INTERNATIONAL STANDARD

ISO
18516

Surface chemical analysis — Determination of lateral resolution and sharpness in beam based methods with a range from nanometres to micrometres

Analyse chimique des surfaces — Détermination de la résolution latérale et de la netteté par des méthodes à base de faisceau utilisant une gamme allant des nanomètres aux micromètres

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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 2, *General procedures*.

This second edition cancels and replaces the first edition, ISO 18516:2006, which has been technically revised as follows:

1. content related to straight edge method expanded;

2. new content addressing the narrow stripe method added;

3. new content addressing the use of gratings in the determination of lateral resolution added;

4. implementation of concepts developed in ISO/TR 19319:2013(E);

5. title and scope changed to address nanotechnology following the recommendations of TC 201/SG 1.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Secondary ion mass spectrometry (SIMS), Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) are surface-analytical techniques that are used to generate chemical maps of surfaces and line scans across surfaces. These techniques can have lateral resolutions for instance as good as 10 nm for AES, 50 nm for SIMS and 5 μm for laboratory XPS and can cover areas as large as many square millimetres by using stitching techniques. Different instruments generate images with different lateral resolutions. Moreover, an analyst needs to have a suitable method to measure the lateral resolution of an instrument for any given settings. In this way, analysts can obtain the optimum lateral resolution from a given instrument, appropriate to the analytical requirements, in a consistent and clear way. The ability of the analyst to realize these resolutions in an effective way will, of course, also depend on the quality of the signal levels obtained and the level of noise. Resolution is a quality parameter of images and line scans and describes the performance of imaging instruments used to deliver them. This document is based on ISO/TR 19319:2013, which explains theoretical backgrounds of a determination of resolution and sharpness parameters used to express the performance of imaging instruments[1].

This document describes different methods for the determination of lateral resolution in beam-based methods as AES, SIMS and XPS. These are (a) the straight edge method, (b) the narrow line method and (c) the grating method. The method to be chosen for use depends on the expected value of the lateral resolution and the specific needs to be addressed. The standard is targeted at the needs of different communities: the manufacturers for specifying or benchmarking an instrument, the analysts in a laboratory for their day-to-day running of instruments to match the needs of good laboratory practice (GLP) and the analysts in testing laboratories operating under a formal accreditation scheme, for example ISO 17025, who must prepare and run standard operation protocols (SOP) for regular function control of instruments.

The annexes provide forthcoming information on how to find appropriate measurement parameters, considerations of the uncertainty of measurement and one practical example, the determination of effective lateral resolution by evaluation of a secondary ion image of a grating.

Surface chemical analysis — Determination of lateral resolution and sharpness in beam based methods with a range from nanometres to micrometres

1 Scope

This document describes methods for measuring lateral resolution and sharpness in imaging surface chemical analysis. It applies to all methods of surface analysis which use a beam to analyse the chemical composition of surfaces under defined settings of an instrument. It applies to scanning instruments, where a finely focused beam is scanned over the sample in a preselected field of view, as well as to full field imaging instruments, where the field of view is simultaneously imaged by a broad beam, an imaging lens system and a pixelated detector. The methods for measuring lateral resolution and sharpness are

— the straight edge method;

— the narrow line method;

— the grating method.

This document applies to instruments and methods that provide information on layers with nanometre thicknesses and to surfaces with nanometre-sized structures and individual nano-objects.

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 16242:2011, *Surface chemical analysis — Recording and reporting data in Auger electron spectroscopy (AES)*

ISO 16243:2011, *Surface chemical analysis — Recording and reporting data in X-ray photoelectron spectroscopy (XPS)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
edge spread function
ESF
normalized spatial signal distribution in the linearized output of an imaging system resulting from imaging a theoretical infinitely sharp edge

[SOURCE: ISO 12231:2012, 3.43, modified — note removed]

3.2
effective lateral resolution

r_e

minimum spacing at which two features of the image can be recognised as distinct and separate

Note 1 to entry: For the analytical methods in the scope of this document the image is formed from chemical composition data.

