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Surface chemical analysis — X-ray photoelectron spectroscopy — Reporting of results of thin-film analysis

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National foreword

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Surface chemical analysis — X-ray photoelectron spectroscopy — Reporting of results of thin-film analysis

Analyse chimique des surfaces — Spectroscopie de photoélectrons X — Rapport des résultats de l'analyse de films minces

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Foreword

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The committee responsible for this document is ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 7, *Electron spectroscopies*.

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Introduction

X-ray photoelectron spectroscopy (XPS) is widely used for the characterization of surfaces of materials, especially for overlayer thin films on a substrate. The chemical composition of the near-surface region of a thin film can be determined by XPS. If the film has a uniform thickness and the thickness is less than about three times the mean escape depth (MED) for the measured photoelectrons, the film thickness and the depth distribution of elements or chemical states of elements in the film can be determined by angle-resolved XPS or peak-shape analysis . For thicker films, the depth distributions of elements in the film can be obtained by sputter-depth profiling. Possible lateral inhomogeneities in film thicknesses or depth profiles can be determined if the XPS system has sufficient lateral resolution. These XPS applications are particularly valuable for characterizing thin-film nanostructures since the MED is typically less than 5 nm for many materials and common XPS measurement conditions.

Clauses 6 and 7 of this International Standard provide guidance to the operator of an XPS instrument in making efficient measurements for determining meaningful chemical compositions and film thicknesses for overlayer films on a substrate. Clause 8 of this International Standard shows the information to be included in reports of the measurements and the analyses of the XPS data. Annex A, Annex B, Annex C, and Annex D provide supplementary information on methods of data analysis for different types of XPS measurements on thin-film samples.

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INTERNATIONAL STANDARD ISO 13424:2013(E)

Surface chemical analysis — X-ray photoelectron spectroscopy — Reporting of results of thin-film analysis

1 Scope

This International Standard specifies the minimum amount of information required in reports of analyses of thin films on a substrate by XPS. These analyses involve measurement of the chemical composition and thickness of homogeneous thin films, and measurement of the chemical composition as a function of depth of inhomogeneous thin films by angle-resolved XPS, XPS sputter-depth profiling, peak-shape analysis, and variable photon energy XPS.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18115-1:2010, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 18115-1:2010 apply.

4 Abbreviated terms

AES	Auger electron spectroscopy
ARXPS	Angle-resolved X-ray photoelectron spectroscopy
IMFP	Inelastic mean free path
MED	Mean escape depth
RSF	Relative sensitivity factor
TRMFP	Transport mean free path
XPS	X-ray photoelectron spectroscopy

5 Overview of thin-film analysis by XPS

5.1 Introduction

XPS analyses of thin films on substrate can provide information on the variation of chemical composition with depth and on film thicknesses. Several XPS methods can be used if the total film thickness is less than three times the largest MED for the detected photoelectrons. The MED for particular photoelectrons is a function of the IMFP and the emission angle of the photoelectrons with respect to the surface normal. The IMFP depends on the photoelectron energy and the material. MED values can be obtained from a database.[1] A simple analytical formula for estimating MEDs has been published for emission angles ≤50°.[2] For such emission angles, the MED is less than the product of the IMFP and the cosine of the

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emission angle by an amount that depends on the strength of the elastic scattering of the photoelectrons in the film.[2] Both the IMFP and the strength versus depend on the chemical composition of the film. The MED is typically less than 5 nm for many materials and common XPS instruments and measurement conditions. If the effects of elastic scattering are neglected, the MED is given approximately by the product of the IMFP and the cosine of the emission angle. The latter estimates of the MED can be sufficient for emission angles larger than 50° although better estimates can be obtained, e.g. from the database.[1] If the total film thickness is greater than three times the largest MED, XPS can be used under certain conditions (see Annex D) together with ion sputtering to determine the variation of chemical composition with depth.

