Summary of ISO/TC 201 Standard: XX
ISO 18118: 2004 — Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy —Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials

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ISO 18118 provides guidance on the measurement and use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials by Auger electron spectroscopy and X-ray photoelectron spectroscopy. This article provides a brief summary of this International Standard.

KEYWORDS: AES; Auger electron spectroscopy; ISO; International Organization for Standardization; relative sensitivity factor; quantitative surface analysis; XPS; X-ray photoelectron spectroscopy.

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It is convenient in many quantitative applications of Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) to utilise relative sensitivity factors (RSFs) for quantitative analyses. Three types of RSF have been used for this purpose: elemental relative sensitivity factors (ERSFs), atomic relative sensitivity factors (ARSFs), and average matrix relative sensitivity factors (AMRSFs). While the ERSFs are the simplest and easiest to apply, they are the least accurate because no account is taken of matrix correction factors. The matrix correction factors for AES can vary between 0.1 and 8 [1] while for XPS they can vary between 0.3 and 3 [2]. The ARSFs are more accurate than ERSFs in that they take account of differences of atomic densities, generally the largest single matrix correction. The AMRSFs are the most reliable RSFs in that there is almost complete correction of matrix effects. This International Standard provides a guide to the use of these different RSFs and a

thorough explanation of their derivations, calculation and differences. It is recommended that ERSFs be used only for semi-quantitative analyses and that ARSFs or preferably AMRSFs be used for quantitative analyses. For the latter applications, ARSFs shall be used only in situations for which it is not possible to make use of AMRSFs.

Different measures of signal intensities are used in AES and XPS, and it is necessary that the same measure be used for the RSF determination as for the analytical application. For some applications of AES, it is convenient to use peak-to-peak heights of Auger-electron signals in the differential mode as measures of Auger-electron intensities. For other applications of AES (e.g., scanning Auger microscopy), the Auger-electron intensity may be determined from the difference of the intensity at a peak maximum in the direct spectrum and the intensity of a nearby background signal. Finally, for many applications in XPS and for some applications of AES, areas of peaks in direct spectra are used as measures of photoelectron or Auger-electron intensities.

In analytical applications of AES and XPS, it is essential that Auger-electron and photoelectron intensities be measured using exactly the same procedure as that used for measurement of the RSFs. RSFs depend on parameters of the excitation source (for example, the incident electron energy in AES and the choice of X-ray energy in XPS), the spectrometer (for example, the angle of incidence of the electron beam in AES, the angle between the X-ray source and the analyser axis in XPS, the sample area viewed by the analyser, and the acceptance solid angle of the analyser), and the orientation of the sample to these parts of the instrument [3]. The sample area viewed by the analyser and the analyser acceptance solid angle can depend on analyser settings (for example, selection of apertures, whether the analyser is operated in the constant analyser energy mode or the constant retardation ratio mode, and the corresponding choices of analyser pass energy or retardation ratio). Finally, the measured Auger-electron or photoelectron intensities can depend on other instrumental parameters (e.g., energy step, scan rate, gain and time constant (for AES instruments with analogue detection systems), and modulation to generate a derivative spectrum) and whether the instrument is operated under conditions so that the detected signal intensities are sufficiently linearly dependent on the excitation intensity. It is therefore essential that Auger-electron and photoelectron intensities be determined using exactly the same instrumental settings and the same sample orientation as those employed for the RSF measurements. It is also essential that the same data-analysis procedures be used in measurements of signal-electron intensities for the unknown sample as those used in the RSF measurements.

Commercial AES and XPS instruments are generally supplied with a set of RSFs for one or more common operating conditions. These RSFs were typically determined on an instrument of the same type or, in some cases, on similar instruments. In some cases these RSFs are ERSFs or ARSFs and in other cases they are closer to AMRSFs. In some cases they may be a combination of all three types of RSF. It is recommended that an analyst check the RSFs supplied with the instrument for those elements expected to be of analytical interest to ensure that the supplied RSFs are correct and are of the relevant type for the application. In addition,

the intensity-energy response function (IERF) of the instrument may change with time. Alternatively, an analyst can check for possible changes in IERF with time by measuring selected ERSFs.

Many factors contribute to the uncertainty in a determination of surface composition from AES or XPS measurements with the use of RSFs [4]. It is not generally possible to determine or estimate standard uncertainties for many of these factors, partly because definitive experiments have not yet been conducted to establish uncertainties for some matrix-effect parameters (for example, values of electron inelastic mean free paths, elastic-scattering correction factors, and backscattering factors). In addition, practical samples often are not chemically homogenous over the analytical volume and their surfaces are not atomically flat, as assumed in the development of equations for RSFs; the analytical uncertainty will thus depend on the extent to which a particular sample deviates from the idealized structure. Finally, other simplifying assumptions (for example, the neglect of matrix effects on spectral line shapes in some types of intensity measurements or the neglect of radiation damage, ion-sputtering effects, and surface contamination) lead to uncertainties whose magnitudes again depend on the particular sample.