Note 2 to entry: "effective" has been added because the lateral resolution characteristic of an image is not only defined by the instrument used for taking it but also by the noise in the image data (see Reference [2] and ISO/TR 19319:2013). Effective lateral resolution r_e as introduced in this document takes account of that noise.

Note 3 to entry: The said minimum spacing can be determined as that distance between two stripes of a square-wave grating at which the dip of signal intensity between two maxima of the measured grating profile is at least four times the reduced noise.

Note 4 to entry: In microbeam analysis [ISO 22493:2008, 7.2] the term "image resolution" is defined in a similar way.

3.3
line spread function
LSF

normalized spatial signal distribution in the linearized output of an imaging system resulting from imaging a theoretical infinitely thin line

[SOURCE: ISO 12231:2012, 3.94]

3.4
noise

time-varying disturbances superimposed on the analytical signal with fluctuations leading to uncertainty in the signal intensity

Note 1 to entry: An accurate measure of noise can be determined from the standard deviation of the fluctuations.

[SOURCE: ISO 18115-1:2013, 4.315, modified — Note 2 to entry removed.]

3.5
reduced noise

standard deviation of noise σ_N multiplied by $(5/S_{pp})^{1/2}$, where S_{pp} is the number of sampling points per period in the measured grating profile

3.6
sharpness

property of an image or line scan to show a sharp line of demarcation between two adjacent areas of different signal intensities

Note 1 to entry: For the analytical methods in the scope of this document the signal intensities in images and line scans relate to chemical composition.

Note 2 to entry: In practical laterally resolved surface analysis objective measures of sharpness are realized as the distance D_{12-88} between the 12 % and 88 % intensity points in a line scan across a part of the sample containing a well-defined step function for the signal relating to the property being resolved.

[SOURCE: ISO 6196-5:1987, 05.04, modified]

3.7
signal-to-noise ratio

$R_{S/N}$

ratio of the signal intensity to a measure of the total noise in determining that signal

[SOURCE: ISO 18115-1:2013, 4.427, modified — Notes to entry removed.]

4 Symbols and abbreviated terms

AES — Auger electron spectroscopy

CRM — certified reference material

d — distance between two narrow stripes

D — dip between two maxima of signal intensity in the line profile over a grating

d_{gr} — distance between two consecutive gratings

$D_{x-(100-x)}$ — ESF steepness parameter giving the distance between points of well-defined intensities x and 100-x (e.g. 20 % to 80 %) of the profile over a straight edge

ESF — edge spread function

F_r — fit range

FWHM — full width at half maximum

I_m — mean value of signal intensity in the image of a three-stripe-grating (A-B-A)

$I_{max\,l}$ — signal intensity of the left maximum in the image of a three-stripe-grating (A-B-A)

$I_{max\,r}$ — signal intensity of the right maximum in the image of a three-stripe-grating (A-B-A)

I_{min} — signal intensity of the minimum in the image of a three-stripe-grating (A-B-A)

L — length

L_{ESF} — length range of measured values of the ESF

L_{pl} — length of a plateau of constant concentration

LSF — line spread function

P — grating period

P_0 — period of the largest non-resolved grating

P_1 — period of the first (finest) resolved grating

P_2 — period of the second resolved grating

P_{im} — period of the image of a grating

P_{int} — period at $R = 4$ determined by interpolation between P_0 and P_1

P_{ext} — period at $R = 4$ determined by extrapolation with P_1 and P_2

PSF — point spread function

q — grading factor of consecutive grating periods $q = P_{n+1} / P_n$

R — $R = D/\sigma_{Nr}$ – dip-to-reduced-noise ratio

r_e — effective lateral resolution

$R_{S/N}$ — signal-to-noise ratio

S_w	sampling step width
S_{pp}	sampling points per period as a variable
SIMS	secondary ion mass spectrometry
U	expanded uncertainty of a quantity
w_{LSF}	full width at half maximum of the line spread function
w_s	width of a stripe in the object pattern
XPS	X-ray photoelectron spectroscopy
σ_N	standard deviation of noise
σ_{Nr}	standard deviation of reduced noise

