
Surface chemical analysis — Scanning-probe microscopy — Determination of geometric quantities using SPM: Calibration of measuring systems

Analyse chimique des surfaces — Microscopie à sonde à balayage — Détermination des quantités géométriques en utilisant des microscopes à sonde à balayage: Étalonnage des systèmes de mesure

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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The committee responsible for this document is ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 9, *Scanning probe microscopy*.

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Introduction

The progress of miniaturization in semiconductor structuring, together with the rapid advance of many diverse applications of nanotechnology in industrial processes, calls for reliable and comparable quantitative dimensional measurements in the micro- and submicrometre range.^[9] Currently, a measurement resolution, in or below the nanometre region, is frequently required. Conventional optical or stylus measurement methods or coordinate measuring systems are not able to offer this level of resolution.

For this reason, scanning-probe microscopes (SPMs) are increasingly employed as quantitative measuring instruments. Their use is no longer confined only to research and development, but has also been extended to include industrial production and inspection.

For this category of measuring instrument, standardized calibration procedures need to be developed, for example, as have been established already long ago for contact stylus instruments (see ISO 12179). For efficient and reliable calibration of SPMs to be carried out, the properties of the measurement standards used need to be documented and be accounted for in the calibration (see Figure 1) and, at the same time, the procedure for the calibration should be clearly defined.

Only if this prerequisite is satisfied, will it be possible to perform traceable measurements of geometrical quantities.

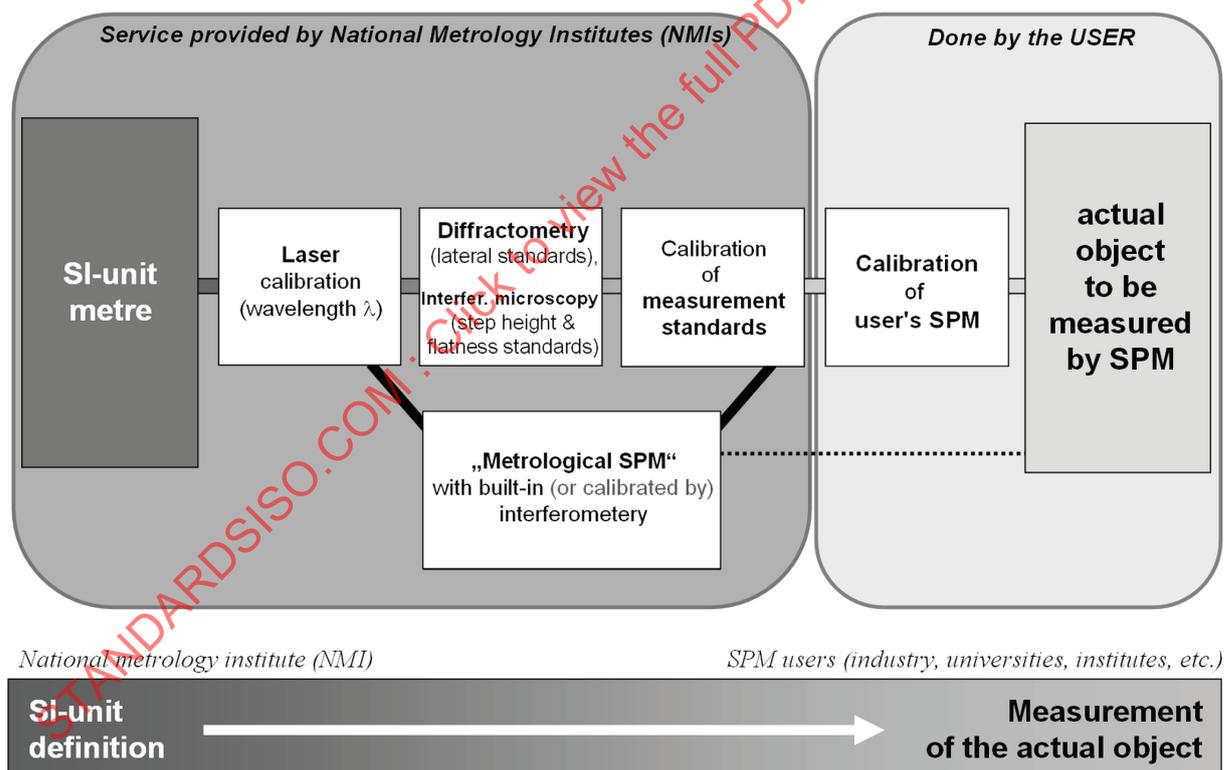


Figure 1 — Traceability chain for scanning-probe microscopes

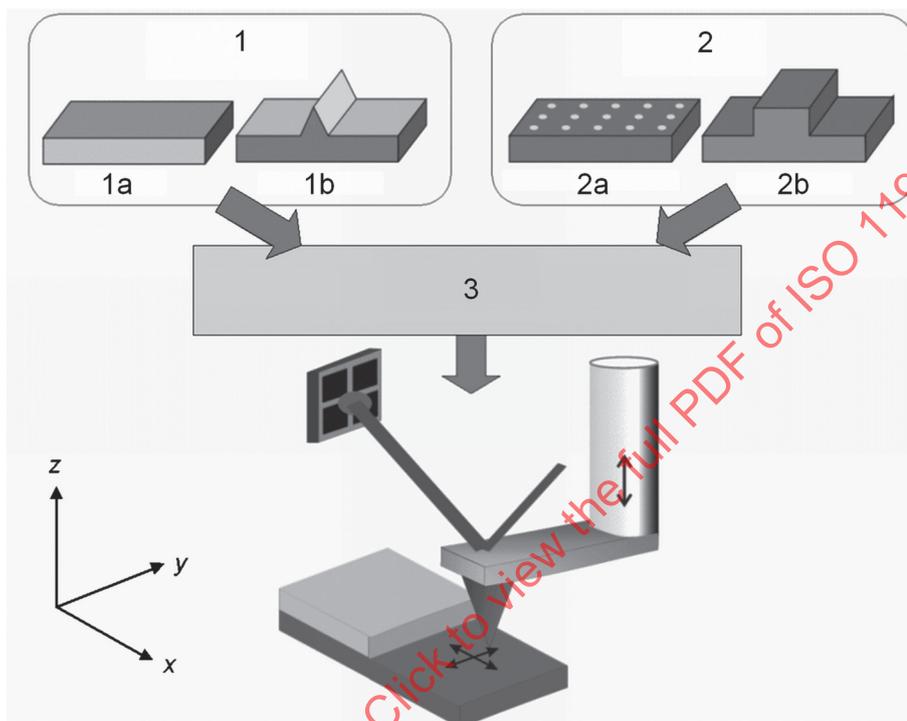
NOTE The calibration of a user's SPM by means of traceably calibrated measurement standards is the object of this International Standard (done by the user).

A scanning-probe microscope is a serially operating measuring device which uses a probe with a tip of adequate fineness to trace the surface of the object to be measured by exploitation of a local physical interaction (such as the quantum-mechanical tunnel effect, interatomic or intermolecular forces, or evanescent modes of the electromagnetic field). The probe and the object to be measured are being displaced in relation to one another in a plane (hereinafter referred to as the *x-y*-plane) according to a defined pattern,^[10] while the signal of the interaction is recorded and can be used to control the distance

between probe and object. In this International Standard, signals are considered which are used for the determination of the topography (hereinafter called the “z-signal”).