Table 1 provides a summary of the XPS methods which can be used for determining chemical composition and/or film thickness. Some methods can be utilized for the characterization of single-layer or multiple-layer thin films on a substrate and some methods can be used to determine the composition-depth profile of a sample for which the composition is a function of depth measured from the surface (i.e. where there is not necessarily an interface between two or more phases). The choice of method typically depends on the type of sample and the analyst's knowledge of the likely or expected morphology of the sample (i.e. whether the sample can consist of a single overlayer film on a flat substrate, multiple films on a flat substrate, or a sample with composition varying continuously with depth), whether the total film thickness is less than or greater than the largest MED for the detected photoelectrons, and the desired information (i.e. film composition or film thickness). The first three methods in Table 1 are non-destructive while the final method is destructive (i.e. the composition of the exposed surface is determined by XPS as the sample is etched by ion bombardment). Brief descriptions of these methods are given in the following clauses and additional information is provided in the indicated annexes.

Table 1 — XPS methods for the characterization of thin films on substrates and for samples with composition varying with depth

Clause	Method	Sample morphology	Film thickness less than three times MED?	Information obtained	Additional information
5.2	General XPS	Single and multiple films on a flat substrate	Yes	Layer order, film thickness, and film composition	Annex A
5.3	Angle-resolved XPS	Multiple films on a flat substrate Sample with composition varying with depth	Yes	Film thickness and film composition Composition as a function of depth	Annex B
5.4	Peak-shape analysis	Multiple films on a flat substrate Sample with composition varying with depth	Yes	Film thickness and film composition Composition as a function of depth	Annex C
5.5	Variable photon energy XPS	Multiple films on a flat substrate Sample with composition varying with depth	No	Film thickness and film composition Composition as a function of depth	
5.6	XPS with sputter-depth profiling	Multiple films on a flat substrate Sample with composition varying with depth	No	Film thickness and film composition Composition as a function of depth	Annex D

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XPS is typically performed with laboratory instruments that are often equipped with monochromated Al Kα or non-monochromated Al or Mg Kα X-ray sources. For some applications, XPS with X-rays from synchrotron-radiation sources is valuable because the energy of the X-ray exciting the sample can be varied. XPS with Ag X-rays is also used to observe deeper regions compared to excitation with Al X-rays. In some cases, X-ray energies less than the Mg or Al Kα X-ray energies can be selected to gain enhanced surface sensitivity while in other cases, higher energies are chosen to gain greater bulk sensitivity and to avoid artefacts associated with the use of sputter-depth profiling.

Analysts should be aware of possible artefacts in XPS analyses. These artefacts include sample degradation during X-ray irradiation, reactions of the sample with gases in the ambient vacuum, and many effects that can occur during sputtering-depth profiling.[3]

5.2 General XPS

For a uniform thin film on a flat substrate, the film thickness can be determined from a ratio of a photoelectron peak intensity of an element in the substrate for a particular emission angle when an overlayer film is present to the corresponding intensity when the film is absent. Alternatively, the thickness can be obtained from a ratio of photoelectron peak intensity for an element in the film to the corresponding intensity for a thick film (i.e. a film with a thickness much greater than three times the MED). The composition of the film can be determined by the RSF method. Additional information is in Annex A.

For multiple thin-film analysis, it is important to determine the relative order of the layers above the substrate. We can estimate the layer order, thicknesses, and compositions by measuring the changes of peak-intensity ratios of components at two widely separated emission angles. Further details are in Annex A.

5.3 Angle-resolved XPS

Angle-resolved XPS (ARXPS)[4] can be utilized to determine composition as a function of depth for depths up to three times the largest MED of the detected electrons. The composition can be found for each film of a multilayer film on a substrate or the distribution of composition with depth can be determined for samples with no phase boundaries. For the former type of sample, film thicknesses can be estimated. Further details are in Annex B.

5.4 Peak-shape analysis

Peak-shape analysis,[5] the analysis of a photoelectron peak and its associated region of inelastically scattered electrons, can be utilized to determine composition as a function of depth for depths up to three times the largest MED of the detected electrons. The analyst can know the expected morphology of the sample (i.e. the distribution of composition with depth) or can often deduce the likely morphology from peak-shape analysis. Further details are in Annex C.