5 General information

5.1 Background

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. This region of interest could be a feature on a semiconductor wafer (such as an unwanted defect particle or contamination stain), a corrosion pit, a fibre or an exposed surface of a composite material. With growing industrial fabrication of devices with dimensions on the micrometre and nanometre scales, particularly in the semiconductor industry and for emerging nanotechnology applications, there is an increasing need to characterize materials using tools with lateral resolutions that are smaller than those of the features of interest. It is generally necessary in these applications to be able to determine that devices have been fabricated as intended (quality control), to evaluate new or current fabrication methods (process development and process control), and to identify failure mechanisms (failure analysis) of a device during its service life or after exposure to different ambient conditions. The lateral resolution is an important parameter in the application of characterization techniques such as SIMS, AES and XPS for the surface characterization of materials containing features with micrometre and nanometre dimensions.

It is clearly desirable that the lateral resolution of the technique be smaller than the lateral dimensions of the feature of interest in order that the feature can be readily imaged. The feature of interest shall 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 transition) 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 the difference in signal intensities for measurements made on and off the possible feature (materials contrast) and the observation time (through the statistical variations in the signal intensities, i.e. noise). The detectability of a feature 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. Reliable detection of a feature will also depend on instrumental stability (particularly the stability of the incident beam current in SIMS and AES or the X-ray flux in XPS, and the positional stability of the sample stage and the chemical stability of the sample during the time needed for acquisition of data.

Many authors have described and discussed the lateral resolution (often referred to as spatial resolution) of SIMS, AES and XPS instruments. ISO/TR 19319:2013 provides guidance and theoretical background on the determination of lateral resolution and related parameters in SIMS, AES and XPS.

5.2 Survey on principal methods to characterize lateral resolution in imaging surface chemical analysis

All methods presented in this document are designed for a determination of parameters that characterize the lateral resolution of an imaging instrument at specific settings. They use images or line scans obtained from specific test samples. These methods are:

— the **straight edge method** (Clause 6);

— the **narrow line method** (Clause 7);

— the **grating method** (Clause 8).

Principally all methods are suitable for any resolution from micrometre down to nanometre scales and work for all beam-based instruments used for imaging surface chemical analysis. They can be used either

— in a quick and easy manner as it is required by an analyst 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; or

— in a sophisticated manner which delivers accurate and objective values for resolution parameters by using numerical data evaluation. These sophisticated methods shall be used for instrument specification including benchmarking of instruments and documented validation of instrument performance in an accredited testing laboratory.

NOTE 1   The lateral resolution might or might not depend either on the measured electron energy, for example in imaging XPS or AES, or the selected secondary ion in SIMS.

The well-known **straight edge method** is a simple method suitable for the characterization of lateral resolution. A line scan across or an image of a straight edge measured with high signal-to-noise ratio and appropriate sampling step width shall be acquired using a test sample which displays a sharp straight chemical edge between material A and material B and appropriately long plateau lengths left and right from the edge. Different test samples for the micro and nano scale are on the market (see References [3-5]). The method delivers lateral resolution expressed as the sharpness parameter D_{12-88} which characterizes the (sigmoidal) edge spread function (ESF). The method is well suited for daily in-house performance tests.

NOTE 2   As a *single* parameter, D_{12-88} cannot fully characterize the complex sigmoidal ESF (see Reference [1] and references therein). Basically, the ESF is determined by the shape of the profile of the probe beam (e.g. Gaussian, Lorentzian, mixed shape) used for imaging. Therefore, the character of D_{12-88} is ambiguous and a minimum spacing at which two features of an image can be recognized as distinct and separate cannot be deduced from that parameter in a simple manner. This ambiguity of D_{12-88} is illustrated by Figure A.1 in Annex A.