This International Standard covers the verification of the device characteristics necessary for the measurement of geometrical measurands and the calibration of the axes of motion (x, y, z), [1] i.e. the traceability to the unit of length via measurement on traceable lateral, step height, and 3D measurement standards (see Figure 2).

While this International Standard aims at axis calibrations at the highest level and is thereby intended primarily for high-stability SPMs, a lower level of calibration might be required for general industry use.



Key

- 1 measurement standards for verification purposes
- 1a flatness
- 1b probe shape
- 2 measurement standards for calibration purposes
- 2a 1D and 2D lateral
- 2b step height
- 3 calibration of the measurement standards by reference instruments (certified calibration, measurement value including uncertainty)

Figure 2 — Verification and calibration of scanning-probe microscopes with test specimens and measurement standards

This International Standard is mainly based on the guideline VDI/VDE 2656, Part 1, drafted by a guideline committee of the VDI (Verein Deutscher Ingenieure/Association of German Engineers) in the years 2004 to 2008, with the final whiteprint of that guideline being released in June 2008.

Surface chemical analysis — Scanning-probe microscopy — Determination of geometric quantities using SPM: Calibration of measuring systems

1 Scope

This International Standard specifies methods for characterizing and calibrating the scan axes of scanning-probe microscopes for measuring geometric quantities at the highest level. It is applicable to those providing further calibrations and is not intended for general industry use, where a lower level of calibration might be required.

This International Standard has the following objectives:

- to increase the comparability of measurements of geometrical quantities made using scanning-probe microscopes by traceability to the unit of length;
- to define the minimum requirements for the calibration process and the conditions of acceptance;
- to ascertain the instrument's ability to be calibrated (assignment of a "calibrate-ability" category to the instrument);
- to define the scope of the calibration (conditions of measurement and environments, ranges of measurement, temporal stability, transferability);
- to provide a model, in accordance with ISO/IEC Guide 98-3, to calculate the uncertainty for simple geometrical quantities in measurements using a scanning-probe microscope;
- to define the requirements for reporting results.

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 11039, *Surface chemical analysis — Scanning-probe microscopy — Measurement of drift rate*

ISO 18115-2, *Surface chemical analysis — Vocabulary — Part 2: Terms used in scanning-probe microscopy*

IEC/TS 62622, *Artificial gratings used in nanotechnology — Description and measurement of dimensional quality parameters*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-2 and IEC/TS 62622 and the following apply.

3.1

scanner bow

additional deflection in the z -direction when the scanner is displaced in the x - y -direction

Note 1 to entry: Scanner bow is also known as out-of-plane motion (see also xtz , yzt in [Clause 4](#)).

3.2

look-up table

table in which a set of correction factors for the scanner are filed for different modes of operation (scan ranges, scan speeds, deflections, etc.)

3.3

step height

height of an elevation (bar) or depth of a groove (ISO 5436-1), in atomic surfaces, the distance between neighbouring crystalline planes

3.4

levelling

correction of the inclination between the ideal x - y -specimen plane and the x - y -scanning plane

4 Symbols

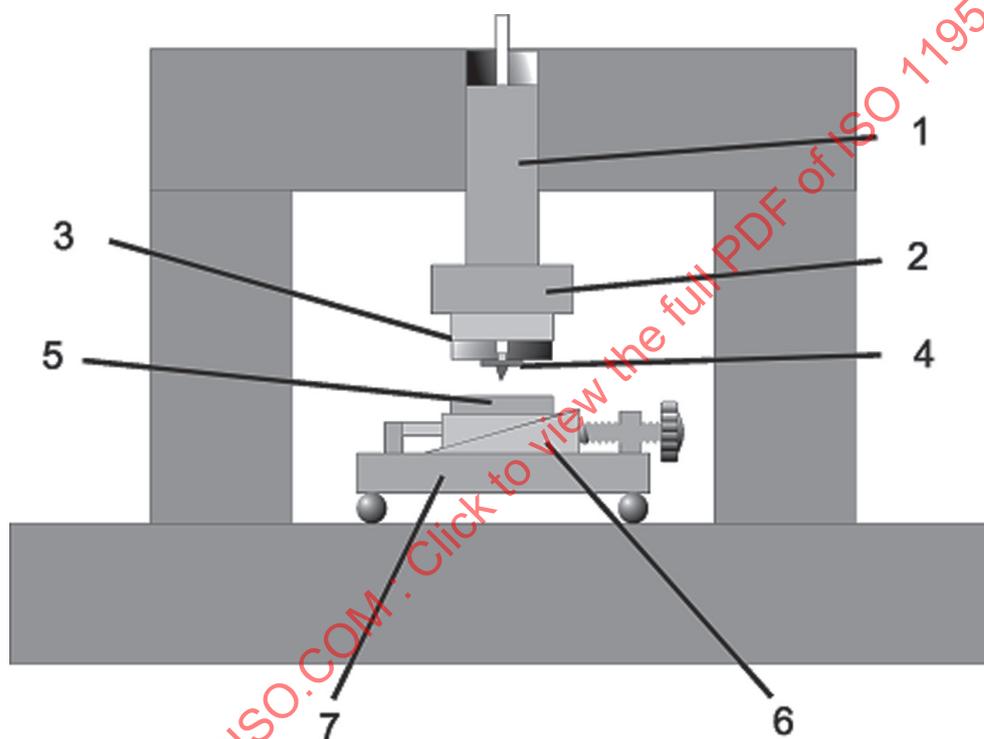
x, y, z	position value related to the respective axis
C_x, C_y, C_z	calibration factors for the x -, y -, and z -axes
h	step height
w	width of a structure of the specimen
N_j	i^{th} pitch value in a profile used for the determination of the pitch/period (number of pitch values i over all lines $j = 1, \dots, N_j$)
p_x	pitch or period in the x -direction
p_y	pitch or period in the y -direction
a_x	vector in the x -direction of a grating (not to be confused with p_x)
a_y	vector in the y -direction of a grating (not to be confused with p_y)
γ_{xy}	non-orthogonality of 2D gratings
$P-V$	peak-to-valley value
r	radius
Rq (Sq)	root mean square deviation of the assessed roughness profile (Rq) or of the assessed area (Sq)
T	temperature
α_m	thermal expansion coefficient
T_L	temperature of the air
T_m	temperature of the specimen during measurement
j_x	angle of rotation about the x -axis
j_y	angle of rotation about the y -axis
j_z	angle of rotation about the z -axis
θ	levelling angle
x_L	value of the measurement standard for shift in the x -direction
x_m	shift in the x -direction measured with the x -displacement transducer