5.5 Variable photon energy XPS

Variable photon energy XPS can be employed to determine composition as a function of depth for depths up to three times the largest MED of the detected electrons.[6] XPS measurements of this type are typically performed with synchrotron radiation over a sufficiently wide photon energy range to give a useful range of MEDs of the detected photoelectrons.

5.6 XPS with sputter-depth profiling

Since 1985, “small-spot” XPS systems have been developed with lateral resolutions of commercial instruments less than 10 µm. Ion guns with focused beams have also become available so that faster sputtering of smaller regions on a sample became possible. Recent materials developments (e.g. the development of new gate oxides for semiconductor devices and the development of many types of nanostructures) have stimulated the growing use of XPS with sputter-depth profiling. It has also become necessary to obtain composition-depth profiles for inorganic and organic thin films without causing significant damage. XPS with sputter-depth profiling of such materials has now become possible with the development of buckminsterfullerene (C_{60}), argon cluster, water cluster, and other cluster

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ion sources. Low damage and low contamination by residual carbon have been reported in XPS depth profiling of several polymers using an Ar cluster-ion beam[7] and a C_{60} ion beam.[8] [9] Further details are in Annex D.

6 Specimen handling

Various types of thin-film specimens of metals, semiconductors, inorganic compounds, and polymers can be analysed by XPS. Guidelines for the preparation and mounting of specimens for analysis are given in ISO 18116[10] and ISO 18117.[11]

7 Instrument and operating conditions

7.1 Instrument calibration

The following ISO procedures should be performed to calibrate or check the performance of the XPS instrument or the analyst should check the instrument's performance by following the manufacturer's instructions or equivalent documentation.

a) calibration and checks of the binding-energy scale with ISO 15472[12]

b) checks of the repeatability and constancy of the intensity scale with ISO 24237[13]

c) checks of the linearity of the intensity scale with ISO 21270[14]

7.2 Operating conditions

7.2.1 Energy resolution

The main purpose of a wide scan is qualitative analysis. A full width at half maximum (FWHM) for the Ag $3d_{5/2}$ photoelectron peak of 2 eV is recommended for a wide scan. Narrow-scan spectra will provide quantitative information and chemical-state information and an energy resolution of less than 1 eV FWHM for the Ag $3d_{5/2}$ peak is recommended.

7.2.2 Energy range and step size

The energy range for a wide-scan spectrum shall be large enough to include the C KLL Auger peak and other potentially valuable peaks for the planned XPS analysis. The energy range should be 1 200 eV for Mg Kα X-rays and 1 400 eV for Al Kα X-rays. A step size of 1,0 eV is adequate when the energy resolution for a wide scan described in 7.2.1 is about 2 eV. For narrow scans (i.e. for chemical state analysis, quantification, or other mathematical manipulations of the XPS data), the step size should be 0,05 eV or 0,1 eV.

7.2.3 Multiple scans

Multiple scans are recommended for the acquisition of both wide scans and narrow scans to allow checks to be made of any changes in the XPS spectrum with time (e.g. can occur due to changes in X-ray intensity or to sample damage under X-ray irradiation).

7.2.4 Charge control and charge correction

Surface charging is likely for insulating samples. Techniques for charge control and charge correction are described in ISO 19318.[15] It is often convenient to use a reference C 1s binding energy between 284,6 eV and 285 eV for an observed peak due to carbonaceous contamination.[16] It is often very difficult to control the surface potential of a rough surface.

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8 Reporting XPS method, experimental conditions, analysis parameters, and analytical results

8.1 XPS method for thin-film analysis

The method chosen for XPS thin-film analysis (as summarized in Clause 5 and described in Annexes A, B, C, and D) shall be reported.

EXAMPLE 1 Angle-resolved XPS.

