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Surface chemical analysis — X-ray photoelectron spectroscopy — Procedures for determining backgrounds

Analyse chimique des surfaces — Spectroscopie de photoélectrons X — Protocoles pour déterminer les fonds continus

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Foreword

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ISO/TR 18392 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 5, *Auger electron spectroscopy*.

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Introduction

This Technical Report gives guidance for determining backgrounds in X-ray photoelectron spectra. The methods of background determination described in this report are applicable for quantitative evaluation of spectra of photoelectrons and Auger electrons excited by X-rays from solid surfaces and surface nanostructures.

The use of background determination in X-ray photoelectron spectroscopy (XPS) has developed from the need (i) for accurate quantitative information on chemical composition (including in-depth composition) of surface/interface layers and nanostructures, (ii) for unambiguous identification of chemical states of surface species and (iii) for extracting electronic-structure information from photoelectron spectra excited from solids. It is therefore necessary to separate the intrinsic part of a spectrum, which is associated with the photoionization or photoexcitation process by the X-radiation of interest in XPS or the Auger-electron decay process and which is needed for further analysis, from other parts of the spectrum (the background) appearing due to other processes. There are widely used procedures available for background subtraction in XPS that are reviewed in detail in References [1] to [4]. Here, the most common procedures and their use are summarized, including methods (i) commonly available in commercial software systems, (ii) available and used in more advanced laboratories and (iii) used in individual laboratories to develop understanding of the processes reflected in the XPS spectra.

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Surface chemical analysis — X-ray photoelectron spectroscopy — Procedures for determining backgrounds

1 Scope

This Technical Report gives guidance for determining backgrounds in X-ray photoelectron spectra. The methods of background determination described in this report are applicable for evaluation of spectra of photoelectrons and Auger electrons excited by X-rays from solid surfaces.

2 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115 [5] apply.

3 Symbols and abbreviated terms

AES Auger electron spectroscopy

PIA Partial intensity analysis

QUASES™ Quantitative analysis of surfaces by electron spectroscopy

REELS Reflection electron energy loss spectroscopy

XPS X-ray photoelectron spectroscopy

4 Types of background in XPS

The electrons produced by X-ray irradiation of surfaces are either photoelectrons (as a result of the primary photoionization process) or Auger electrons (as a result of the secondary, core-hole decay process). Contributions to the measured spectra (i.e., electron energy distributions) from electrons scattered inelastically in the sample, from the secondary electron cascade, and — in the case of excitation by non-monochromatic X-ray irradiation — from photoelectrons induced by X-ray satellites and by bremsstrahlung radiation constitute the background. It is usually not necessary in practical XPS to determine the secondary-electron cascade background at low energies.

In this Technical Report, a description of methods for removing X-ray satellites is given in Clause 5 and for removing inelastic electron scattering in Clause 6. A brief comparison is given in Clause 7 of the effectiveness of procedures for removing the effects of electron inelastic scattering from electron spectra.

NOTE 1 In some cases, the intensity of the intrinsic part of a spectrum is distributed among features attributable to the “no-loss” main peak and to various electronic excitations associated with the creation of the core hole. The latter intrinsic contributions are sometimes denoted as the “intrinsic background”. The identification of the intrinsic loss features and measurement of their intensities can be important for quantitative applications of XPS.

NOTE 2 Time-varying fluctuations of the analytical signal due to sources of noise [5] will lead to uncertainty in the signal intensity. Intensity contributions due to noise are not included in the types of background discussed in this Technical Report.

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5 Removal of X-ray satellites from electron spectra

For XPS with non-monochromated X-ray sources, a fixed satellite structure is associated with the exciting main X-ray line (often Al or Mg $K\alpha$ radiation). These X-ray satellites lead to corresponding features in the XPS spectra.

