INTERNATIONAL STANDARD

ISO 16129

Second edition
2018-11

Surface chemical analysis — X-ray photoelectron spectroscopy — Procedures for assessing the day-to-day performance of an X-ray photoelectron spectrometer

Analyse chimique des surfaces — Spectroscopie de photoélectrons X — Modes opératoires d'évaluation de la performance au jour le jour d'un spectromètre de photoélectrons X

Reference number
ISO 16129:2018(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 16129:2012), which has been technically revised.

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

XPS instruments are complex, and unsatisfactory performance is not always obvious to an operator. It is therefore necessary to provide a test for the correct operation of the system that can be performed regularly and frequently without interfering excessively with the normal work of the laboratory.

A full diagnostic test can require many hours or even days; such a test can be appropriate only when the instrument is known to have a fault that needs to be remedied or following a major maintenance procedure. Data acquired before a problem is uncovered become suspect if the spectrometer has not been routinely tested, leading to a loss of confidence in those data. If a regular check of the instrument is made, changes in performance can be monitored and corrective action taken in good time to ensure that the data supplied are fit for purpose. In the event that a serious fault is uncovered, then only the data since the last check can be in doubt and need to be repeated.

The purpose of this document is to provide users with a procedure which is not excessively time-consuming so that it can be completed on a regular and frequent basis – daily if required. The user will then gain an awareness of the current characteristics of the instrument so that a decision can be made as to whether or not a more complete and time-consuming action is required to return the instrument to a satisfactory level of performance.

This procedure is intended to be applied to an XPS instrument that has been correctly calibrated and aligned in accordance with ISO standards or manufacturer's instructions. It is designed to highlight aspects of the instrument's characteristics that differ significantly from those that were measured immediately following the calibration procedure. The procedure does not show how the instrument can be returned to its original state. Instead, it guides the user to possible areas of concern. The procedure provides data that can be used in control charts, allowing trends to be observed and acted upon before data quality deteriorates to an unacceptable level for the needs of the analyst.

Surface chemical analysis — X-ray photoelectron spectroscopy — Procedures for assessing the day-to-day performance of an X-ray photoelectron spectrometer

1 Scope

This document is designed to allow the user to assess, on a regular basis, several key parameters of an X-ray photoelectron spectrometer. It is not intended to provide an exhaustive performance check, but instead provides a rapid set of tests that can be conducted frequently.

Aspects of instrument behaviour covered by this document include the vacuum, measurements of spectra of conductive or non-conductive test specimens and the current state of the X-ray source. Other important aspects of the instrument performance (e.g. lateral resolution) fall outside the scope of this document.

The document is intended for use with commercial X-ray photoelectron spectrometers equipped with a monochromated Al Kα X-ray source or with an unmonochromated Al or Mg Kα X-ray source.

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, definitions and abbreviations

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/

The following abbreviations are used:

FWHM	full width at half maximum
PET	poly(ethylene terephthalate)
PTFE	polytetrafluoroethylene
XPS	X-ray photoelectron spectroscopy

4 Initial approach

Most instruments are fitted with a vacuum gauge or gauges. These shall be read frequently and the reasons for large variations understood. A large increase in the pressure can be due to the properties of a test specimen inside the instrument, a fault in the pumping system, an increase in the temperature of the vacuum system or a leak.

Similarly, most instruments have status indicators, either for the system as a whole or for sub-systems or modules. Examples of such indicators include water flow rate, data system communications status and electrical power. These can be visible as part of the instrument hardware itself or on screen through an instrument control (data acquisition) system. Such indicators again shall be carefully monitored, along with any measured values that are reported.

5 Initial instrument calibration, alignment and assessment

Before undertaking the procedure described in the following clauses, it is essential that the instrument be calibrated and aligned to an optimum performance level. This is achieved by following the relevant International Standards (References [5] to [9]) or the manufacturer's instructions. Choose the two settings of the instrument operating conditions that are needed to obtain survey spectra and high-resolution spectra. These should be settings that you regularly use and are described in Clause 7. Since this is a rapid check, only these two settings are chosen, but these can show faults that are common to all settings. These settings shall always be used in future checks unless they are later found to be less effective than other settings. If the designated settings are changed, data at both the new and old settings shall be recorded at the time of change.