xtx	positional deviation Δx measured along an x -coordinate line
xy	straightness deviation Δy measured along an x -coordinate line
xtz	straightness deviation Δz measured along an x -coordinate line
xrx	rotational deviation j_x measured along an x -coordinate line
xry	rotational deviation j_y measured along an x -coordinate line
xrz	rotational deviation j_z measured along an x -coordinate line
xwy	measured rectangularity deviation in the coordinate plane x - y
xwz	measured rectangularity deviation in the coordinate plane x - z
y_L	value of the measurement standard for displacement in the y -direction
y_m	displacement measured with the y -displacement transducer in the y -direction
ytx	positional deviation Δx measured along a y -coordinate line
yty	straightness deviation Δy measured along a y -coordinate line
ytz	straightness deviation Δz measured along a y -coordinate line
yrx	rotational deviation j_x measured along a y -coordinate line
yry	rotational deviation j_y measured along a y -coordinate line
yrz	rotational deviation j_z measured along a y -coordinate line
ywz	rectangularity deviation measured in the coordinate plane y - z
z_L	value of the measurement standard for displacement in the z -direction
z_m	displacement in the z -direction measured with z -displacement transducer
ztx	straightness deviation Δx measured along a z -coordinate line
zty	straightness deviation Δy measured along a z -coordinate line
ztz	straightness deviation Δz measured along a z -coordinate line
zrx	rotational deviation j_x measured along a z -coordinate line
zry	rotational deviation j_y measured along a z -coordinate line
zrz	rotational deviation j_z measured along a z -coordinate line
$\cos(\varphi_i)$	rotational correction, e.g. in pitch measurement
$\cos(\theta_i)$	tilt-related correction, e.g. in pitch measurement
λ_s	short-wavelength filter (see ISO 4287 for details)
λ_c	long-wavelength filter (see ISO 4287 for details)
Λ	correlation length
ϕ_{xy}	angle between the x - and y -direction, counterclockwise
ϕ_{xz}	angle between the x - and z -direction, counterclockwise

- ϕ_{yz} angle between the y - and z -direction, counterclockwise
- R_{qx} noise in the x -direction
- R_{qy} noise in the y -direction
- R_{qz} (S_{qz}) noise in the z -direction in a measured profile (or within a measured area)
- v scan speed (i.e. distance travelled by the probe tip per unit time, not to be confused with the scan rate, i.e. the number of scanlines recorded per unit time)

5 Characteristics of scanning-probe microscopes

5.1 Components of a scanning-probe microscope



Key

- 1 x - y -scanner
- 2 z -scanner
- 3 position detector
- 4 probe
- 5 specimen
- 6 coarse z -approach, i.e. move the probe or the specimen in the vertical direction to bring it close enough to the specimen or probe, respectively (afterwards, start automatically approach techniques).
- 7 coarse x - y -positioning, i.e. move the specimen or probe laterally close to or into the region of interest on the specimen, respectively

Figure 3 — Schematic sketch of a scanning-probe microscope

Several components shown in Figure 3 are defined in ISO 18115-2. In this International Standard, they fulfil the following functions.

- *Probe*: equipped with a tip at its apex. This probes the specimen surface, exploiting a local physical interaction whose changes can be detected, e.g. as cantilever bending in the case of an atomic force microscope.
- *Position detector*: Transformation of the probe's interaction response (e.g. bending or oscillation of the cantilever) into an electrical signal.
- *z-scanner*: Element for the realisation of the vertical tracking of the specimen/probe distance during *x-y*-scanning to a constant value of the physical interaction used for distance control (e.g. of the action of force on the probe in the case of an atomic force microscope), to ensure an approximately constant distance between specimen and probe.
- *x-y-scanner*: Element for realisation of the lateral displacement of the probe (or of the specimen) in the *x-y*-plane (the plane parallel to the seating face of the specimen), which is used, among other things, to record a location-dependent interaction signal that contains information about a local property of the specimen (above all, the local height).
- *Specimen holder*: where appropriate, with coarse positioning and coarse approach mechanics.
- *Casing/mounting*: Structure for mounting the scanner and specimen.

5.2 Metrological categories of scanning-probe microscopes

SPMs can generally be subdivided into the three following categories, depending on their metrological equipment:

- category A: Reference instruments with integrated laser interferometers, allowing direct traceability, via the wavelength of the laser used, to the SI unit of length.¹⁾
- category B: SPMs with position measurement using displacement transducers, e.g. capacitive/inductive sensors, strain gauges or encoders calibrated by temporarily connecting laser interferometers to the instrument or by making measurements on high-quality measurement standards. A distinction is made between the following two types:
 - those with active position control: tracking to a scheduled position by means of a closed loop (so-called closed-loop configuration);
 - those with position measurement but without a closed loop for position control (so-called open-loop configuration).
- category C: SPMs in which the position is determined from the electrical voltage applied to the adjustment elements and, if need be, corrected using the look-up table. Calibration is against measurement standards.

These definitions of metrological categories imply that it is not possible for certain instruments to be assigned to a single category, but that, with respect to their scan axes, they need to be considered separately.

5.3 Block diagram of a scanning-probe microscope

The block diagram shown in [Figure 4](#) has been obtained from the schematic diagram of an SPM in [Figure 3](#). The characteristics of the essential components are given below and need to be investigated individually in the course of verification and calibration.

1) Instruments of this category are often referred to as “metrological SPMs”, although the definition of a “metrological SPM” in ISO 18115-2:2010/Amd.1 (to be published) does not necessarily imply laser-interferometric position control.

For category C:

- casing/mounting (mechanical, acoustic, electromagnetic, and thermal characteristics);
- specimen holder, where appropriate with coarse positioning and coarse approach mechanics (acoustic, mechanical, and thermal characteristics);
- z-scanner;
- x-y-scanner;
- detector loop, e.g. using the beam deflection method, with a beam on the rear side of the cantilever in the case of an atomic force microscope and detection of the reflected beam from the rear side of the cantilever with a position-sensitive photodiode. The signal of the position-dependent photodiode serves as input to the feedback loop of the z-scanner in order to keep the set-point constant;
- probe.

Additionally, for category B:

- category B2: x-, y-, and/or z-displacement transducer, e.g. encoder, capacitive, or inductive displacement transducer or strain gauge;
- category B1: where appropriate, active (closed-loop) position control.

Additionally, for category A:

Traceability by integrated laser interferometers, i.e. systems as for category B, but equipped with

- integrated laser interferometers for position measurement/control and
- where appropriate, additionally provided with angle sensors.