For selected photoelectron peaks measured with Al or Mg X-ray sources, intensity is removed from higher-kinetic-energy channels corresponding to the energy differences between the $K\alpha_{3,4}$, $K\beta$, etc., X-ray satellite positions and the $K\alpha_{1,2}$ main peak and the corresponding intensity ratios [6] to remove the satellite contributions in the given spectral region. In such a way, scaled photoelectron peaks corresponding to the peaks excited by the X-ray satellites are subtracted. This subtraction process can be applied in turn to remove satellite peaks associated with other photoelectron peaks. The subtraction process may also erroneously remove an equivalent intensity from Auger peaks present in the spectrum if these are mistakenly identified as photoelectron peaks.

6 Estimation and removal of inelastic electron scattering from electron spectra

6.1 General Information

Various procedures have been developed for separating the part of intrinsic origin in the measured photo-excited electron spectra from the contributions due to electrons that are inelastically scattered in the sample [1-4]. These procedures (including those described in Clause 5) are usually applied to XPS data following data acquisition and require digital-data acquisition and handling capability.

Prior to application of a procedure for removing the inelastic electron scattering, a measured spectrum normally should be corrected for the spectrometer response function [7, 8] in cases where the distortion of the spectral shape due to instrumental effects is not negligible. To remove the effect of inelastic electron scattering in the spectrum, two different strategies can be followed: either to remove (subtract) the contribution attributable to electron inelastic scattering from the spectrum, or to include a background component in the model function being used to fit the spectrum. The electron-scattering contribution can be considered either as a background for the whole spectrum or as a sum of tail contributions [9, 10] from individual photoelectron peaks. In the case of background removal/subtraction, the parameters of the background components are fixed and, after creating such a background, can be subtracted from the measured spectrum. In the case of background fitting, some or all of the parameters of the background components are allowed to vary in the fitting process.

NOTE The methods described here for removing the contributions of electron inelastic scattering from a spectrum may not be used for some specific applications of XPS (i.e., total reflection XPS or Auger-photoelectron coincidence spectroscopy) without further consideration.

6.2 Procedures to account for inelastic electron scattering

6.2.1 Introduction

Even in the case of very thin samples, a considerable fraction of the electrons in a spectrum have been inelastically scattered; therefore, the estimation of the background for inelastic electron scattering is very important for quantitative applications. Common procedures for removing the effects of inelastic scattering are briefly described.

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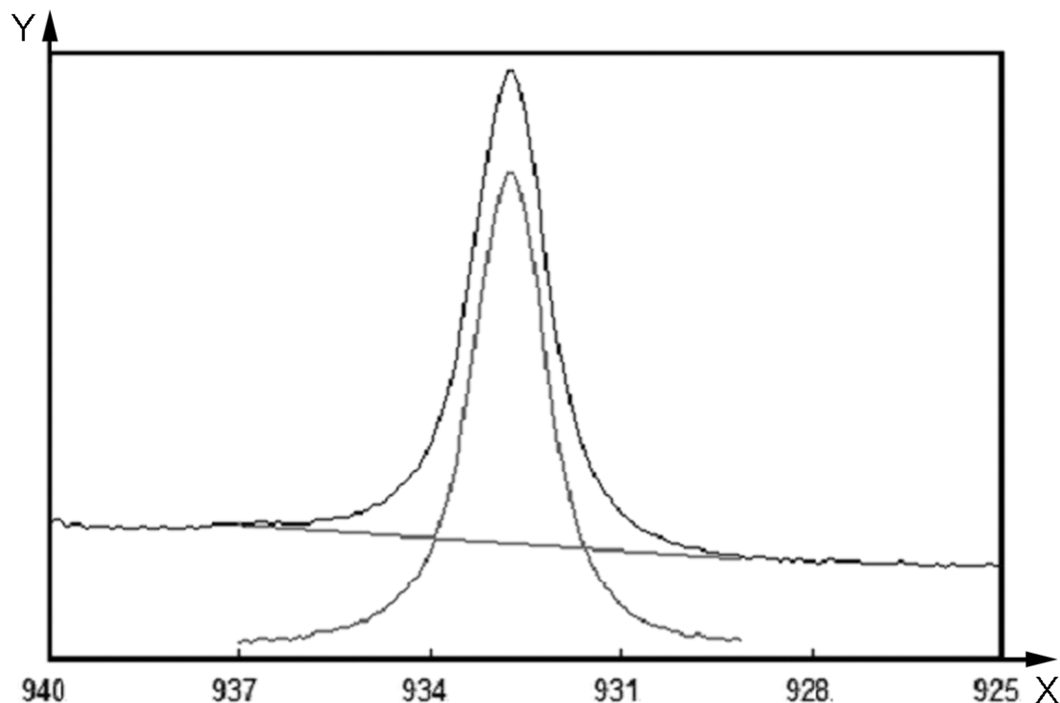
6.2.2 Estimation of the linear background and its removal