EXAMPLE 2 Peak-shape analysis.

EXAMPLE 3 XPS with sputter-depth profiling.

8.2 Experimental conditions

8.2.1 Introduction

The experimental conditions for the XPS measurements shall be reported. Values of the parameters described in 8.2 shall be reported. In addition, information on the XPS instrument and the experimental conditions described here shall be reported. Examples of experimental parameters and their descriptions are given in Table 2.

8.2.2 XPS instrument

The name and model of the instrument used for the XPS measurements shall be reported. If any components on the instrument are not standard for the particular model, information shall be provided on the manufacturer or on the relevant design characteristics.

EXAMPLE The instrument used for the XPS experiments was a PHI Quantera SXM.

8.2.3 XPS analyser

Analyser conditions including the electron energy analyser, the acceptance angle of the input lens, the analysed area on the sample from which signals are detected, the pass energy in eV, the energy resolution in eV, the measured binding energy ranges for each peak in eV, and the energy step in eV shall be reported.

EXAMPLE The acceptance angle of the analyser was ±20°, the acceptance area was 1 × 0,5 mm^2, the pass energy was 55 eV, the energy resolution for the XPS measurements with the X-ray source of 8.2.4 was 0,6 eV, the measured binding energy range for the Si 2p peak was 115 eV to 95 eV, and the energy step was 0,1 eV.

8.2.4 X-ray source

The type of X-ray source (e.g. Mg Kα, Al Kα, monochromatic Al Kα, use of other anodes in the X-ray source, or synchrotron radiation), the photon energy in eV, the irradiation area on the sample, and the power dissipated in the X-ray anode shall be reported. The X-ray spot size should be described together with its measurement method, if known.

EXAMPLE 1 Monochromatic Al Kα X-rays were used, the photon energy was 1 486,6 eV, the power in the X-ray anode was 50 W, and the irradiation area on the sample was 1,5 × 0,4 mm^2. The X-ray spot was circular with a diameter estimated using the knife-edge method of 100 µm. The spot diameter was measured from a line scan and corresponded to the distance between the points where the photoelectron intensity was 50 % of the difference in the intensities in the plateau regions away from each edge in the direction of the scan.

EXAMPLE 2 Conventional Mg Kα X-rays were used, the photon energy was 1 253,6 eV, and the irradiation area on the sample was approximately 10 × 20 mm^2 at 300 W.

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8.2.5 XPS configuration

The XPS configuration including the angle between the X-ray direction on the sample and the average analyser acceptance direction, the angle of X-ray incidence on the sample with respect to the surface normal, the photoelectron emission angles with respect to the surface normal, and the analyser azimuth angle with respect to the plane of X-ray incidence shall be reported.

EXAMPLE The angle between the X-ray direction and the analyser axis was 45°, the X-rays were incident normally on the sample surface, the emission angles of the photoelectrons were 0°, 25°, 37°, 45°, 53°, and 58° with respect to the surface normal, and the analyser azimuth was 22,5° with respect to the plane of X-ray incidence.

8.2.6 Charge control

The particular instrumental component(s) used for charge control shall be reported. The particular experimental conditions for charge control (such as the beam voltage in V and the total beam current in µA for the electron beam from a flood gun) shall be reported.

EXAMPLE For the flood gun, the beam voltage was –1,4 V (with respect to instrumental ground) and the total beam current was 10 µA measured on clean silver.

8.2.7 Ion gun parameters for sputter-depth profiling

Ion gun parameters for sputter-depth profiling such as ion species, beam voltage, beam current, spot size, raster size, incidence angle, sputter rate, and mass filter (if used) shall be reported.

EXAMPLE 1 The ion species was Ar^+, the beam voltage was 1 kV, the beam current was 500 nA, the spot size was 300 µm, the raster size was 2 × 2 mm^2, the incidence angle was 45°, and the sputter rate for SiO_2 was 3 nm/min.

