



Summary of ISO/TC 201 International Standard ISO 18516:2019 Surface chemical analysis—Determination of lateral resolution and sharpness in beam-based methods with a range from nanometres to micrometres and its implementation for imaging laboratory X-ray photoelectron spectrometers (XPS)

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ISO 18516:2019 *Surface chemical analysis—Determination of lateral resolution and sharpness in beam-based methods with a range from nanometres to micrometres* revises ISO 18516:2006 *Surface chemical analysis—Auger electron spectroscopy and X-ray photoelectron spectroscopy—Determination of lateral resolution*. It implements three different methods delivering parameters useful to express the lateral resolution: (1) the straight edge method, (2) the narrow line method and (3) the grating method. The theoretical background of these methods is introduced in ISO/TR 19319:2013 *Surface chemical analysis—Fundamental approaches to determination of lateral resolution and sharpness in beam-based methods*. The revised International Standard ISO 18516 delivers standardized procedures for the determination of the (1) effective lateral resolution by imaging of square-wave gratings, the (2) lateral resolution expressed as the parameter D_{12-88} characterizing the steepness of the sigmoidal edge spread function (ESF) determined by imaging a straight edge and (3) the lateral resolution expressed as the full width of half maximum of the line spread function (LSF), w_{LSF} , determined by imaging a narrow line. The last method also delivers information on the shape of the LSF, which characterizes an individual imaging instrument. Finally, the implementation of all three standardized methods in the field of imaging laboratory X-ray photoelectron spectroscopy (XPS) is shortly presented. This part of the letter is based on the use of a new test sample developed at ETH Zurich, Switzerland. This test sample displays a micrometre scaled pattern motivated by the resolving power of recent imaging XPS instruments.

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KEYWORDS

grating method, imaging AES, imaging SIMS, imaging XPS, lateral resolution, narrow line method, noise in image, resolution criterion, straight edge method

1 | INTRODUCTION

1.1 | History of ISO 18516:2019 and contributions by Martin Seah

Martin Seah as a delegate of BSI, the UK national standards body, was a co-founder of the ISO Technical Committee TC 201 on Surface Chemical Analysis in 1991 (for a survey of Martin's activity in this committee, see reference¹). In the subsequent years, Martin was the project leader of many standardization projects and a welcomed consultant for other project leaders of ISO/TC 201. To name only one of his highly important standardization projects, we refer to the two terminology standards: ISO 18115-1:2013 *Surface Chemical Analysis—Vocabulary—Part 1: General terms and terms used in spectroscopy* and ISO 18115-2:2013 *Surface Chemical Analysis—Vocabulary—Part 2: Terms used in scanning-probe microscopy* (available on the ISO.org web site at no charge). Martin was also rather helpful to develop the infrastructure of ISO/TC 201 in many directions. For example, the template we were using here to write this 'Summary of ISO ...' letter for *Surface and Interface Analysis* was designed by him.

The history behind the development of ISO 18516:2019 points back to the early 2000 years. At that time, the interest in imaging surface chemical analysis was substantially increasing because more and more commercial instruments for surface chemical analysis were offering imaging modes. This led to a need to determine the lateral resolution in images delivered by such instruments in a comparable way. ISO/TC 201 took that challenge and launched a standardization project considering the state of the art at this time. This was represented by the application of the so-called straight edge method executed with appropriate test samples. The first achievement was the Technical Report 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* (cf. reference²), result of a standardization project coordinated by ANSI, the American National Standards Institute, with Cedric Powell as the project leader. Subsequently, the ISO international standard ISO 18516:2006—*Surface chemical analysis—Auger electron spectroscopy and X-ray photoelectron spectroscopy—Determination of lateral resolution* was published (cf. reference³). The project was coordinated by BSI with John Wolstenholme as the project leader and Martin delivered substantial input. Some years later, BAM, the German Federal Institute for Materials Science and Testing, developed a new test sample for a measurement of lateral resolution, BAM L200.⁴ This test sample provides a pattern enabling the application of known and new approaches for a comparable measurement of lateral resolution (see below) down to the deep nanometre scale. Subsequently, delegates of DIN, the German national standards body, presented these new

