General

Many of the methods commonly used for charge correction are summarized in Clause A.3. The critical specimen and experimental parameters in 8.2 and 8.3 shall be reported.

8.2 Approach

The general method for correcting measured binding energies (peak positions) for charging effects shall be specified in sufficient detail so that the method can be reproduced and the effectiveness judged.

8.3 Value of correction energy

Information shall be given on the magnitude of the correction energy (Δ_{corr}) for each spectrum and how this correction energy was determined. The corrected binding energies and values of the reference energies shall be reported.

The correction energy (Δ_{corr}) is determined by taking the difference between the measured binding energy of a reference line ($BE_{\text{ref,meas}}$) and an appropriate binding energy value (BE_{ref}) for the reference line (obtained from the literature or other trusted source) using [Formula \(1\)](#):

$$\Delta_{\text{corr}} = BE_{\text{ref}} - BE_{\text{ref,meas}} \quad (1)$$

The corrected binding energy for another photoelectron peak in the same spectrum (BE_{corr}) can then be found from the sum of the measured binding for that peak (BE_{meas}) and the correction energy:

$$BE_{\text{corr}} = BE_{\text{meas}} + \Delta_{\text{corr}} \quad (2)$$

NOTE [Formulae \(1\)](#) and [\(2\)](#) apply only when charge compensation has adequately removed differential charging effects.

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