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

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National foreword

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Foreword

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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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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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at.%	atomic percent concentration
α	average emission angle of electrons relative to the surface normal of the sample
$B(E)$	function of E describing background, counts or cps
BE	binding energy
b	number of data points that define the background under a peak
c	number of data points from b that are used in a summed intensity measurement
cps	counts per second
d	diameter of the atoms of the specified element
$\delta_{A(x)}$	relative uncertainty of peak intensity, A_x
δ_S	relative uncertainty of the detection limit, S
$\delta_{\sigma(\mathrm{B})}$	relative uncertainty of the standard deviation of the background σ_B
$\delta_{X(\mathrm{D})}$	relative uncertainty of the detection limit, X_D
E	numerical value of either BE or KE, eV
E_j	numerical value of either BE or KE of the photoelectron peak used to detect element j, eV
eV	electron volts
ε	step size in spectrum, eV
F	factor by which the acquisition time should be changed to achieve target detection limit
FWHM	full width at half maximum
G	goodness of fit
G_min	minimized value of G
Γ_D	XPS detection limit of a thin overlayer of the specified element expressed as areic density
$I(E)$	function of E describing intensity in a spectrum
i	element within the sample
j	element for which the detection limit is to be estimated
k	coverage factor
λ	inelastic mean free path of electrons with kinetic energy E_j in the sample
KE	kinetic energy
M	number of terms, excluding the constant term, in a polynomial description of B
m	index in a general polynomial equation with integer values 0 to M
N	number of data points in background spectrum
n	data point indicator with integer values 1 to N

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q	factor to account for data smoothing introduced by electron detectors
θ_D	XPS detection limit of a thin overlayer of the specified element expressed in monolayers
$R(E)$	function of E describing the difference between I and B, counts or cps
RSF	elemental relative sensitivity factor
S_i	RSF of the photoelectron line used to measure the concentration of element i
σ_A	standard deviation of a summed intensity measurement
σ_B	standard deviation of I in background region
$T(E)$	factor that transforms $I(E)$ to have units of counts
t_D	XPS detection limit of a thin overlayer of the specified element expressed as a thickness
W_i	FWHM in eV of the photoelectron line used to detect element i
x	element used as a photoelectron intensity reference
X_i	atomic fraction of element i expressed in units of at.%
X_D	minimal detectable concentration of an element expressed in units of at.%
X_T	target XPS detection limit
XPS	X-ray photoelectron spectroscopy
y	number of data points from b that solely describe the peak

5 Calculating and reporting detection limits from XPS data

5.1 General

This clause provides a step-by-step procedure for the calculation of a detection limit for a specified element from XPS data. The data should cover the binding energy (BE) region in which a photoelectron peak is expected from the specified element for which the detection limit is to be calculated. The element may be below detectable levels or present at low concentrations; slightly different procedures will be used in each case. These procedures are described in 5.3.3.2 and 5.3.3.3, respectively. Various simplifying approximations are used in this document to enable the calculations to be performed in a practical manner. The typical uncertainty in the calculated XPS detection limits using this procedure is between 10 % and 25 %, which is sufficient for practical XPS analysis. A flow chart describing the steps required for the collection of data is provided in Figure 1, and a flow chart describing the steps required to calculate XPS detection limits is provided in Figure 2.

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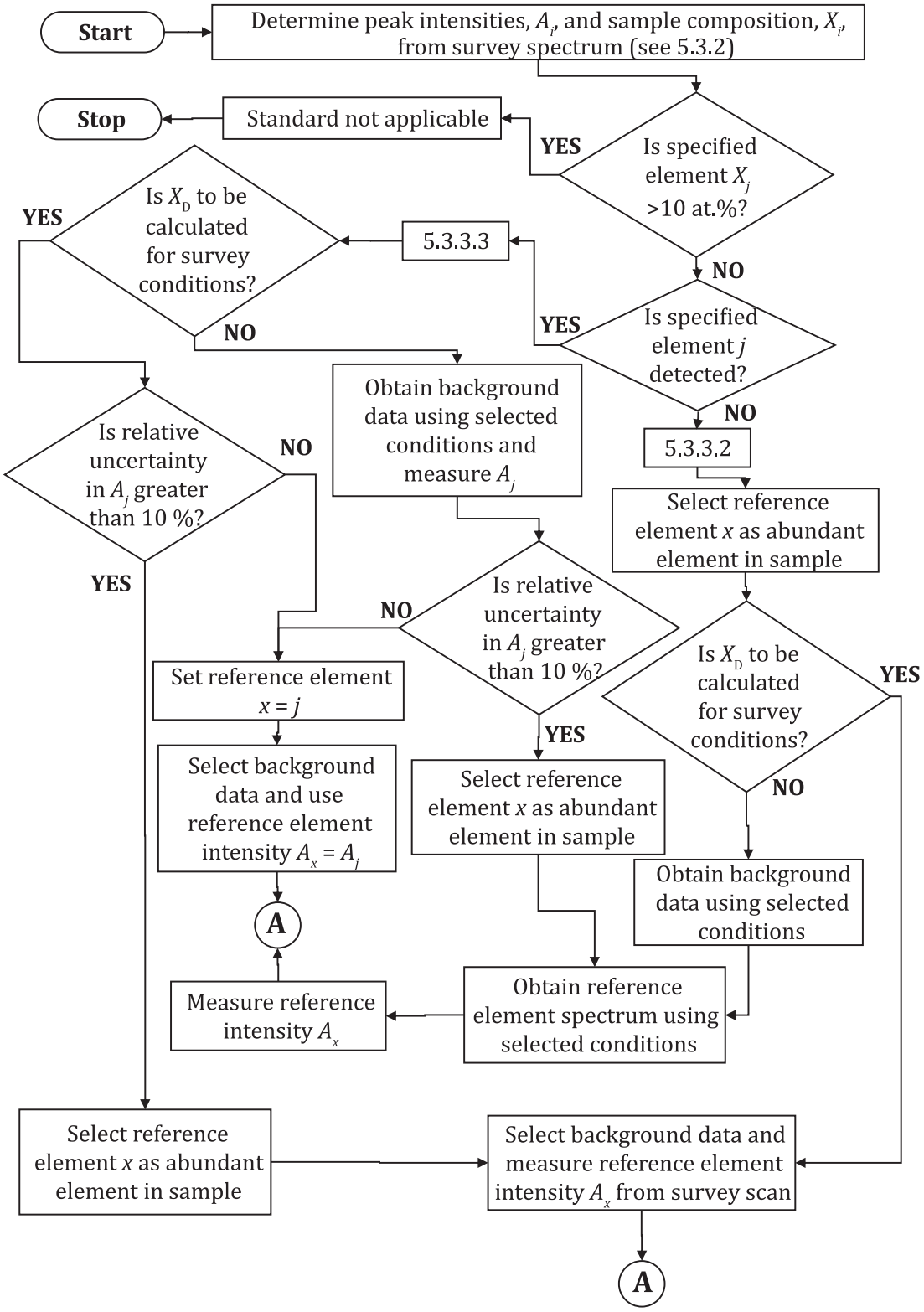

NOTE Point "A" connects to the flow chart in Figure 2.

Figure 1 — Flow chart describing steps in data collection for estimating XPS detection limits

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