measurement capabilities to ISO/TC 201. At the Budapest annual meeting of ISO/TC 201 in 2005, after previous consultation with Cedric Powell and Martin Seah, an update for ISO/TR 19319:203 was proposed and positively decided. Both consultants supported the project by very fruitful discussions. The intention of the new ISO/TR 19319 published in 2013 was to describe theoretical concepts of image formation and lateral resolution in a detailed manner, also addressing the issue of noise in the images obtained.⁵ New and well-established practical approaches to deliver valid data for lateral resolution were introduced as well. Subsequently, a new project, the revision of ISO 18516:2006 by considering knowledge presented in ISO/TR 19319:2013, was launched with the leadership at DIN. The most important comments were submitted by BSI and JISC, the Japanese Industrial Standards Committee. In that process, Martin used the BSI channel and personal communication by email for fruitful discussions with the BAM scientists. He as the project leader behind the terminology standard ISO 18115-1:2013 was rather interested in the issue of a definition of lateral resolution for the different approaches standardized in the new release of ISO 18516. Finally, we agreed on a method-specific wording like 'lateral resolution expressed as <method specific measurand>' for each of all three methods of measurements addressed in the standard and introduced as summaries later in this letter.

1.2 | Introduction to ISO 18516:2019

Secondary ion mass spectrometry (SIMS), Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) are techniques of surface chemical analysis that are used to generate chemical maps of surfaces, that is, images, and line scans across chemically heterogeneous surfaces. It is of interest for an analyst to determine and control the lateral resolution as a quality parameter of the images or line scans acquired at specified instrument settings. ISO 18516:2019 is based on ISO/TR 19319:2013,⁶ which delivers theoretical backgrounds of image formation and the determination of lateral resolution used to express the performance of beam-based imaging instruments used in surface chemical analysis.

ISO 18516:2019 standardizes different methods for the determination of lateral resolution in beam-based methods as AES, SIMS and XPS. These are (a) the straight edge method, already addressed by ISO 18516:2006; (b) the narrow line method, which was mentioned in ISO 18516:2006 as an alternative method for a determination of lateral resolution but not elaborated in detail; and (c) the grating method.

The standard addresses the needs of instrument manufacturers, for example, for instrument specification or benchmarking activities

and analysts in testing laboratories for their day-to-day control of instrument performance to match the needs of good laboratory practice (GLP) or the requirements of an accreditation under ISO 17025 or other schemes of accreditation.

There are annexes providing forthcoming information on how to find appropriate measurement parameters, considerations of the uncertainty of measurement and a practical example of an implementation for imaging SIMS using the BAM L200 test sample.

2 | SUMMARY OF ISO 18516:2019

A common need in imaging surface chemical analysis by methods such as SIMS, AES and XPS is the measurement of composition as a function of position on the sample surface. Typically, an analyst wishes to determine the local surface composition of some identified region of interest. Features at the heterogeneous surface of a sample with micrometre and nanometre dimensions must be addressed, and, therefore, the lateral resolution of the technique must be smaller than the lateral dimensions of the feature in order that the feature can be readily imaged. The feature of interest must generally be detected from an image or a line scan in which a particular signal (the intensity of a selected secondary ion, a photoelectron peak or an Auger electron) is displayed as a function of position on the sample surface. In practice, the detectability of a feature in SIMS, AES and XPS measurements depends not only on the lateral resolution but also on the difference in signal intensities for measurements made on and off the possible feature (material contrast) and the observation time (through the noise in the signal intensities; cf. reference⁵). The distinction and separation of features thus depends on an instrumental setting enabling the required lateral resolution, the particular constituents of the sample and the measurement time needed to reach a required (low) noise level.

The methods standardized in ISO 18516:2019 are designed for a determination of specific parameters that characterize the lateral resolution of an imaging instrument at specific settings. They use images or line scans obtained from test samples displaying well-defined patterns. The standardized methods are as follows:

1. the straight edge method;
2. the narrow line method; and
3. the grating method.

Principally, all methods are suitable for any resolution from micrometre down to nanometre scales, work for all beam-based instruments used for imaging surface chemical analysis and are simple enough to be used in a ‘quick and easy’ manner for daily performance tests. Data evaluation is made by visual real-time inspection of an image on a screen or simple graphical analysis of a line scan by only using pencil and ruler. Furthermore, the same methods can be executed in a sophisticated manner that delivers accurate and objective values for resolution parameters by using numerical data evaluation as it is often necessary for a binding instrument specification,

benchmarking of instruments or a documented validation of instrument performance in an accredited testing laboratory.