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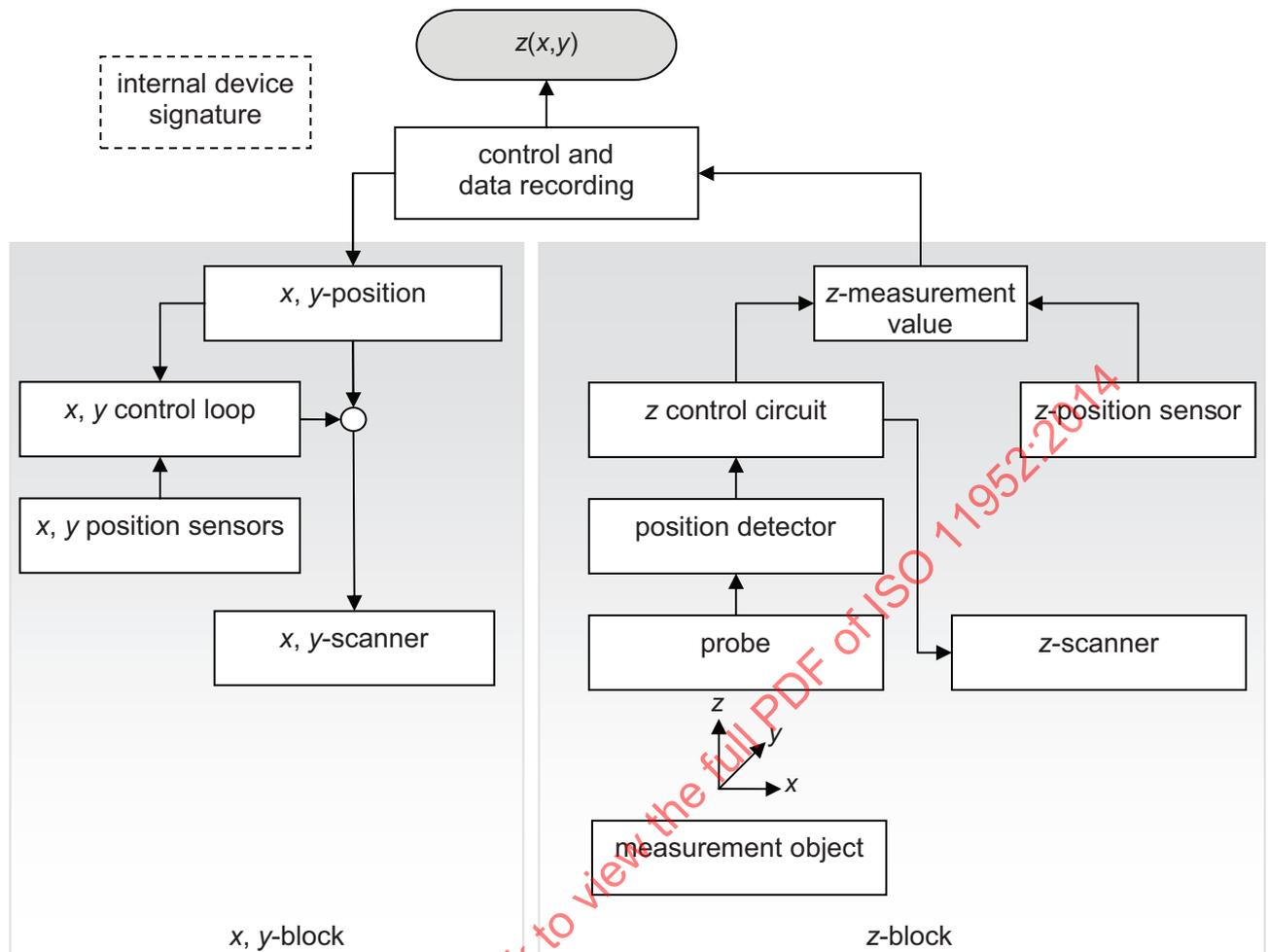


Figure 4 — Block diagram of a scanning-probe microscope

The classification above is a first rough estimation of the effort necessary to achieve the desired accuracy of calibration. It is not necessary, for example, to purchase a set of measurement standards with minimum uncertainties of measurement for the calibration of category C instruments. Less sophisticated measurement standards are usually sufficient here.

5.4 Calibration interval

The interval at which the instrument will need to be calibrated depends on the type of instrument (i.e. the metrological category), its stability, especially with respect to time, the intended purpose of the measurements and the constancy of the ambient conditions. As most calibrations are of a complex nature, and thus, are labour- and time-intensive, a compromise needs to be found between the cost of calibration and the measurement uncertainty which can be tolerated.

Generally, the following repetition patterns for calibrations (K) and measurements (M) are suitable.

KMM ..., KMM ...	for instruments of high stability in the medium term: calibration is necessary only at defined intervals of time, e.g. once weekly/monthly/yearly.
KM, KM, KM ...	for instruments with acceptable short-term but bad long-term stability: calibration is necessary before each measurement.
KMK, KMK ...	when the maximum precision of the instrument is to be used for measurements with as small an uncertainty of measurement as possible or for instruments which are unstable with time and therefore require the drift in their characteristics to be taken into account as far as possible.

Especially after putting into operation an SPM which is new or has been modified or relocated, it is advisable in the initial phase to repeat a defined calibration pattern several times in order to gain experience with the stability of the instrument.

6 Preliminary characterization of the measuring system

6.1 Overview of the instrument characteristics and influencing factors to be investigated

In order to define a calibration schedule for a particular SPM, three groups of influencing factors need to be investigated in detail (see [Figure 5](#)): the instrument's characteristics (as described above), the ambient conditions, and the effects of operation by the user.

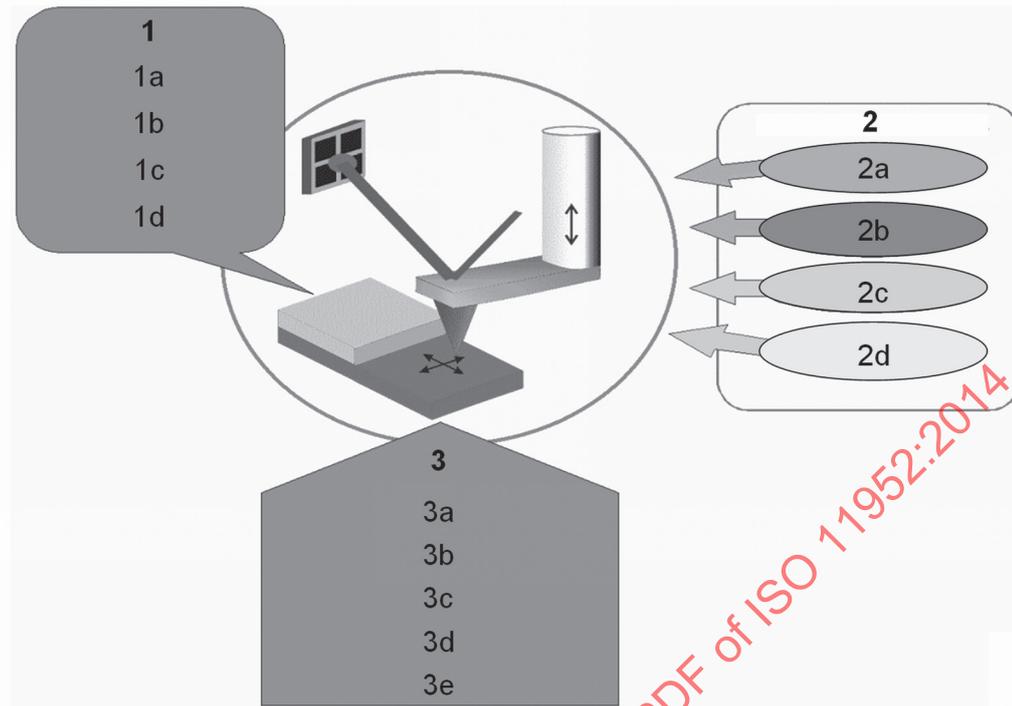
These investigations should be carried out in the following order, prior to the calibration process proper:

- investigation of the waiting time after putting the instrument into operation (warm-up, initial drift, etc.) (see [6.2](#));
- investigation of the waiting time after changing the specimen or probe or other interventions before sufficiently stable conditions of measurement are reached (see [6.2](#));
- the influence of the ambient conditions, producing a temporary drift and/or changes in temperature, air humidity, air flow, mechanical, and acoustic vibrations, electromagnetic interference, etc. (see [6.3](#));
- the noise of the instrument (see [6.3](#) and also [Table 1](#));
- xy-scanner/z-scanner-guidance deviations (cross-talk from one scan axis to other axes, which can, at times, be detectable only by repeated measurements) (see [7.3](#));
- long-term stability (reproducibility) (see [5.4](#)).

These investigations can be carried out as qualitative and/or as quantitative tests. For qualitative tests, specimens with the desired properties (e.g. silicon wafers, glass plates) are sufficient, whereas for quantitative tests calibrated measurement standards are required for precise work. This is described in [Clause 7](#).