NOTE 3   For laboratory XPS instruments where the best lateral resolution reached nowadays is around 5 µm, knife edges or mesh bars are principally suitable as test samples. In this case the vacuum becomes material B. However, when using knife edges or mesh bars the individual geometry of the straight edge has, via edge effects, impact on the resulting value of D_{12-88} and comparability of those results will be limited. There is no comprehensive investigation of the impact of edge geometry on D_{12-88} published so far.

The use of gold islands (on graphite) to run the straight edge method is not recommended because of imperfections of the straight edge due to arbitrary topography (leading to arbitrary D_{12-88} data) and the risk of too small plateau lengths for material A or B.

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 shall be acquired using a test sample which displays a narrow line of material A in B where the line width shall be sufficiently low in comparison with the expected lateral resolution. As a rough estimate the resolution shall be three to five times the width of the narrow line (see Annex E). The method delivers lateral resolution expressed as the parameter w_{LSF}, the full width at half maximum

(FWHM) of the line spread function (LSF). Different test samples for the micro and nano scale are on the market (see References [3-5]). The method is well suited for daily in-house performance tests.

NOTE 4   As a *single* parameter, w_{LSF} cannot fully characterize the complex line spread function (LSF). Basically, the LSF is determined by the shape of the profile (e.g. Gaussian, Lorentzian, mixed shape) of the probe beam used for imaging. Therefore, the character of w_{LSF} is ambiguous and a minimum spacing at which two features of an image can be recognized as distinct and separate cannot be deduced from that parameter in a simple manner.

The **grating method** enables the determination of the lateral resolution in terms of 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 gratings of material A in B measured with high signal-to-noise ratio and appropriate sampling step width (see Annex I) shall be acquired using a test sample which displays a series of gratings narrower and broader than the expected resolution. The method delivers the effective lateral resolution r_e, estimated by using 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. Different test samples for the micro and nano scale are on the market (see References [3-5]). Using its visual inspection mode (see 8.7) the method is well suited for (daily) in-house performance tests. Using its more sophisticated modes (see 8.9) it enables instrument specification and formal validation of instrument performance.

NOTE 5   The advantage of the grating method is that it is linked to a widely accepted and overarching definition of lateral resolution in microscopy. Results obtained by using the grating method for different instruments (settings) can be compared with each other.

Considerations of the uncertainty of measurement related to all three methods used for a determination of parameters that characterize the lateral resolution are given in Annexes D, F, J and K.

Test files containing data of real or simulated line profiles representing results obtained by each of the three methods are available at: http://isotc.iso.org/livelink/livelink?func=ll&objId=1864863&objAction=browse&viewType=1 .

5.3   Measurement of lateral resolution in imaging surface chemical analysis

The lateral resolution in imaging surface chemical analysis typically depends on either the characteristics of the incident radiation or the characteristics of the lens-analyser-detector system used in the spectrometer. In the former case, the lateral resolution will depend mainly on the cross-sectional dimensions (e.g. the beam diameter) of the incident radiation (e.g. electron beam in AES, X-ray beam in XPS or primary ion beam in SIMS) at the sample surface, and will improve as the beam diameter decreases.

All methods described in Clauses 6 to 8 involve measurements of the intensity of a selected spectral feature while a sufficiently sharp chemical gradient on the sample is translated through the analysis position defined by the incident beam or the analysis position is translated across the chemical gradient. The measured lateral resolution will depend on the instrumental design (i.e. the beam diameter or the electron/ion-optical design of the spectrometer) and the intrinsic sharpness of the chemical gradients used for the measurements.

NOTE   For AES, the magnitude and width of the Auger signal excited by back-scattered electrons have an impact on lateral resolution.