The well-established straight edge method is a simple method suitable for the characterization of lateral resolution expressed by a single parameter. Executing the method, a line scan across or an image of a straight edge measured with high signal-to-noise ratio and appropriate sampling step width is acquired using a planar test sample that displays a sharp and straight chemical edge between Material A and Material B and appropriately long plateau lengths left and right from the edge. The method delivers lateral resolution expressed as the parameter D_{12-88} , which characterizes the steepness of the (sigmoidal) edge spread function (ESF). The method is well suited for daily in-house performance tests. However, as a single parameter, D_{12-88} cannot fully characterize the sigmoidal ESF as outlined in ISO/TR 19319:2013⁶ and references therein. Basically, the ESF is determined by the shape of the profile of the probe beam (e.g., Gaussian, Lorentzian and mixed shape) used for imaging. Therefore, the character of D_{12-88} remains somewhat ambiguous. This ambiguity is illustrated by a simulation displayed in fig. A1 in Annex A of the standard ISO 18516:2019.

The narrow line method is a simple method suitable for the characterization of lateral resolution. A line scan across or an image of a narrow stripe measured with high signal-to-noise ratio and appropriate sampling step width must be acquired using a planar test sample that displays a narrow line of Material A in B where the line width must be sufficiently low in comparison with the expected lateral resolution. As it is recommended in Annex E of the standard, the expected lateral resolution must be three to five times the width of the narrow line. The method delivers lateral resolution expressed as the parameter w_{LSF} , the full width at half maximum (FWHM) of the line spread function (LSF). This easy method is well suited for daily in-house performance tests, too. Again, as a single parameter, w_{LSF} cannot fully characterize the LSF. Basically, the LSF is determined by shape and FWHM of the profile (e.g., Gaussian, Lorentzian and mixed shape) of the probe beam used for imaging. However, because a determination of the parameter w_{LSF} requires a measurement of the full LSF profile, the narrow line method may, after careful numerical analysis, deliver also information on its shape.

The third standardized method, the grating method, enables the determination of the lateral resolution expressed as the minimum spacing at which two features of an image can be recognized as distinct and separate. To run the grating method, a line scan across or image of a series of A–B–A square-wave gratings of Material A in B measured with high signal-to-noise ratio and appropriate sampling step width (for details, see Annex I of ISO 18516:2019) must be acquired using a planar test sample that displays a series of gratings narrower and broader than the expected resolution. The method delivers the effective lateral resolution r_e , estimated by determining the period P of the finest resolved grating. Hence, r_e is the minimum spacing at which two features of the image can be recognized. Using the ‘visual inspection mode’ of the grating method, it is also suited for regular in-house performance tests. The advantage of the grating method is that it is linked to a widely accepted and overarching definition of lateral resolution in microscopy.

Finally, Annexes D, F, J and K of the standard cover considerations of the uncertainty of measurement related to the results of all three methods introduced for a determination of parameters that characterize the lateral resolution.

3 | IMPLEMENTATION FOR IMAGING LABORATORY XPS

3.1 | Introduction

The layout of the BAM L200 test sample covers a straight edge, several narrow lines and a wide series of A-B-A gratings of Material A in B.⁴ The dimensions of the narrow lines and the A-B-A gratings are all in the submicrometre range. Most applications of BAM L200 are seen for the use of the grating method for a determination of lateral resolution in nanoscale imaging by SIMS (cf., e.g., references^{4,7-15}) and AES (cf., e.g., references^{4,16-19}). For XPS, there are only some implementations of the grating method using NanoESCA instruments (Omicron Nanotechnology) working as experimental end stations at synchrotron radiation sources.^{20,21} With that XPS set-up, submicrometre lateral resolution can be achieved.

3.2 | Test sample

Recently, a test sample made by using an innovative technology²² and displaying a layout (see Figure 1) inspired by the pattern on BAM L200 was developed by colleagues at ETH Zurich, Switzerland.^{22,23} The Ti pattern in Au is planar on the deep nanometre scale. Edge effects will be negligible for XPS instruments. This test sample was designed to enable a determination of the lateral resolution for recent imaging XPS laboratory instruments.²³ Typical lateral resolutions achieved by those instruments are on the deep micrometre scale, that is, somewhat below 10 μm . A short introduction on how to implement the different methods for a determination of lateral resolution of a Kratos Analytical AXIS Nova imaging XPS instrument installed at the Institut des Matériaux Jean Rouxel, Nantes, France, is presented in the following paragraphs. Note that always the Au 4f signal was used

for imaging because of a higher photoionization cross section in comparison with Ti 2p.