The investigations described below should be performed with probes which are usually used for measurements with the instrument in question and on the specimens to be examined. Ageing of the probe tips can be identified with the aid of suitable tests.^[44-47] Tips showing excessive wear should not be used.

The first step should be aimed at separating the various influences, e.g. by cutting out external influences and allowing them back in (to the extent possible), and then successively varying the operator-related settings.

**Key**

1	intrinsic influences	2d	temperature changes
1a	probe-guidance deviations	3	operator-related settings
1b	signal drift	3a	parameter settings for the feedback loop, i.e. proportional (<i>P</i>) and integral (<i>I</i>) gain
1c	mechanical stress	3b	scan range
1d	electronic noise	3c	scan speed/scan rate
2	extrinsic influences	3d	forward/backward scan
2a	mechanical vibration	3e	features of probe and specimen
2b	acoustic vibration		
2c	electrical noise		

Figure 5 — The three groups of factors influencing the measurement process

Table 1 — Influence of ambient conditions and noise of the instrument

Characteristics		Specimen and method of investigation	Subclause
Drift	Vertical	Flatness measurement standard or specimen with known flat regions (Rq or $Sq < 2$ nm, $P-V < 10$ nm) Variation of ambient conditions, opening of chamber, switching on/off of instrument components, etc.	6.2
	Lateral	Specimen with straight edges or lines of small step height, aligned parallel or vertical to the scan direction 2D grating of small step height.	6.2 7.5
z-Noise	Static: without movement in x or y	After stabilization of the instrument measurement of flatness measurement standards with x - y -movements switched off. In addition, variation of ambient conditions, i.e. mechanical damping, acoustic oscillations, electromagnetic shielding.	
	Dynamic: scanning in x or y	After stabilization of the instrument fast recording of two or more scan lines. The difference between the lines provides information about dynamic noise components.	

The separation of the contributions is the basis for the introduction of suitable optimization procedures, the use of correction procedures (if these two are not feasible) or adequate inclusion in the uncertainty budget. [14][16][17] Table 1 can serve to make a distinction between temporary drift and permanent guidance deviations as well as between different contributions to the noise (see also example in Annex A).

6.2 Waiting times after interventions in the measuring system (instrument installation, intrinsic effects, carrying out operation, warm-up, tip/specimen change, etc.)

6.2.1 Adjustment of the instrument to ambient conditions

The waiting times investigated in this subclause relate to an instrument which has already adjusted to its ambient conditions. After rearrangement and installation of the instrument or relocation to another room, about 24 h are typically to be allowed for acclimatization.

6.2.2 Potential causes of drift

During the warm-up phase after switching-on the instrument, or after interventions such as changing or repositioning the probe/tip and specimen, the following effects might influence the measurements:

- piezo drift or piezo creep in the lateral/vertical direction;
- mechanical stresses, e.g. acting on the specimen holder and its mounting (e.g. adhesive);
- mechanical expansion of the components (casing, measurement circle);
- changes in the properties of the electronics.

As drift usually disappears after some time, the required waiting time is to be determined. For the electronics, a warm-up period of at least 30 min is to be reckoned with. For the other drift contributions, however, no generally valid decay times can be given, as they depend on the particular type of instrument.

6.2.3 Procedure

Follow the procedure for drift determination as specified in ISO 11039.

6.3 External influences

6.3.1 Kinds of external influence

As SPMs are most sensitive to interference from the environment, the following influence quantities are to be accounted for:

- variations of temperature and air humidity;
- air motion (e.g. air-conditioning, air circulation, draughts, exhaust heat);
- dust;
- mechanical vibrations (e.g. structural vibrations, foot fall sounds/human traffic, pumps);
- acoustic disturbances (e.g. impact sound, ambient noise);
- electrical and electromagnetic sources of interference;
- presence of staff.

These external influences can produce drifts (see 6.2), noise (see Table 1), and systematic errors.

6.3.2 Consequences of external influences and countermeasures

In the presence of mechanical and acoustic vibrations in particular, it often is sufficient to take relatively simple countermeasures (avoidance of sound, vibration damping, sound-proofing hood or the like, see Annex C). Air currents and dust can be prevented by suitable encapsulation. Electrical interferences can, if necessary, be compensated for by appropriate measures (e.g. net filters, avoidance of ground loops).

Some external effects, such as errors due to electromagnetic disturbances, are, however, observed only in the measurements; their cause or source can then sometimes be identified or remedied, but only at great expense.

Countermeasures sometimes require careful consideration since they can also produce undesired effects.^[16] For example, sound-proofing hoods often have the disadvantage that they have a high thermal insulation effect (see example in Annex C). Consequently, heat sources (especially conventional lamps) should preferably be arranged outside the casing as far as possible; active vibration damping stages with external power supply and controllers should be used. As SPMs usually also have some remaining heat sources directly in or on the instrument, the temperature inside such casings will increase.

6.4 Summary

After completion of these preliminary investigations, the results should be included in a set of working instructions. These are, in detail:

- a) For measurement
 - instructions concerning the waiting times after the instrument is switched on
 - the procedure for specimen or probe change, repositioning and other modifications, and resulting waiting times
 - statements on the performance of prescans (prescan times) intended to contribute to the stabilization of the instrument for the measurement proper
 - the procedure in the case of deviations from the conventional conditions of measurement
- b) For installation or familiarization of the staff
 - the type of vibration damping/sound-proofing to be used

- the type of electromagnetic shielding to be used
- the ambient conditions to be observed (temperature/humidity range)
- the rules of conduct for the staff

These working instructions thus lay down the range of validity of the subsequent calibrations.

7 Calibration of scan axes

7.1 General

Calibration should be carried out using certified measurement standards. The results of the preliminary investigations in [Clause 6](#) should be taken into account when selecting the measurement standards best suited for the instrument in question in view of the measurement tasks to be performed.

These preliminary considerations also need to take account of the evaluation methods which will actually be available. The software supplied with the SPM normally differs significantly from manufacturer to manufacturer. The user is encouraged to use certified or at least validated software as far as possible, and to check any other software if it is applicable for the envisaged purpose. On the one hand, the procedures offered are in most cases inappropriately documented and, on the other hand, standardized procedures are usually not available, e.g. step height measurement in accordance with ISO 5436-1. This is why, in the following subclauses, reasonable alternatives are presented, if available, and their advantages and disadvantages discussed.

In most cases, different measurement standards are used for the individual calibration steps (see [7.3](#) to [7.5](#) and [Table 2](#)); alternatively, or additionally, 3D measurement standards can be used which, with suitable evaluation software, allow the calibration factors C_x , C_y , and C_z and the cross-talk between all three axes to be determined simultaneously (see [7.6](#) and [Table 2](#)).

NOTE A total of 21 deviations (or degrees of freedom) can be identified for the motion process of the SPM in analogy to coordinate-measuring machines.^[39] Complete separation and individual characterization is not possible with standard SPM equipment and tools available to the typical user, and often not practical. This International Standard therefore focuses on the calibration of the axis scales and cross-talk between the axes. It thereby takes the effects of many of the deviations into account, together with the instrument characterization in [Clause 6](#).

7.2 Measurement standards²⁾

7.2.1 Requirements for measurement standards

This subclause only gives general information about measurement standards; detailed statements on the requirements are contained in [7.3](#) to [7.6](#).