5.4   Dependence of lateral resolution on scan direction

The measured lateral resolution can depend upon the direction in which the translation of the sample with respect to the incident beam or the spectrometer is made. This variation can arise in any of the following three situations:

a)   if an X-ray, electron or primary ion beam of circular cross-section (i.e. the beam has axial symmetry) is incident on the sample at a non-zero angle θ relative to the surface normal; the beam-intensity profile on the sample will then be an ellipse, as shown in Figure 1;

b)   if the lateral resolution is defined by the analyser or lens, and the sample normal is not parallel with the entrance axis of the analyser;

c)   if the incident beam is astigmatic.

Lateral resolution should therefore be measured in at least two directions. In the case of a circular beam incident on a sample at some angle with respect to the surface normal, the measurements should be made along the directions of the short and long axes of the ellipse shown in the plan view of Figure 1. In the case of an astigmatic beam, the measurements should be made in at least two directions; normally, these directions should be orthogonal to each other. If possible, these directions should be chosen to show the smallest and the largest values of the lateral resolution.
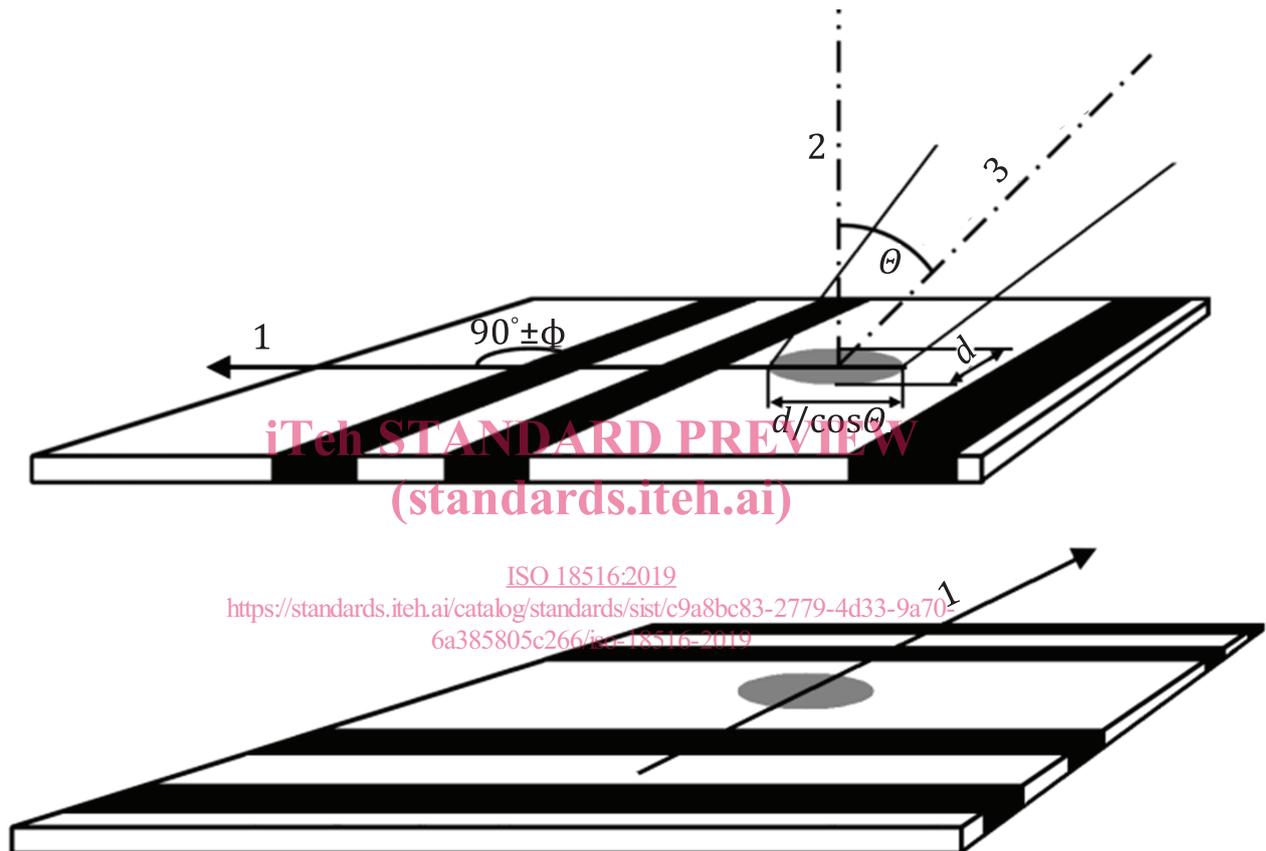
Key
1   direction of linescan
2   surface normal
3   axis of primary beam