EXAMPLE 2 The ion species was C_{60}^+, the beam voltage was 10 kV, the beam current was 10 nA, the spot size was 100 µm, the raster size was 2 × 2 mm^2, the incidence angle was 20°, the sputter rate for SiO_2 was 3 nm/min, and a mass filter was used to choose a 10 keV C_{60} ion beam.

8.3 Analysis parameters

8.3.1 Introduction

All methods and parameters used in the data analysis shall be reported. Some methods and parameters such as the transmission-function correction for the analyser, the method used for peak-intensity calculation (such as peak area or peak height), and the method used for background subtraction (and the starting and ending energies) are common to all XPS methods described here. If film compositions are reported, the type of relative sensitivity factor and the values of these factors shall be reported for each peak. Examples of analysis parameters and their descriptions are given in Table 3.

EXAMPLE The transmission-function correction was made from measurements of peak area/pass energy versus retarding ratio, peak areas were used for intensity calculations, the iterated Shirley background was used, the starting and ending binding energies for the Si 2p peak were 107 eV and 97 eV, respectively, and the average matrix relative sensitivity factors for the Si 2p was 0,368.

8.3.2 IMFP

Values of the IMFPs used in film-thickness calculations by general XPS, peak-shape analysis, and XPS with sputter-depth profiling shall be reported together with the source of the data.

EXAMPLE The IMFP for the Si 2p peak with Al Kα X-rays of 3,2 nm was obtained from the TPP-2M equation.[17]

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8.3.3 Single-scattering albedo

Values of the single-scattering albedo, if used in film-thickness calculations as described in Annex A, should be reported.

EXAMPLE The single-scattering albedo for the Si 2p peak with Al Kα X-rays was 0,111. This value was calculated from the ratio of the IMFP to the sum of the IMFP and TRMFP,[18] as described in Annex A.

8.3.4 Parameters for peak-shape analysis

The chosen structure model (e.g. buried thin film, exponential depth profile, homogeneous depth profile, substrate with overlayer) and values of the parameters B, C, and D in the selected Tougaard inelastic-scattering cross-section formula (e.g. for metals and oxides, polymers, SiO_2, Si, Ge, and Al[83]) shall be reported. Information on the structure models and the various parameters is given in Annex C.

EXAMPLE A substrate with an overlayer was the chosen morphology model and recommended values of the parameters B and C for metals and oxides of 2 866 eV^2 and 1 643 eV^2, respectively, were used (the parameter D was not used).

8.3.5 Parameters for angle-resolved XPS

The type of algorithm used for depth profile reconstruction shall be reported. If the maximum entropy algorithm is used, the value of the regularizing constant for the final results shall be reported. Any corrections applied in the calculation of the depth profiles (e.g. for the asymmetry parameter, sample crystallinity, surface roughness, and elastic scattering) shall be reported. Information on analysis algorithms and corrections is given in Annex B.

EXAMPLE The maximum entropy method was used. The value of the regularizing constant α was fixed at 5×10^{-4} during the calculation.[19]

8.3.6 Special methods

Any special methods used for data analysis (e.g. curve fits to extract chemical states, linear least-square fitting, target factor analysis) shall be reported.

EXAMPLE A curve fit was applied to the Si 2p spectrum to determine the intensities of the metal and oxide chemical states.

8.4 Examples of summary tables

Summary tables for methods, acquisition parameters, and analysis parameters, as shown in Tables 2 and 3, can be convenient and useful for day-to-day use.