6 Test specimen selection

6.1 General information

Three types of test specimen are required for the full procedure described in this document: a conductive specimen, a non-conductive specimen and a specimen suitable for assessing the quality of the X-ray beam (e.g. X-ray spot size, shape and uniformity). The conductive specimen provides information that the basic energy and intensity calibrations of the instrument are consistent from day to day. When using a non-monochromatic X-ray source with more than one anode material, the survey spectrum shall be examined to check for peaks arising from specimen irradiation from the anode that is not currently being used (this is due to "cross-talk" within the X-ray source). Similarly, when anode coatings wear, X-rays can be emitted from the substrate material, which is often copper. The survey spectrum shall be examined for peaks arising from X-rays produced from the substrate material. When using a magnesium anode, there can be peaks due to radiation from O Kα arising from oxidation of the anode coating. These peaks are often called ghost peaks. In some instances, ghost peaks may be removed from the spectrum by degassing the X-ray source (following the instrument manufacturer's recommended procedure). Table 1 shows the approximate positions of commonly encountered ghost peaks when acquiring an XPS spectrum from silver using Mg Kα radiation.

Table 1 — Examples of approximate positions on the binding-energy scale of frequently encountered ghost peaks in a silver spectrum collected using a magnesium anode

Radiation giving rise to ghost peaks	Photon energy eV	Possible origin of radiation	Peak position on the binding-energy scale eV
Al Kα	1 486,6	From second anode in a twin anode source	135
O Kα	524,9	From oxide on the surface of the magnesium anode	1 097
Cu Lα	929,7	From anode substrate	692

NOTE The photon energy of Mg Kα radiation is 1 253,6 eV.

The non-conductive specimen is required to confirm that the charge compensation system is operating satisfactorily when non-conducting specimens are being analysed. The nature of the specimen that is required for assessing the X-ray beam depends upon the type of instrument being used. If the analysis position is visible during the normal operation of the instrument, the quality of the focus and the alignment of the X-ray beam from a monochromated source can be assessed using a phosphor specimen. If the analysis position is not visible in normal operation and the instrument is capable of imaging, a uniform (there should be no features visible in an image of the specimen when the instrument is in its optimum condition), conductive specimen can be used; this can be the conductive specimen mentioned previously. All specimens used shall be large enough to completely fill the defined analysis region of the spectrometer.

6.2 The conductive specimen

A suitable conductive specimen shall be selected. This should be a material that produces several peaks in the photoelectron spectrum. Preferably, these peaks should be widely spaced in binding energy. The specimen shall be one whose surface can be cleaned easily by sputtering with noble-gas ions.

A pure (≥99,8 %) specimen of silver foil is suitable for this measurement and is recommended. If, however, a different material is commonly analysed in the user's laboratory and conforms to the above criteria, this may be used instead. For convenience, it is assumed here that silver has been selected as the conductive reference material. The same conductive specimen shall be used for all measurements.

6.3 The non-conductive specimen

Non-conductive specimens, and conductive specimens with a non-conductive surface layer, charge under the X-ray flux, resulting in shifts in the peak binding energies relative to the uncharged state.