The properties of the measurement standards shall be documented and be accounted for in the calibration. For instance, the properties of grating standards that are usually used as lateral standards for SPM need to be documented following IEC/TS 62622. Important properties are

- the provision of a defined reference marking or reference field (e.g. in the form of a suitable mark or suitable coordinates) within which the measurements are to be carried out, or the provision of a larger field which is documented to be of sufficient homogeneity so that the measurements can be carried out at any point within this field,
- identification (manufacturer, kind of measurement standard, nominal or reference values, serial number),

2) The products mentioned in this subclause are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products, nor are they necessarily the best products available for the purpose in question.

- documented calibration values, including their uncertainty and how they are traced back to the SI unit of length, the metre, the measurement position, and the date and time of calibration, and
- an explicit statement of any regions with irregularities, scratches, contamination, etc., within the reference field.

Suitable sets of measurement standards are available from various manufacturers.[23][24][26][32]

Table 2 — Overview of guidance deviations of *xy*-scanner or *z*-scanner, measurement standards to be used and calibration measurements

Objective/measurement of	Measurement standard/requirement	Calibration: measurement procedure	Subclause
Cross-talk from the lateral movements to the <i>z</i> -axis <i>xtz, ytz</i>	Flatness measurement standard	Out-of-plane movement of <i>xy</i> -scan system	7.3
Rectangularity deviation <i>ywx</i>	2D measurement standard	Angle formed by the two axes, on orthogonal structures	7.4, 7.6
Rectangularity deviation <i>zwx, zwy</i>	3D measurement standard		7.6
Calibration of the <i>x</i> - and <i>y</i> -axis C_x and C_y , followed by determination of deviations <i>xtx, yty</i> (non-linearities)	1D or 2D lateral measurement standard	Pitch, rotation, linearity, and distortions	7.4, 7.6
Cross-talk from the lateral axes <i>xy, ytx</i>	2D lateral measurement standard	Pitch, rotation, linearity	
Calibration of the <i>z</i> -axis C_z , followed by determination of deviations <i>ztz</i> (non-linearities)	Set of step height measurement standards	Step height, linearity	7.5, 7.6

7.2.2 Handling of measurement standards

Like all sensitive specimens, measurement standards shall be stored and handled with care and very carefully protected from dust. Storage and handling of such objects should therefore always be dealt with in the user training. Avoidance of contamination shall be given preference over cleaning which would otherwise be required. If cleaning is nevertheless unavoidable, the relevant instructions of the manufacturer are to be observed. As a rule, contamination with nanoscopic particles is hard to remove without residues being left, and this might affect the function of the measurement standard. Should the surface undergo change due to cleaning, the measurement standard needs to be recalibrated.

It is further to be borne in mind that the user might also alter the quality of the measurement standard during assembly and mounting, possibly without being aware of it. For instance, when bonding or clamping measurement standards in place, it is therefore to be ensured that the mechanical stress in the measurement standard is kept as small as possible so as to prevent the measurement standard from bending.

7.3 *xy*-scanner guidance deviations of the *x*- and *y*-axes (*xtz, ytz*)

7.3.1 Definition of *xy*-scanner guidance deviations in vertical direction (*z*-plane)

Guidance deviations of the *xy*-scanner will in the following be understood as long-wave deviations (i.e. greater than 1/5 of the maximum scan range) from an ideal plane.

7.3.2 Measurement strategy

[Figure 6](#) shows the measurement strategy for the determination of the out-of-plane deviations *xtz* and *ytz*. This flow diagram can be divided into three sections. The left-hand column gives the basis for the procedure, the centre column the sequence of measurements, their analysis and the values determined.

The right-hand column gives information about the prerequisites for the measurement standard, for the measurements, for the analysis and lists the consequences to be drawn.

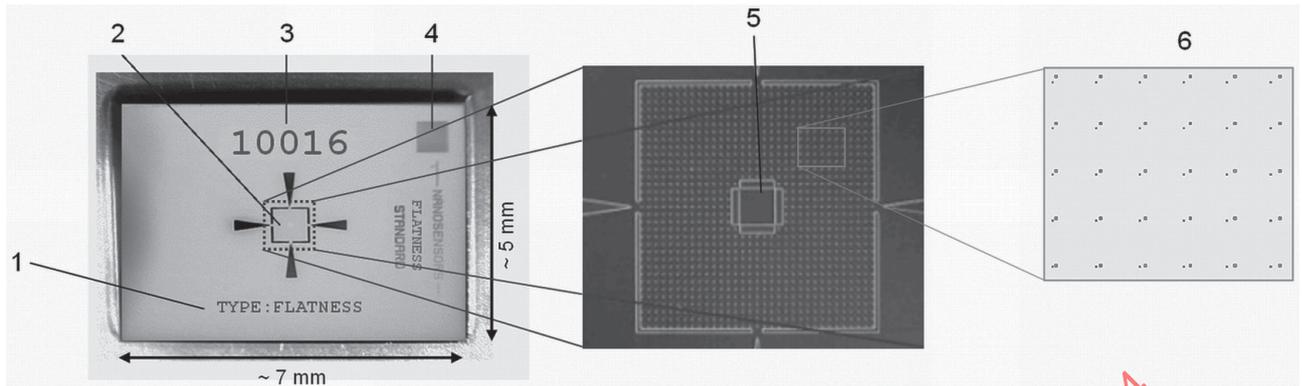
	Device characteristics	
Flatness	Measurement standards	Flatness standard $P-V < 10$ nm, smooth samples with small roughness $P-V, Rz, Ra, Rq (Sz, Sa, Sq)$
Measurement strategy	Measurement	Ambient conditions, scan conditions, scan direction (forward, backward), scan speed v , angle α
	Analysis	Data processing: levelling θ , rotation ϑ , filtering $\lambda_{c,s}$, averaging over several images (correlation length Λ)
Theory/model	Analysis	First-order least-squares fit for levelling
		Criteria: correlation length Λ of sample with respect to roughness
	Result x_{tz}, y_{tz} z -noise	Reproducibility? Measures: noise \rightarrow reduction of sources, reproducibility \rightarrow correction (polynomial fit)

Figure 6 — Flow diagram for carrying out a flatness calibration in accordance with Reference [35]

7.3.3 Flatness measurement standards

Figure 7 shows an example of a flatness measurement standard for scanning-probe microscopy. The four big arrows (left) are visible to the naked eye and help to coarse-position the measurement standard in the instrument. The inner area (right) shows double-dot structures which can be easily seen both under an optical microscope and in the SPM and indicate the direction towards the reference field in the middle. This reference field has been calibrated by interference microscopy.

Flatness measurement standards (see Figure 7) have a defined reference field whose surface shape, like its long-wave deviations, are traceably calibrated. For this purpose, interference microscopes are frequently used (see Figure 8). For the comparison, it is to be considered that the two procedures, interference microscopy and scanning-probe microscopy, usually show different transfer functions for the spatial wavelengths. The interference microscope has a limited spatial resolution so that very small details (i.e. small spatial wavelengths) cannot be imaged, resulting in higher SPM-measured roughness. P_t -values (unfiltered, i.e. without λ_c -filter) should be smaller than 10 nm.