3.3 | The straight edge method

The ETHZ test sample offers a $200 \times 500\text{-}\mu\text{m}^2$ Ti box to execute the straight edge method. ISO 18516:2019, clause 6.7, demands that the

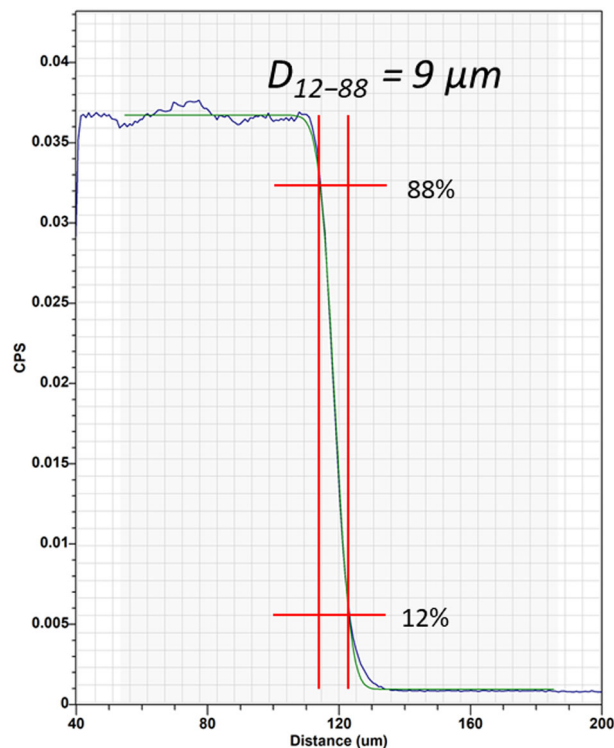


FIGURE 2 Au 4f intensity profile across a straight edge between Au and Ti derived from an image taken by an AXIS Nova XPS instrument. The $200 \times 500\text{-}\mu\text{m}^2$ Ti box in Au displayed in the layout of the test sample in Figure 1 was used to provide a straight edge. Green line: Fitted edge spread function (EFS). Detailed information on instrument settings, image treatments and analysis by using different CasaXPS software components²⁴ are given in the supporting information. Analysis of straight edge image data by CasaXPS is also explained in a video: <https://www.youtube.com/watch?v=F0qnM55kbco&t=0s>

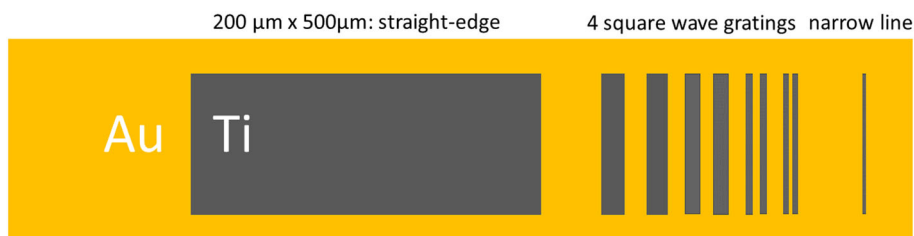


FIGURE 1 Principal layout of the ETHZ sample developed for testing the measurement capabilities in the imaging mode achieved by recent XPS laboratory instruments (cf. references^{22,23}). The pattern comprises (a) a Ti box in Au for executing the straight edge method; (b) a series of consecutive square-wave A-B-A gratings (A = Ti, B = Au) with bar widths of 30, 20, 10 and 5 μm , that is, the respective grating periods ('pitches') $P = 60, 40, 20$ and 10 μm (cf. ISO 18516:2019, clause 8.2), to run the grating method; and (c) a narrow Ti line of 2- μm width in Au to execute the narrow line method

length range of measured values of the ESF on both sides of the straight edge, that is, the plateau length, must be large enough to enable an accurate determination of the 0% and 100% levels of signal intensity used for the calculation of the ESF parameter D_{12-88} . Recommendations for the plateau length ranges are given in Annex C of the standard. In brief, at an appropriate signal-to-noise-ratio, the plateau length must be ≥ 4 times the expected value of D_{12-88} for a Gaussian LSF and ≥ 10 times D_{12-88} for a Lorentzian LSF. The given maximum plateau length of 500 μm , that is, the length of the Ti box, nicely complies to the required plateau length in the context of expected D_{12-88} values. Looking at the experimental data, the Au 4f profile displayed in Figure 2, the measured parameter D_{12-88} of 9 μm , expresses the lateral resolution of the AXIS Nova instrument used to image the straight edge.