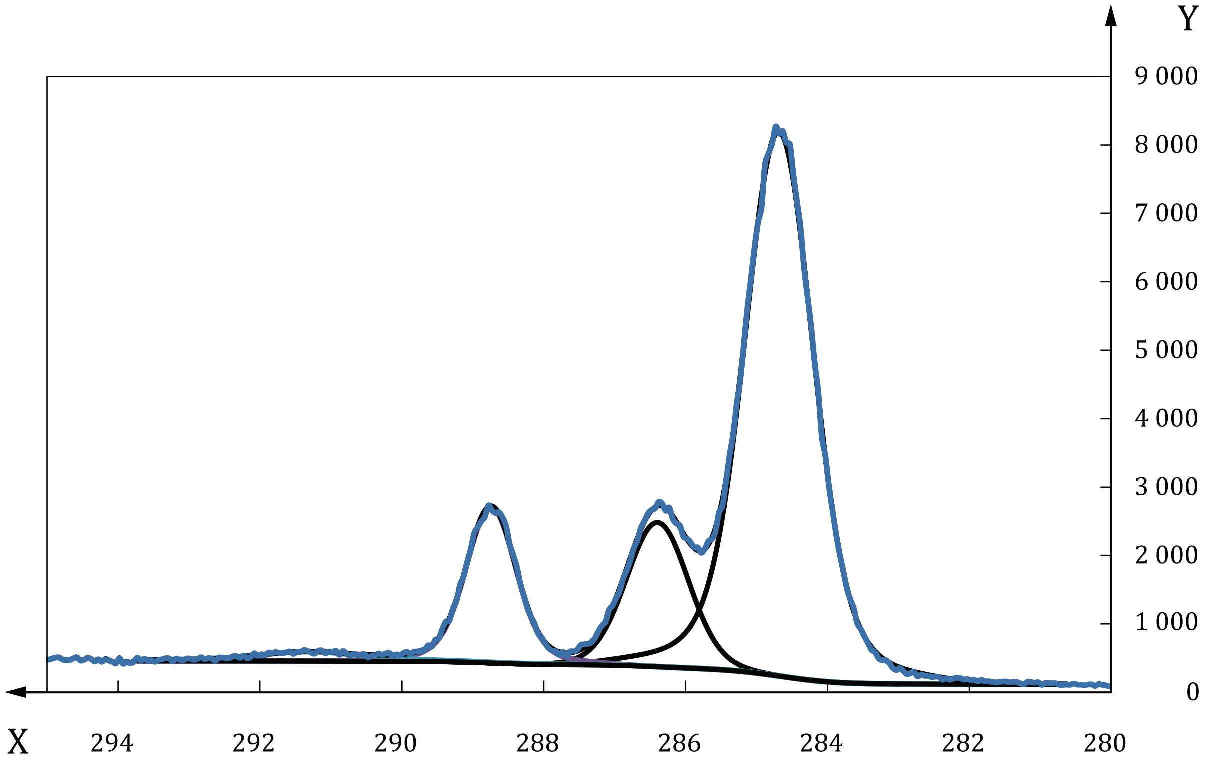
Select a non-conductive material often studied in your laboratory, of which you have a good stock and which can be maintained with a surface in a reproducible state. If you are unsure of a material to use, examples that have been found to be useful for tests of instrumental performance are shown in Table 2. Examples are given with different forms. You might wish to select a specimen with a form similar to that of the specimens most commonly analysed with the instrument. Care shall be taken to select a material which does not exhibit degradation under the X-ray beam during the analysis.

Table 2 — Examples of non-conductive specimens that may be used for this procedure

Material	Form	Cleaning
PET	Sheet or fibre	Not required
Laboratory filter paper	Sheet	Not required

Poly(ethylene terephthalate) (PET) has long been used to evaluate both the energy resolution and the effectiveness of charge control in XPS. It shows a structure of three C 1s peaks together with shake-up intensity. The minimum between the peak at the lowest binding energy and the adjacent peak at a separation of ~1,5 eV is highly sensitive to the combination of the instrumental resolution and the uniformity of the charge correction. The ease of achieving suitable and consistent energy resolution will depend on both the operator and the instrumental capability.

An example of the C 1s spectrum from PET is shown in Figure 1.



Key

X	binding energy (eV)
Y	number of counts

Figure 1 — Example of a C 1s spectrum from PET

More recently, careful studies on cellulose-based materials (paper) have indicated that these are suitable materials for use in this test (see References [1] and [2]). It has been shown that laboratory filter paper1) provides reproducible XPS spectra. If using paper, avoid releasing loose fibres into the instrument, as they can cause a deterioration in performance.

Alternatively, the specimen can be one with which the user is familiar. It shall be a material that provides a reliably reproducible spectrum with little or no specimen preparation.

6.4 Specimen for assessing the X-ray source

A phosphor specimen is suitable for those instruments that are fitted with a monochromatic X-ray source and in which the analysis position is visible, preferably with a microscope and camera. The phosphor specimen should be as flat and uniform as possible and produce easily visible light under the X-ray bombardment.

Not all phosphor specimens are vacuum-compatible and not all phosphor materials are designed for optimum excitation by X-rays. Ensure that a phosphor specimen of suitable quality is obtained from the supplier of the instrument or from another reputable supplier.2)

---

1) A suitable type of filter paper is S&S 589 Blue Ribbon Ashless, which can be obtained from Whatman plc, Springfield Mill, James Whatman Way, Maidstone, Kent, ME14 2LE, UK, or one of this company's international distributors. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

2) Suitable phosphor specimens can be obtained from a number of suppliers, including TMS Vacuum Components, Unit 21 Stirling Road, Castleham Industrial Estate, Hastings, East Sussex, TN38 9NP, UK. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this supplier.