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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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Foreword

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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 19318 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 5, *Auger electron spectroscopy*.

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Introduction

X-ray photoelectron spectroscopy (XPS) is widely used for 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. First, experimental steps can be taken to minimize the amount of surface charging (charge-control methods). Second, corrections for the effects of surface charging can be made after acquisition of the XPS data (charge-correction methods). Although the buildup 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 buildup is a well-studied [1, 2] three-dimensional phenomenon that occurs along the specimen surface and into the material. Charge buildup may 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. Such specimens may never achieve steady-state potentials.

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There is, at present, no universally applicable method or set of methods for charge control or for charge correction [3, 4]. This International Standard specifies the information that shall be provided to document the method of charge control during data acquisition and/or the method of charge correction during data analysis. Information is given in Annex A on common methods for charge control and charge correction that can be useful for many applications. The particular charge-control method that may be 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 International Standard is expected to have two main areas of application. First, it identifies information on methods of charge control and/or charge correction 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. Second, adherence to this International Standard will enable 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 International Standard specifies the minimum amount of information describing the methods of charge control and charge correction in measurements of core-level binding energies for insulating specimens by X-ray photoelectron spectroscopy that shall be reported with the analytical results. Information is also provided on methods that have been found useful for charge control and for charge correction in the measurement of binding energies.

2 Normative reference

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.

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ISO 18115, *Surface chemical analysis — Vocabulary*

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3 Terms and definitions

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

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_{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

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5 Apparatus

5.1 One or more of the charge-control techniques mentioned in Clause A.2 may be employed in most XPS spectrometers. The XPS instrument shall be operated in accordance with the manufacturer's or other documented procedures.

5.2 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.

5.3 Certain specimen-mounting procedures, such as mounting the specimen under a fine metal mesh [5], can enhance electrical contact of the specimen with the specimen holder, or reduce the amount of surface charge buildup. This and other methods of specimen mounting to reduce static charge are described in detail in ASTM E 1078 and ASTM E 1829 [6, 7].

6 Calibration of binding-energy scale

The binding-energy scale of the X-ray photoelectron spectrometer shall be calibrated using ISO 15472 [8] or another documented method before application of this International Standard.

7 Reporting of information related to charge control

7.1 Methods of charge control

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Many of the methods commonly used to control the surface potential and to minimize surface charging are summarized in Clause A.2. Information on the following critical specimen and experimental conditions shall be reported for individual specimens or collections of similar specimens.

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7.2 Information on specimen

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 [1, 2]. 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 specimen holder by tape

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

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EXAMPLE 3 Specimen held to 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 includes any physical or chemical treatment that can affect charging of the specimen during XPS measurements.

NOTE Such treatment of the specimen may 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 include details of the particular XPS instrument, the nature of the X-ray source, the approximate size of the X-ray beam on the specimen surface, the analyser pass energy, a measure of energy resolution such as the FWHM of the silver $3d_{5/2}$ photoelectron line for the selected operating conditions, the angle between the specimen normal and the X-ray source, and the 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

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EXAMPLE 2 Electron flood gun in combination with an ion gun

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EXAMPLE 3 Specimen heating

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EXAMPLE 4 Irradiation with ultraviolet light

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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 As supplied to us, the portion of the specimen of interest was isolated from ground. We supplied flood gun electrons 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, we totally isolated these specimens 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 our 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.

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.

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