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Surface chemical analysis — X-ray photoelectron spectroscopy — Estimating and reporting detection limits for elements in homogeneous materials

Analyse chimique des surfaces — Spectroscopie de photoélectrons par rayons X — Estimation et production de rapports sur les limites de détection des éléments contenus dans les matériaux homogènes

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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 on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *Electron spectroscopies*.

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Introduction

X-ray photoelectron spectroscopy (XPS) is a technique widely employed to measure the chemical composition of material surfaces. In many applications, it is used to either confirm or deny the presence of an elemental species at a surface. In either case, it is important to understand the minimal concentration of the element that can be detected by XPS under the measurement conditions either to provide an assessment of confidence in a result or to understand how the measurement conditions should be changed to achieve the required detection limit.

This document provides a straightforward approach to calculating detection limits in X-ray photoelectron spectroscopy from experimental data in common analytical situations. It also provides informative annexes which allow the uncertainty in the calculated detection limit to be determined (see Annex A) and describe how the XPS detection limit is defined (see Annex B). Example data and calculations are provided in Annex C. Annex D contains useful conversions and references which describe how detection limits may be estimated for an X-ray photoelectron spectrometer in the absence of any data except that from a reference material such as clean silver.

These calculations are of critical importance because the technique is routinely used to measure the concentration of elements, which are present in low concentrations at a material surface, and knowledge of the limit of detection provides a statement of confidence when no element can be detected. Furthermore, if a particular detection limit is required, it permits the analyst to calculate the acquisition time required to achieve the specified limit of detection.

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Surface chemical analysis — X-ray photoelectron spectroscopy — Estimating and reporting detection limits for elements in homogeneous materials

1 Scope

This document specifies a procedure by which elemental detection limits in X-ray photoelectron spectroscopy (XPS) can be estimated from data for a particular sample in common analytical situations and reported. This document is applicable to homogeneous materials and is not applicable if the depth distribution of elements is inhomogeneous within the information depth of the technique.

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*

ISO 18115-2, *Surface chemical analysis — Vocabulary — Part 2: Terms used in scanning-probe microscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1 and ISO 18115-2 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
reference element
chemical element present in the sample for which a peak area and relative composition can be measured

3.2
specified element
chemical element for which the detection limit calculation is being undertaken

4 Symbols and abbreviated terms

A_i	summed intensity of the photoelectron line of element *i*, counts or cps
A_C	critical level of detection for a peak in summed intensity, counts or cps
A_D	minimal detectable summed intensity for a peak at the required level of confidence
AMRSF	average matrix relative sensitivity factor
a_m	coefficients of order *m* in a polynomial equation

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