3.4 | The narrow line method

The narrow line method was executed using the 2- μm narrow Ti line on the ETHZ test sample. As ISO 18516:2019, clause 7.2 informs the user that a line on a test sample is a ‘narrow line’ if its width is small compared with the FWHM of the LSF, w_{LSF} . Then a line scan or a profile across the image of the narrow line reveals the LSF. For that reason, the recommended line width on the test sample is $0.2 w_{\text{LSF}} \leq$ line width $\leq 0.5 w_{\text{LSF}}$. Annex E in ISO 18516:2019 delivers detailed

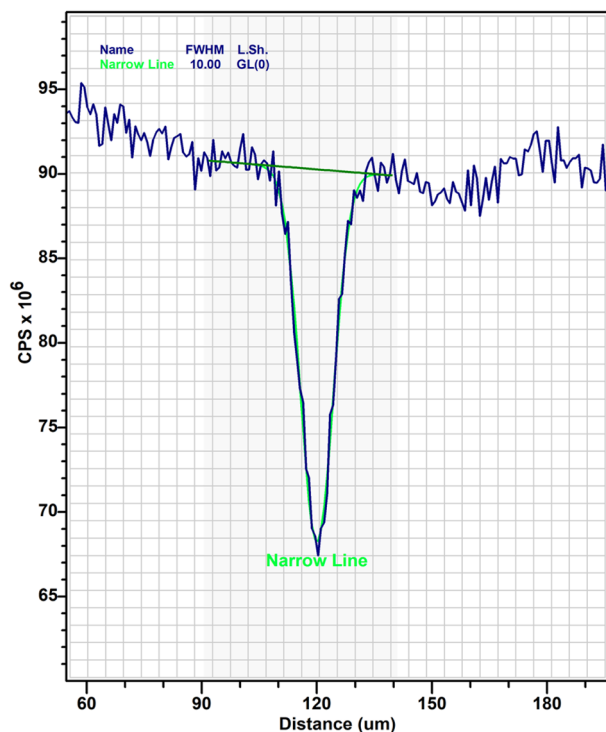


FIGURE 3 Au 4f intensity profile across the Ti narrow line in Au derived from an image of the ETHZ test sample (cf. Figure 1) taken by an AXIS Nova XPS instrument used in the image mode. Green line: Fitted line spread function (LSF). Detailed information on instrument settings and image treatment by using different CasaXPS software components²⁴ are given in the supporting information. FWHM, full width at half maximum

information on how to avoid systematic overestimation of w_{LSF} caused by an inappropriately large width of the imaged narrow line. In the given experiment, where we expect a w_{LSF} of around 10 μm , the 2- μm width of the Ti narrow line complies to the recommendation in ISO 18516:2019. The analysis of the image reveals a w_{LSF} of 10 μm , which expresses the lateral resolution of the AXIS Nova instrument used to image the narrow line (see Figure 3). Furthermore, numerical analysis of the respective profile in Figure 3 by fitting reveals a distinct Gaussian character of the shape of the LSF.

3.5 | The grating method

To run the grating method, an image of a series of consecutive square-wave gratings is taken and evaluated. In the easiest approach, the smallest resolved grating period is used to estimate the effective lateral resolution r_e as a ‘better as’ information. Because there is always noise in an image, a resolution criterion is set in ISO 18516:2019, clause 8.8.4, to decide whether the grating is resolved or not. The separation of an A–B–A grating in a line profile across the gratings must be visible as a dip (or little peak) between two maxima (or minima) of signal intensity. A dip between two maxima is seen when a signal related to Material A was used for imaging. Use of a signal related to Material B leads to minima and a little peak in between. The latter case applies to the profile displayed in Figure 4. The visibility of the dip D (or the