Figure 1 — Circular beam incident on a sample impinging at an angle θ with respect to the surface normal and direction of line scan used for determination of the lateral resolution

5.5   Reporting results

Basic experimental conditions and analysis parameters shall be reported for XPS and AES instruments following the standards ISO 16242:2011 and ISO 16243:2011, respectively. Clauses 4.6 "Maps and line scans" of those standards should be considered carefully.

For SIMS such a standard is not available. Basic experimental conditions and analysis parameters to be reported for SIMS are:

—   nature of analytical ion gun and mode of its use;

— primary ion species, energy of primary ions, primary ion density, nominal beam diameter and beam shape (if known), impact geometry;

— kind of mass analyser, mode and parameters of use;

— calibrations (intensity scale, mass scale, length scale for images and line scans);

— validation of linearity of intensity scale.

There are additional parameters to be reported for the straight edge method, the narrow line method and the grating method. These method-specific parameters are summarized at the ends of Clauses 6 to 8.

## 6 Measurement of lateral resolution using the straight edge method

6.1 Introduction

Sharpness is a quality parameter of images and related to the performance of imaging instruments[6]. The term is in use in photography, microscopy and other imaging techniques. In all cases, sharpness refers to the ability to show a sharp line of demarcation between two adjacent areas of different signal intensities. This ability can be characterized by the ESF, which is the response of an imaging system to a sharp edge between material A and material B.

6.2 Model functions and sharpness parameters

A line profile over a straight edge, i.e. a step transition, yields the ESF, which is the integral over the LSF (see ISO/TR 19319:2013, Figure 2 and 4.3). The shape of the measured ESF characterizes the sharpness of images and line scans. Usually, the ESF is approximated by a specific sigmoidal function characteristic of the used imaging instrument and its settings (see ISO/TR 19319:2013, 4.3.1). For practical applications, a comparison of those specific sigmoid functions is not straightforward and a single parameter for characterizing sharpness is needed. The solution here is to use parameters $D_{x-(1-x)}$ which characterize the steepness of the ESF by distances between points of well-defined relative intensities x.

