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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 œuvre pour le contrôle et la correction de la charge

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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**

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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.

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.

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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*

3 Terms and definitions

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
$BE_{ref,meas}$	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

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