CURRENT LIST OF STANDARDS FROM ISO/TC 201 (A summary is available in *Surface and Interface Analysis* in the volume given in square brackets).

(I) ISO 14976:1998 – *Surface chemical analysis – Data transfer format* [*SIA*1999; **27**: 693].
(II) ISO 14237:2000 – *Surface chemical analysis – Secondary ion mass spectrometry – Determination of boron atomic concentration in silicon using uniformly doped materials* [*SIA* 2002; **33**: 361].
(III) ISO 14707:2000 – *Surface chemical analysis - Glow discharge optical emission spectrometry – Introduction to use* [*SIA*2002; **33**: 363].
(IV) ISO 14606:2000 – *Surface chemical analysis – Sputter depth profiling – Optimisation using layered systems as reference materials*. [*SIA* 2002; **33**: 365].
(V) ISO 14975:2000 – *Surface chemical analysis – Information formats* [*SIA* 2002; **33**: 367].
(VI) ISO 14706:2000 – *Surface chemical analysis – Test method of surface elemental contamination on silicon wafers by total reflection X-ray fluorescence spectrometry* [*SIA* 2002; **33**: 369].
(VII) ISO 15472:2001 – *Surface chemical analysis – X-ray photoelectron spectrometers – Calibration of energy scales* [*SIA* 2001; **31**: 721].
(VIII) ISO 18115:2001 – *Surface chemical analysis – Vocabulary* [*SIA* 2001; **31**: 1048].
(IX) ISO TR 15969:2000 – *Surface chemical analysis – Depth profiling – Measurement of sputtered depth* [*SIA* 2002; **33**: 453].
(X) ISO 17560:2002 – *Surface chemical analysis – Secondary-ion mass spectrometry – Method for depth profiling of boron in silicon* [*SIA* 2005; **37**: 90].

(XI) ISO 17974:2002 – *Surface chemical analysis – High resolution Auger electron spectrometers – Calibration of energy scales for elemental and chemical state analysis* [*SIA* 2003; **35**: 327].
(XII) ISO 17973:2002 – *Surface chemical analysis – Medium resolution Auger electron spectrometers – Calibration of energy scales for elemental analysis* [*SIA* 2003; **35**: 329].
(XIII) ISO 18114:2003 – *Surface chemical analysis – Secondary-ion mass spectrometry – Determination of relative sensitivity factors from ion-implanted reference materials*.
(XIV) ISO TR 19319:2003 – *Surface chemical analysis – Auger electron spectroscopy and X-ray photoelectron spectroscopy – Determination of lateral resolution, analysis area and sample area viewed by the analyser* [*SIA* 2004; **36**: 666].
(XV) ISO 20341:2003 – *Surface chemical analysis – Secondary ion mass spectrometry – Method for estimating depth resolution parameters with multiple delta-layer reference materials* [*SIA* 2005; **37**: 646].
(XVI) ISO 15470:2004 – *Surface chemical analysis – X-ray photoelectron spectroscopy – Description of selected instrumental performance parameters*
(XVII) ISO 15471:2004 – *Surface chemical analysis – Auger electron spectroscopy – Description of selected instrumental performance parameters*
(XVIII) ISO 19318:2004 – *Surface chemical analysis – X-ray photoelectron spectroscopy – Reporting of methods used for charge control and charge correction* [*SIA*. 2005; **37**: 524].
(XIX) ISO 17331:2004 – *Surface chemical analysis – Chemical methods for the collection of elements from the surface of silicon-wafer working reference materials and their determination by total-reflection X-ray fluorescence (TXRF) spectroscopy* [*SIA* 2005; **37**: 522].
(XX) ISO 18118:2004 – *Surface chemical analysis – Auger electron spectroscopy and X-ray photoelectron spectroscopy – Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials*
(XXI) ISO 21270:2004 – *Surface chemical analysis – X-ray photoelectron and Auger electron spectrometers – Linearity of intensity scale* [*SIA* 2004; **36**: 1645].
(XXII) ISO 22048:2004 – *Surface chemical analysis – Information format for static secondary-ion mass spectrometry* [*SIA* 2004; **36**: 1642].

ISO standards may be purchased from national standards bodies, directly from the ISO Central Secretariat, Case Postale 56, CH-1211 Geneva 20, Switzerland, or through the internet at http://www.iso.ch. More information about ISO/TC 201 on Surface Chemical Analysis may be obtained from this internet site or from Mr Yukio Hirose, Secretariat of ISO/TC 201, Japanese Standards Association, Toraya Bldg 7F, 4-9-22 Akasaka, Minato-ku, Tokyo 107-0052, Japan.

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