In this widely used method, two arbitrarily chosen points in the spectrum are selected and joined by a straight line [2] as an approximation of the true background. These points are generally chosen such that the peak is positioned between them. The intensity values at the chosen points may be the values at the corresponding energies or the average value over a small energy interval around the chosen points. Figure 1 illustrates a linear background for a Cu $2p_{3/2}$ XPS spectrum [11]. This is the most popular method for insulators, where the straight line is horizontal. This approach is used for polymers with great success for peak fitting. However, the use of the linear background in the case of peaks of transition metals (e.g., Fe 2p) leads to significant systematic errors in estimating the peak areas.



Key

X binding energy (eV)

Y intensity (arbitrary units)

Figure 1 — Example of linear background and its subtraction
(The XPS spectrum used here is copper $2p_{3/2}$. The upper curve shows the measured spectrum and the linear background. The lower curve is the spectrum after subtraction of the background.)

6.2.3 Integral background removal

This (widely used) method, proposed by Shirley [12, 13], employs a mathematical algorithm to approximate the inelastic scattering of electrons as they escape from the solid. The algorithm is based on the assumption that the background is proportional to the area of the peak above the background at higher kinetic energies. This method has been modified to optimize the required iterations [14], to provide for a sloping inelastic background [15], to provide for a background based upon the shape of the loss spectrum from an elastically backscattered electron [16], and to include a band gap for insulators [2]. Figure 2 shows the Shirley or integral background for the Cu $2p_{3/2}$ XPS spectrum given in Figure 1 [11] and the spectrum after this background has been subtracted. It should be emphasized that the correct use of this method requires the application of the valid algorithm [12] and the proper iteration limit [14].

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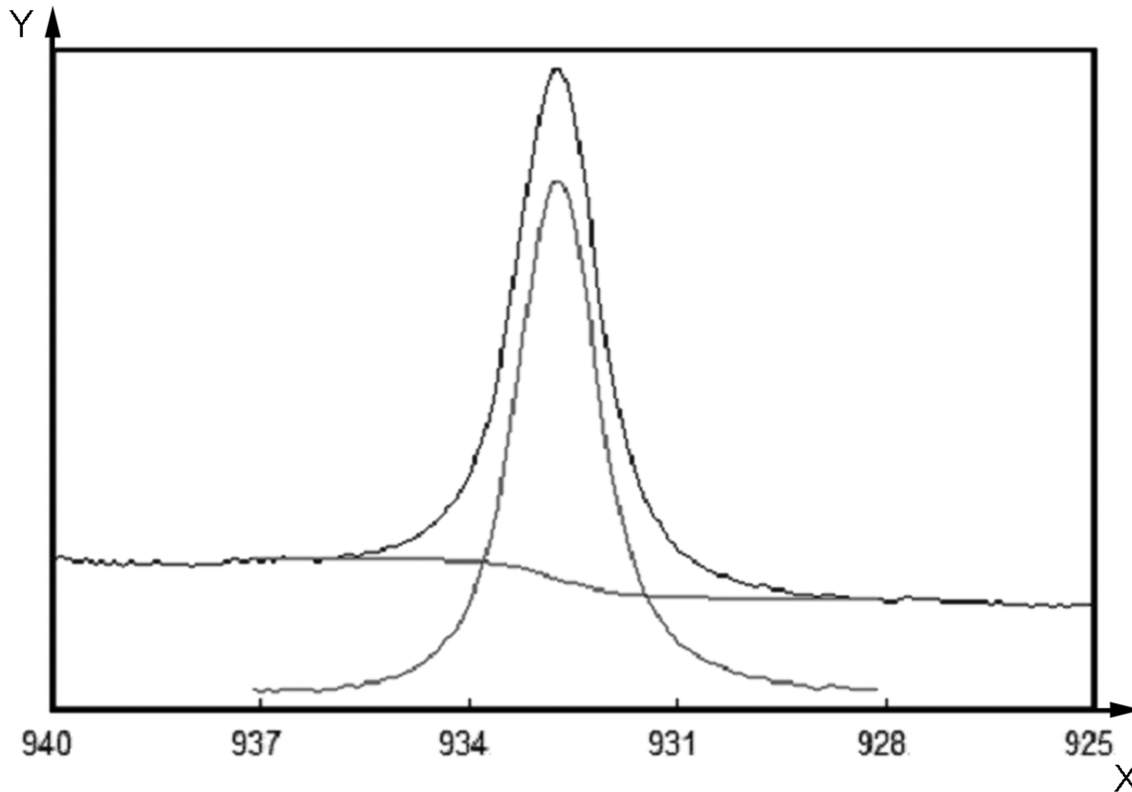