A variety of such parameters with different values of x is in use. Among them, only the parameter D_{12-88} is recommended. The reason is that for the limiting case of a Gaussian LSF the parameter D_{12-88} is equal to the FWHM of the LSF, expressed as w_{LSF}, and is close to the value of effective lateral resolution r_e (see ISO/TR 19319:2013, 4.3.4, Clause 7 and Annex A).
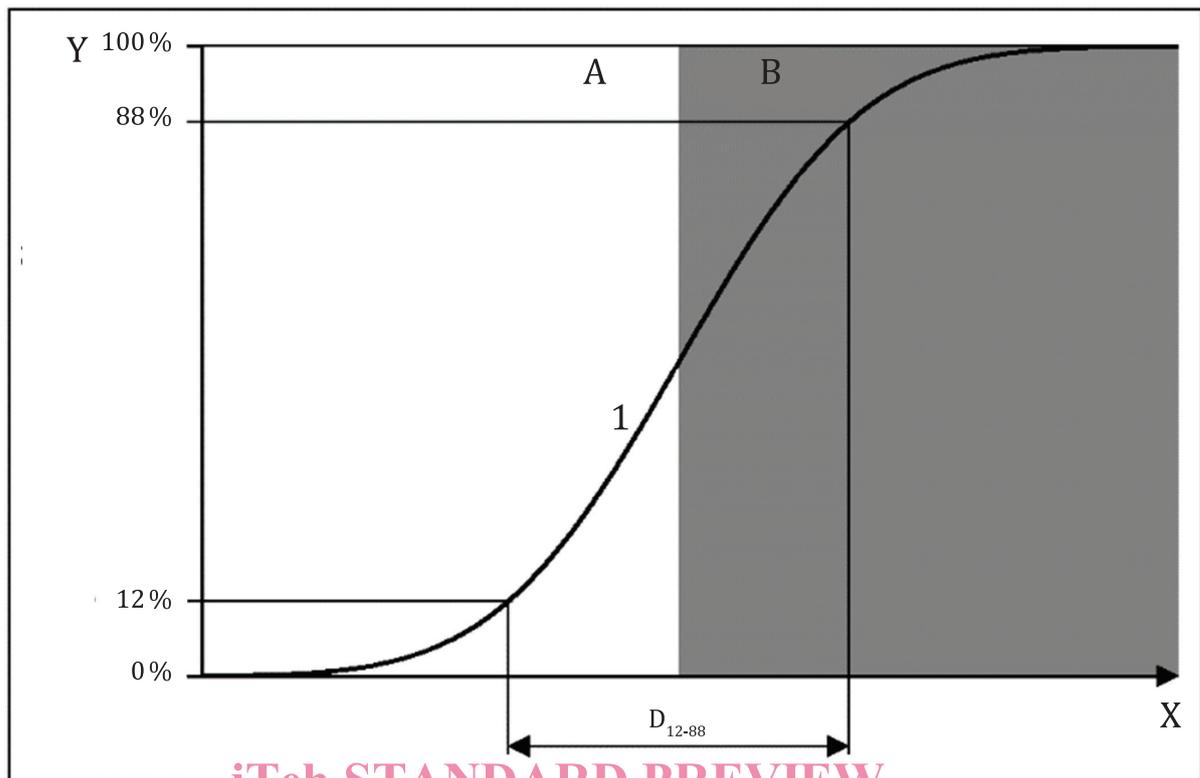
Key

A   material A
B   material B
X   length
Y   signal intensity
1   ESF profile

Figure 2 — Schematic profile over a straight edge and definition of the sharpness parameter D_{12-88}

6.3   Requirements for a test sample

Test samples shall have a sharp transition between two different materials, A and B, each with a homogeneous concentration of the detected analyte. Sharp transition means that the transition region between the materials is narrow compared with the expected rise of the ESF. The straight-edge specimen shall have a straight sharp edge whose length is at least 10 times larger than the lateral resolution to be measured. It is an advantage if the material has a large cross-section for photoelectron or Auger-electron emission or a high secondary ion yield because this minimizes the time needed to produce a signal of sufficient intensity. The specimen shall also be as smooth as possible so that signal variations due to the changing topography of the specimen are minimized.

The length of the two plateaus of constant concentration of the analytes on both sides of the chemical edge shall be large enough to enable an accurate determination of the 0 % and 100 % levels of signal intensity leading to the respective ESF parameter $D_{x-(1-x)}$. Insufficient plateau length regularly results in underestimations of $D_{x-(1-x)}$. For recommended plateau lengths for the limiting cases of Gaussian and Lorentzian beam profiles see Annex B.

In practice, common test samples are specimens with a slot, copper or gold grids on carbon, respectively. However, slots and grids are characterized by a 3D topography and edge effects, i.e. emission from non-horizontal parts of the analysed surface, will distort the intensity distribution within images and line