Key

- 1 type of measurement standard (i.e., flatness)
- 2 central field with orientation aids and the reference field
- 3 serial number
- 4 orientation mark
- 5 reference field
- 6 further orientation aids with double-dot pairs pointing in the direction of the reference field

Figure 7 — Example of a flatness measurement standard for scanning-probe microscopy

7.3.4 Measurements

- Wherever possible, align the flatness measurement standard so that its plane lies parallel to the x - y -plane.
- Adjust the z -position of the scanner in such a way that the z -scanner operates symmetrically around the central position in the z -deflection range (see also [Figure 18](#)).
- Carry out several SPM measurements over the maximum range of the xy -scanner in the certified reference field, using the scan speed and scan direction normally used. To separate the characteristics of the measurement standard from those of the scanning system, the position of the flatness measurement standard should be varied at random several times between the measurements (shift > correlation length).

In general, at least nine measurements should be carried out.

7.3.5 Evaluation of results

- Carry out averaging over all the data sets, without making any tilt correction beforehand.
- This averaging furnishes significant information about the deviations of the scanner $z(x,y)$ from the ideal plane (xtz, ytz).
- Fit a polynomial $P(x,y)$ of appropriate order, with the maximum being of third order, into the averaged data set.
- Using the least-squares approach, fit a plane $E(x,y)$ to $P(x,y)$.
- The Pt -value of the difference between the two functions, $P(x,y) - E(x,y)$, is a measure of the guidance deviation xtz or ytz , respectively (out-of-plane deviation).

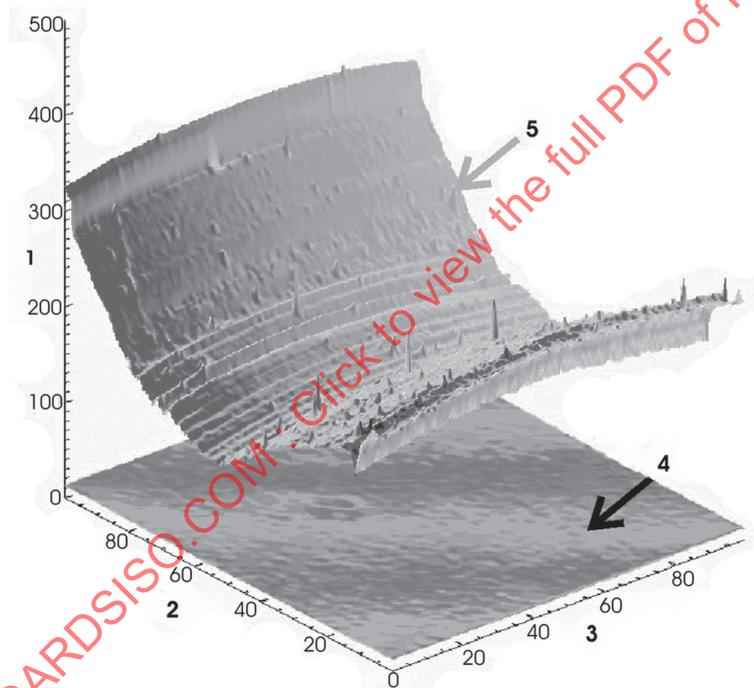
7.3.6 Summary

Good systems can reach $P-V$ values <10 nm for a scan range of $100 \mu\text{m}$. If the $P-V$ values vary by less than 20 % over a prolonged period of observation, the polynomial $P(x,y)$ can be used to correct measurements carried out under identical conditions (same scan range, same scan speed). The amount of variation in the $P-V$ value should be taken into account in the uncertainty budget.

7.3.7 Extended calibration measurements

Depending on the type of instrument, its stability, and its intended use, further investigations might need be carried out

- making separate measurements at different scan ranges and speeds,
- using another direction of scan (forward \Leftrightarrow backward),
- exchange fast (x -axis) and slow (y -axis) scan axis, and
- adjust the z -position of the scanner in such a way that the z -scanner operates, e.g. by 20% (see also [Figure 20](#)) above or below the central position in the z -deflection range (see also [Figure 18](#) and [20](#)).



- Key**
- 1 height, in nm
 - 2 y -axis, in μm
 - 3 x -axis, in μm
 - 4 measured by interference microscope
 - 5 observed by SPM

Figure 8 — Use of a flatness measurement standard to determine guidance deviations and noise

NOTE The lower plane shows the surface of a flatness measurement standard as measured with an interference microscope. The upper plane is the same region but observed with an SPM with a tube scanner. The deviations on the fast axis (x -axis) due to the scanner bow and individual peaks can be seen. In the direction of the slow scan axis (y -axis), interference and drift effects are obvious. By taking the difference, the deviations from an ideal guide plane can be determined.

7.4 Calibration of x - and y -axis (C_x, C_y) and of rectangularity (ϕ_{xy}) and determination of deviations (xtx, yty, ywx)

7.4.1 General

The calibration of the lateral scan axes is usually done with 1D or 2D lateral measurement standards. These show equidistant structures with defined features whose mean spacing (the pitch) serves to calibrate the lateral axes (see Figure 11). Local deviations are a measure of the non-linearity of the axis (xtx, yty), leading to distortions. In addition, the rectangularity deviation (ywx) and the cross-talk (xty, ytx) between of the lateral scan axes can be determined.[41]

In measurements on lateral measurement standards, not only the selection of the scan range but also the scan speed or rate are of decisive importance, as the calibration factors are strongly influenced by dynamic non-linearities and image distortions.[17][37] This is also true for systems of metrological category B1 with active position control. In the basic calibration, the scan speed shall therefore be adjusted as is usual for measurements on ordinary specimens.

For the extended calibration, carry out additional series of calibrations at different scan speeds, if appropriate. Depending on the scan speed and the scan range, calibration functions for image rectification can also be calculated.[37]

7.4.2 Definition of pitch p_x and p_y and rectangularity (ϕ_{xy}) in the x - y -plane

For definitions of grating properties, see IEC/TS 62622.