Key

X binding energy (eV)

Y intensity (arbitrary units)

Figure 2 — Example of integral background and its subtraction

(The XPS spectrum used is copper $2p_{3/2}$. The upper curve shows the measured spectrum and the integral background. The lower curve is the spectrum after subtraction of the background.)

6.2.4 Removal based on the electron inelastic-scattering cross-section

This (often used) method, proposed by Tougaard [4], uses an algorithm that is based on a description of the inelastic-scattering processes taking place in the sample. The algorithm requires knowledge of the inelastic-scattering cross-section for the signal electrons in the sample material. This cross-section can be obtained from analysis of experimental reflected electron energy loss spectra [4, 17] or from a simple approximate formula, the so-called universal cross-section [4]. While the universal cross-section can be applied successfully in the case of many solids, more accurate simple formulas are available as well [18] for particular classes of material (e.g. polymers, Si, Al and other solids). Alternatively, the parameters used in the universal cross-section formula may be varied in an algorithm for estimating the inelastic background [19]. Figure 3 provides an example of the Tougaard background for Au 4p, 4d spectra and of these spectra after this background was subtracted [20].

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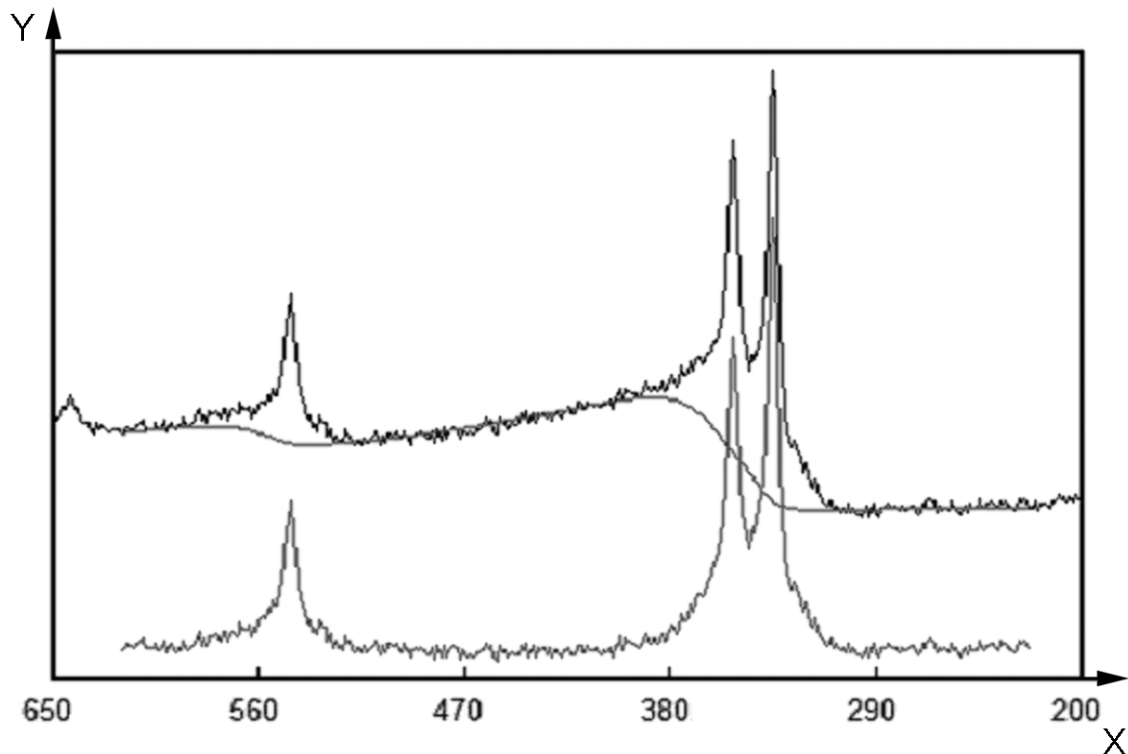