Table 2 — Examples of experimental conditions to be reported, as described in 8.2

Parameters	Description
Date	2010–04–01
Sample description	SiO_2(2,0 nm)/Si(100) (substrate)
XPS method	Film thickness analysis Peak-shape analysis
XPS instrument	PHI Quantera SXM
XPS configuration	
Angle between analyser and X-ray source	45°
Emission angle	45°
Analyser azimuth	22,5° with respect to the plane of X-ray incidence
Analyser condition	

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Table 2 *(continued)*

Parameters	Description
Type of electron energy analyser	Concentric hemispherical analyser (CHA)
Acceptance angle	±20°
Acceptance area	1 × 0,5 mm^2
Photoelectron peak 1	Si 2p
Energy range	112 ~ 92 eV
Energy step	0,1 eV
Pass energy	55,0 eV
Photoelectron peak 2	O 1 s
Energy range	542 ~ 522 eV
Energy step	0,1 eV
Pass energy	55,0 eV
Photoelectron peak 1	C 1s
Energy range	298 ~ 278 eV
Energy step	0,1 eV
Pass energy	55,0 eV
X-ray source condition	
Type of X-ray source, energy, and power	Monochromatized Al Kα, 1 486,6 eV, 25 W
Expected spot size	100 µm in diameter
Charge control	1,4 eV 10 µA electron and 7 eV 35 nA Ar ion beam irradiation
Sputter on beam	Not used for this analysis but typical value for sputter cleaning is described below
Gas species	Ar
Beam voltage and current	1 kV, 500 nA
Spot size	300 µm in diameter
Raster size	2 × 2 mm^2
Incident angle	40°
Sputter rate for SiO_2	3 nm/min
Mass filter	None

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Table 3 — Examples of analysis parameters to be reported, as described in 8.3

Parameters	Description
Analysis mode	Film thickness
General parameter	The transmission-function correction was made from measurements of peak area/pass energy versus retarding ratio, peak areas were used for intensity calculations, the iterated Shirley background was used, the starting and ending binding energies for the Si 2p peak were 107 eV and 97 eV, respectively, and the average matrix relative sensitivity factors for the Si 2p peak was 0,368
Inelastic mean free path	3,2 nm for Si 2p peak with Al Kα X-rays
Single-scattering albedo	ω = 0,111 for Si 2p peak with Al Kα X-rays
Parameters for peak-shape analysis	Not applicable
Parameters for angle-resolved XPS	Not applicable
Special method	Curve fit to extract elemental Si and Si oxide peaks

8.5 Analytical Results

Depending on the detail requested by a customer, the following analytical results shall be reported together with the chosen analysis method (as listed in Table 1).

a) film layer order

b) film thickness and composition

c) composition as a function of depth

The recording and reporting of these information should follow ISO 16243.[20]

Details and examples of the XPS analysis methods described are shown in Annexes A, B, C, and D. Table 1 can be used to select an analysis method that is suitable for the desired information.

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Annex A
(informative)

General XPS

A.1 Introduction

Methods to obtain thin-film thickness, thin-film composition, and the structure of a multilayer film are described. In structure analysis, the relative order of the layers above the substrate or the relative depths of different functional groups can be obtained.

A.2 Symbols and abbreviated terms

AMRSF Average matrix relative sensitivity factor

ARSF Atomic relative sensitivity factor

EAL Effective attenuation length

E_f Energy of the photoelectron emitted from the overlayer

E_s Energy of the photoelectron emitted from the substrate

ERSF Elemental relative sensitivity factor

$I_A^{\theta_L}$ Intensity of component "A" at lower emission angle

$I_A^{\theta_H}$ Intensity of component "A" at higher emission angle

$I_B^{\theta_L}$ Intensity of component "B" at lower emission angle

$I_B^{\theta_H}$ Intensity of component "B" at higher emission angle

I_f Intensity of photoelectrons emitted from the overlayer ("overlayer signal")

I_n Intensity of photoelectrons emitted from *n*th layer from the top

I_{n+1} Intensity of photoelectrons emitted from *n+1*th layer from the top

I_s Intensity of photoelectrons emitted from the substrate and transmitted through the thin-film overlayers

I_0^f Intensity of photoelectrons emitted from the semi-infinite overlayer material

I_0^s Intensity of photoelectrons emitted from the semi-infinite substrate

L_n Effective attenuation length of the photoelectron in the *n*th layer

L_{n+1} Effective attenuation length of the photoelectron in the *n+1*th layer

$L(E)$ Effective attenuation length at kinetic energy E

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