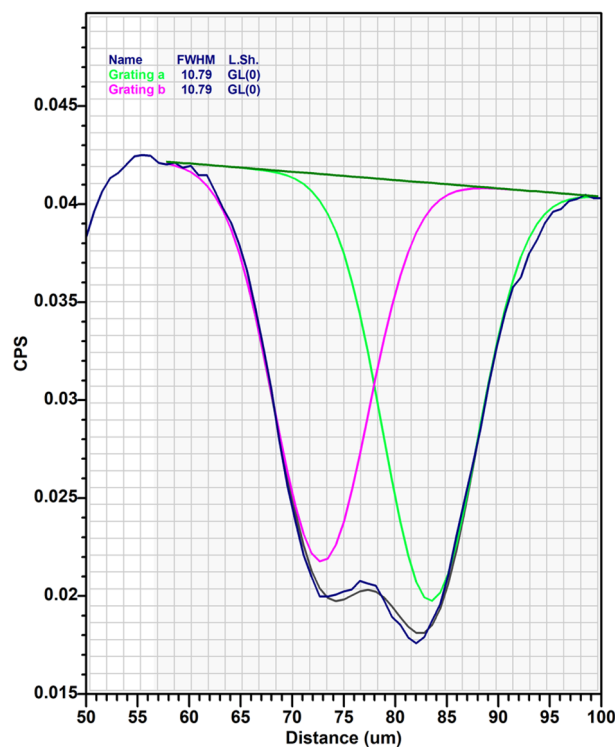


FIGURE 4 Au 4f intensity profile across the finest Ti grating in Au derived from an image of the ETHZ test sample (cf. Figure 1) taken by an AXIS Nova XPS instrument used in the image mode. Detailed information on instrument settings and image treatment by using different CasaXPS software components²⁴ are given in the supporting information. FWHM, full width at half maximum

corresponding peak) depends not only on the resolving power of the instrument but also on the noise in the image. A profile established by summing up image data as many parallel line scans helps to reduce the noise. ISO 18516:2019 defines the dip-to-reduced-noise ratio $R = D/\sigma_{Nr}$ where D is the depth of the dip and σ_{Nr} is the reduced noise to be calculated as defined in clause 8.8.2 of ISO 18516:2019. The agreed resolution criterion set in ISO 18516:2019 is $R > 4$. Looking at the Au 4f intensity line scan in Figure 4 established by summing up many line scans from the image of that grating on the ETHZ test sample with the smallest period P , 10 μm for a A–B–A grating with bar widths of 5 μm each, reveals that this grating is resolved, and the conclusion is that effective lateral resolution r_e is 10 μm or even a little bit better. It is the minimum spacing at which two features of an image can be recognized as distinct and separate by the imaging instrument used.

The technology used by ETHZ to manufacture the test samples allows to adapt the sizes of gratings and narrow lines on the pattern according to future improvements of the lateral resolution achieved with imaging XPS instruments.

4 | CURRENT LIST OF RELATED STANDARDS FROM ISO/TC 201

Standards and Technical Reports developed by ISO/TC 201 are listed below by topics covered. Summaries of many of these have been published in *Surface and Interface Analysis*, as indicated by the references in square brackets. Amendments (Amd) may modify an instruction in the standard or may, as in the Vocabularies, add many new terms. To simplify the texts, the following abbreviations have been used:

- AES—Auger electron spectroscopy.
- SCA—Surface chemical analysis.
- SIMS—Secondary ion mass spectrometry.
- XPS—X-ray photoelectron spectroscopy.

4.1 | General

ISO 18115-1:2013 SCA—Vocabulary—Part 1: General terms and terms used in spectroscopy [SIA 2014; 46: 357].

ISO 18115-2:2013 SCA—Vocabulary—Part 2: Terms used in scanning-probe microscopy [SIA 2014; 46: 361].

ISO 18116:2005 SCA—Guidelines for preparation and mounting of specimens for analysis.

ISO 18117:2009 SCA—Handling of specimens prior to analysis.

ISO 20579-3:2021 SCA—Sample handling, preparation and mounting—Part 3: Biomaterials.

ISO 20579-4:2018 SCA—Guidelines to sample handling, preparation and mounting—Part 4: Reporting information related to the history, preparation, handling and mounting of nano-objects prior to surface analysis [SIA 2018; 50: 902].

ISO/TR 14187:2020—SCA—Characterization of nanostructured materials [SIA 2012; 44: 1305].

ISO 14976:1998 SCA—Data transfer format [SIA 1999; 27: 693].

ISO 14975:2000 SCA—Information formats [SIA 2002; 33: 367].

ISO 22048:2004 SCA—Information format for static SIMS.