Key

X binding energy (eV)

Y intensity (arbitrary units)

Figure 3 — Example of the Tougaard background and its subtraction
(The XPS spectrum used shows the gold 4p and 4d peaks. The upper curve shows the measured spectrum and the background obtained from use of the inelastic scattering cross-section. The lower curve is the spectrum after subtraction of the background.)

6.3 Procedures accounting for both inelastic and elastic scattering

It is the inelastic scattering of photoelectrons that produces the background. However, elastic scattering of the signal electrons affects their trajectories and therefore has an indirect effect on the background, which can be significant. For the case of homogeneous solids, this effect has been described analytically [21].

A useful method for modelling scattering effects in photo-induced electron spectra is to express the spectral shape in terms of the partial intensities of electrons that have participated in a given number of inelastic collisions [22]. This approach, known as partial intensity analysis (PIA), is based on the assumption that elastic deflections and energy losses of the scattered electrons are independent and can be considered separately. It is not necessary for the transmission function of the analyser to be known.

A further, although less frequently applied (because of its higher computational needs and limited practical use), method to account for scattering effects is the Monte Carlo simulation of the photo-induced electron spectra using various models and data sets for the material-dependent energy-loss functions, inelastic mean free paths, and differential cross-sections for elastic scattering [23].

6.4 Less commonly used procedures

Deconvoluting the photo-induced electron spectrum with the spectral shape of inelastically scattered electrons that typically have the same initial energy as the main peak in the photo-induced spectrum and its associated loss spectrum may also be used for background removal [24, 25] although an overcompensation of surface-loss

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features is possible in this case. The intensity of the loss spectrum relative to that of the elastic peak is sometimes optimized, to allow for the angular dependence of the elastic-scattering cross-section in reflection electron energy loss spectroscopy (REELS) [26, 27]. Deconvolution is usually accomplished using Fourier transforms or iterative techniques [24, 25].

For removing the background formed by the low-energy secondary-electron cascade, the method proposed by Sickafus [28, 29] can be applied in which the logarithm of the measured spectral intensity is plotted as a function of the logarithm of the electron energy. Alternatively, a smoothing spline algorithm [30] can be used to obtain a structureless background between two chosen spectral regions, and this background can then be subtracted from the measured spectrum at intermediate energies.

6.5 Role of surface and core-hole effects in background determination

Surface excitations can significantly influence the shape of an energy loss spectrum and, as a consequence, the background. Methods for background determination that make use of inelastic-scattering cross-sections derived from experimental REELS spectra account for surface effects to some extent; it should be noted, however, that the magnitude of these effects strongly depends on the electron emission angle at the sample surface and the electron energy. The effect of surface excitations can essentially be accounted for even in the case of use of the universal inelastic-scattering cross-section by varying an appropriate parameter in the formula in a well-defined way [31]. Influence of surface roughness can also be significant, especially in the case of analysis at a near-grazing emission angle and of analysis with standards having a surface roughness different from that of the sample analysed.