ISO/TR 19319:2013 SCA—Fundamental approaches to determination of lateral resolution and sharpness in beam-based methods [SIA 2013; 27: 1313].

4.2 | Auger and X-ray photoelectron spectroscopies

ISO 15471:2016 SCA—AES—Description of selected instrumental performance parameters.

ISO 15470:2017 SCA—XPS—Description of selected instrumental performance parameters [SIA 2008; 40: 966].

ISO 10810:2019 SCA—XPS—Guidelines for analysis.

ISO 19318:2021 SCA—XPS—Reporting of methods used for charge control and charge correction [SIA 2005; 37: 524].

ISO 16242:2011 SCA—Recording and reporting data in AES.

ISO 16243:2011 SCA—Recording and reporting data in XPS.

ISO 17973:2016 SCA—Medium-resolution Auger electron spectrometers—Calibration of energy scales [SIA 2003; 35: 329].

ISO 17974:2002 SCA—High-resolution Auger electron spectrometers—Calibration of energy scales for elemental and chemical-state analysis [SIA 2003; 35: 327].

ISO 15472:2010 SCA—XPS—Calibration of energy scales [SIA 2001; 31: 721].

ISO 20903:2019 SCA—AES and XPS—Methods used to determine peak intensities and information required when reporting results [SIA 2007; 39: 464].

ISO/TR 18392:2005 SCA—XPS—Procedures for determining backgrounds [SIA 2006; 38: 1173].

ISO 21270:2004 SCA—X-ray photoelectron and Auger electron spectrometers—Linearity of intensity scale [SIA 2004; 36: 1645].

ISO 24236:2005 SCA—AES—Repeatability and constancy of intensity scale [SIA 2007; 39: 86].

ISO 24237:2005 SCA—XPS—Repeatability and constancy of intensity scale [SIA 2007; 39: 370].

ISO 29081:2010 SCA—AES—Reporting of methods used for charge control and charge correction [SIA 2011; 43: 1444].

ISO/TR 18394:2016 SCA—AES—Derivation of chemical information [SIA 2007; 39: 556].

ISO 18118:2004 SCA—AES and XPS—Guide to the use of experimentally determined relative sensitivity factors for the quantitative analysis of homogeneous materials [SIA 2006; 38: 178].

ISO 14701:2018 SCA—XPS—Measurement of silicon oxide thickness [SIA 2012; 44: 876].

ISO 16129:2018 SCA—XPS—Procedures for assessing the day-to-day performance of an X-ray photoelectron spectrometer.

ISO 22581:2021 SCA—Near real-time information from the X-ray photoelectron spectroscopy survey scan—Rules for identification of, and correction for, surface contamination by carbon-containing compounds.

ISO 18554:2016 SCA—Electron spectroscopies—Procedures for identifying, estimating and correcting for unintended degradation by X-rays in a material undergoing analysis by X-ray photoelectron spectroscopy.

ISO 19668:2017 SCA—XPS—Estimating and reporting detection limits for elements in homogeneous materials [SIA 2017, 50, 87].

ISO 19830:2015 SCA—Electron spectroscopies—Minimum reporting requirements for peak fitting in X-ray photoelectron spectroscopy.

ISO/TR 23173:2021 SCA—Electron spectroscopies—Measurement of the thickness and composition of nanoparticle coatings [SIA 2021, 53, 893].

4.3 | Secondary ion mass spectrometry

ISO 13084:2018 SCA—SIMS—Calibration of the mass scale for a time-of-flight secondary-ion mass spectrometer.

ISO 17862:2013, SCA—SIMS—Linearity of intensity scale in single ion counting time-of-flight mass analysers.

ISO 20411:2018 SCA—SIMS—Correction method for saturated intensity in single ion counting dynamic secondary ion mass spectrometry.

ISO 23830:2008 SCA—SIMS—Repeatability and constancy of the relative intensity scale in static secondary-ion mass spectrometry.

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 the internet (<https://www.iso.org/committee/54618.html>) or from Dr. Hidehiko Nonaka (hide.nonaka@aist.go.jp), Secretariat of ISO/TC 201, Japanese Standards Association, Toraya Bldg 7F, 4-9-22 Akasaka, Minato-ku, Tokyo 107-0052, Japan.

DATA AVAILABILITY STATEMENT

Data are available on request due to privacy/ethical restrictions.

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SUPPORTING INFORMATION

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