The core hole created in the photoionization process can lead to the appearance of loss structures in the spectra induced by intrinsic excitation, and these loss structures can depend on the chemical environment of the ionized atom. While the methods for background subtraction that are based on the application of the inelastic electron-scattering cross-section model remove only the extrinsic part of the background, a Shirley-type background removes most contributions of both intrinsic and extrinsic origin. The contribution from intrinsic processes can be estimated by using the shape parameter (obtained by extrapolating the background to the peak centre to obtain a “background height” normalized by dividing by the peak area) as suggested by Castle and Salvi [32, 33]. The shape parameter is expected to be characteristic of a given element or its particular chemical form [32, 33].

6.6 Determining the background for inhomogeneous materials

For the case of inhomogeneous materials, it has been shown [4] that appreciably differing atomic concentration distributions (i.e. in-depth profiles) can result in identical photo-induced peak intensities for a particular XPS experimental configuration. Formulae to determine the inelastic background for inhomogeneous systems have been developed by Tougaard [34-36]. He showed that analysis of the inelastic spectral shape accompanying the photoelectron or Auger-electron peak for an element can yield the concentration distribution of that element in the sample, i.e. the in-depth concentration profile and the surface morphology on the nanometre scale [22, 37-45]. A non-destructive procedure for quantitative characterization of surface nanostructures is based on a formalism using an adjustable function describing the in-depth atomic concentration profile and the Tougaard universal inelastic-scattering cross-section; use may also be made of the reference spectrum for a standard sample with a known concentration profile such as a single elemental solid. The procedure is applicable either for obtaining the intrinsic atomic excitation spectrum (background removal) by adjusting the profile function or for determining the compositional profile (using a known atomic spectrum from analysis of a reference spectrum). The accuracy of the technique and other details are available in a review [4], and a software package known as QUASES™ (Quantitative Analysis of Surfaces by Electron Spectroscopy) [4] is available for spectrum analysis. Recent studies of the growth and in-depth compositional distribution of thin metal films on silicon from analyses of the inelastic spectral shape accompanying XPS lines have demonstrated the utility of this software [43, 44].

The PIA approach [22, 45] described in 6.3 can also be used to determine backgrounds in photo-induced electron spectra of inhomogeneous materials. In this formalism, a function that depends on the partial intensities of normalized energy distributions after a specified number of inelastic collisions is minimized. An XPS peak for the selected species is often measured at different emission angles when applying this procedure.

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7 Comparisons of procedures for removing effects of inelastic electron scattering from electron spectra

Comparisons have been published of the most popular procedures for background removal [14, 15, 46-59]. The effect of the choice of the energy end points/regions on determinations of the peak area has been discussed for the cases of the linear background (6.2.2) and the integral background (6.2.3) [14, 15]. Further comparisons of these procedures and of the Tougaard procedure (6.2.4), including tests of their validity, have been published [46-59]. The general behaviour of Tougaard-style backgrounds in XPS, including effects of different cross-sections for inelastic scattering, has been analysed [27, 60]. A comprehensive review of electron transport in solids relevant to AES and XPS and of the influence of this electron transport on quantitative surface analysis has been published [61]. This review contains a detailed discussion and comparison of different methods for removing inelastic backgrounds and other spectral contributions from electron spectra.

Table 1 shows root-mean-square scatter factors for the ratios of measured XPS intensities for some 60 elemental solids to the corresponding calculated intensities with respect to the average value of this ratio for excitation by Al and Mg Kα X-rays and for inelastic backgrounds determined from the Tougaard universal cross-section and from angle-averaged REELS cross-sections [27]. This analysis shows that the scatter factors are smaller (i.e. there is closer agreement between measured and calculated XPS intensities) if inelastic backgrounds are determined from REELS cross-sections instead of the Tougaard universal cross-section. The REELS cross-sections used in the analysis have not yet been published.

Table 1 — Root-mean-square scatter factors for the ratios of measured XPS intensities for some 60 elemental solids to the corresponding calculated intensities with respect to the average value of this ratio for excitation by Al and Mg Kα X-rays and for inelastic backgrounds determined from the Tougaard universal cross-section and from angle-averaged REELS cross-sections [60]

Cross-section for inelastic scattering used to determine backgrounds	Exciting X-rays	
	Al Kα	Mg Kα
Universal	1,20	1,20
REELS (angle-averaged)	1,11	1,12

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