7.4.3 Measurement strategy

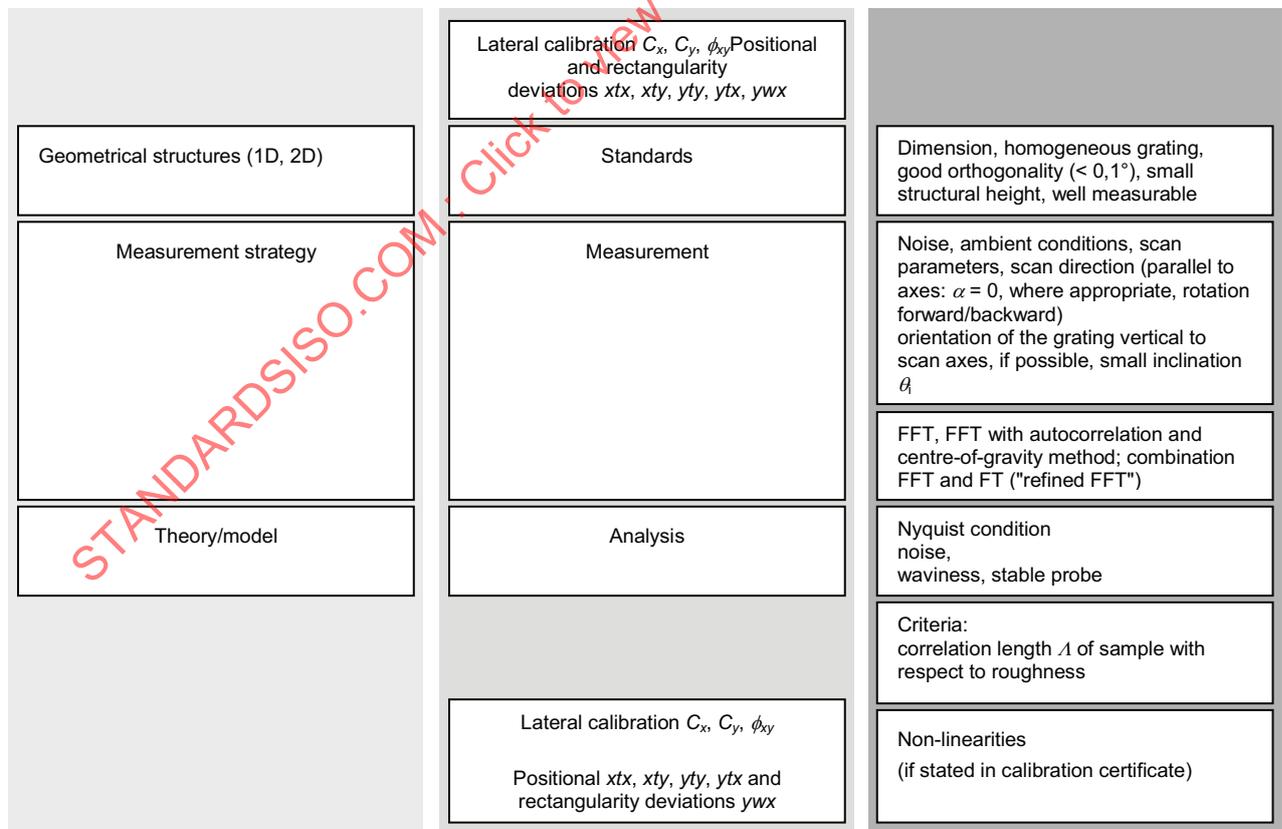


Figure 9 — Flow diagram of calibration of the lateral axes[35]

7.4.4 Selection of lateral measurement standards

Much valuable information on the selection criteria for lateral measurement standards can be found in IEC/TS 62622. In the following, only some criteria specific for SPM will be discussed as a help for the SPM user (see [Table 3](#)). Lateral measurement standards show regular periodical or two-dimensional structures with a known, traceable grating period and thus allow the calibration factors C_x and C_y to be determined for the x - and the y -axis. With 1D measurement standards the calibration factors can be determined only successively in two measurements, with the measurement standard being rotated by 90° between measurements. For two-dimensional structures, the angle, ϕ_{xy} , or the deviation from rectangularity of the structures can be determined. This also allows the orthogonality of the scan axes to be checked.

The periods of the measurement standards are selected according to the purpose of the measurement (see [6.1](#)) and the lateral lengths to be measured or the conventional scan ranges.

As a rule, to allow the non-linearities of the lateral axes (x , y) to be accounted for, a set of measurement standards with different periods (pitch values) is to be used.

If complete investigations of the distortions within the x - y -plane (straightness, rectangularity and possibly also angular errors) are envisaged, measurement standards with two-dimensional gratings are to be used. If axis calibration factors and distortions shall be determined only along the individual scan axes, it is sufficient to use a 1D measurement standard.[\[21\]](#)

Another criterion for the selection of the measurement standards is the evaluation method available (see [7.4.7](#)): For evaluation in the local space, as good an imaging (low-noise in particular) of the individual structures as possible is to be aimed at; the tip motion should be able to completely follow the surface modulation in order to reach a good signal-to-noise ratio. As a rule, at least seven pixels are necessary but preferably more than 10 pixels per structural period should be selected; where appropriate, the minimum number of pixels is, however, to be chosen higher depending on the shape of the structures, the shape of the tip and the interaction. Always several periods shall lie in the image (at least five) so that the structural spacing can be averaged over an adequate number of periods.[\[33\]](#)[\[34\]](#)[\[37\]](#)

Where an evaluation in the Fourier space has to be performed, the measurement standard used has to comprise many periods in the scan range envisaged, with the individual structures still being resolved. The sampling rate shall be sufficiently high, that each period contains at least five pixels.

For frequency-based evaluation, a finer structure or a higher resolution than for evaluations in the local space is thus generally to be selected. For Fourier-based evaluations, at least seven (but preferably 10 periods) shall be imaged. At the same time, it needs to be taken into account that in a measurement over a small number of periods the statistical uncertainty of the position of the individual structures (depending on the measurement standard type) can be rather great due to the design and that, as a result, averaging is to be performed over a sufficient number of periods with sufficient resolution. A system with pronounced distortions along its axes might not allow Fourier-based analysis.

Table 3 — Evaluation methods and their significance (++ very good, + good, o fair)

	C_x, C_y, a_{xy}	Non-linearities	Remarks
Manual image evaluation	o	No statement	<ul style="list-style-type: none"> — Normally only a few structures are used — Part of the data is not used — Depends on the user — Is time-consuming — Great uncertainty involved
FFT[31][33][34]	+	No statement	<ul style="list-style-type: none"> — The whole image information is used — Less sensitive to noise — Insensitive to punctual disturbances — Accuracy limited by mathematics
Centre-of-gravity method[31][33][34]	+	+	<ul style="list-style-type: none"> — Sensitive to noise, roughness, local disturbances (particles) and waviness of the specimen (use of a waviness filter possibly required) — Local structural defects detectable — Local scanner properties detectable
FFT + cross-correlation[37]	++	+	<ul style="list-style-type: none"> — Combines advantages of FFT and centre-of-gravity method

Furthermore, the kinds of irregularity in the lateral measurement standards, and the extent to which such irregularities are present should be considered when selecting suitable lateral measurement standards. In many cases, they show (relatively feeble) distortions produced during manufacture[25][33] or there are local discontinuities of the grating constant, e.g. so-called “stitching errors”. [30]. On high-quality measurement standards, such irregularities are so small that in the scanned image they are not directly visible to the naked eye, but can be proved to be present uniquely by specific methods. [30][33][34] In high-precision calibrations such as for category B instruments, they can, however, produce unintended errors.

When procuring the measurement standard, it should therefore be checked whether local irregularities can be avoided in measurements by careful navigation to intact regions, or whether irregular regions can actually be identified in the measured data afterwards and be accounted for in the evaluation process.

7.4.5 Basic calibration — Adjustments and measurements

In principle, calibration measurements should be carried out under the same conditions as are usually used when making measurements on ordinary specimens and using the same instrument settings, as the lateral calibration factors C_x and C_y are more strongly influenced by the measurement parameters than other calibration parameters. Attention should be paid in particular to the stable-state conditions referred to below; in addition to the instrument settings the scope and times of the prescan, for example, should be documented to ensure that subsequent measurements on other specimens are carried out in the same way and that comparability of the results is guaranteed.

Preparation:

1. Mount the lateral measurement standard in such a way that mechanical stress is minimized and preferably aligned such that
 - its plane lies as nearly parallel as possible to the x - y scan plane, i.e. θ_i is as small as possible, and
 - the grating is oriented as nearly perpendicular as possible to the scan axis/axes considered (1D measurement standard: lines vertical to the fast scan axis or, after rotation by 90° , vertical to the slow scan axis).

2. Then adjust the z-position of the scanner so that it operates symmetrically around the central position in the z-range (see also [Figure 18](#)).
3. Carry out all the measurements described below:
 - in the certified reference field of the measurement standard;
 - symmetrically around the central position of the ranges of the x- and y-scanners;
 - using the usual scan direction (forward/backward, fast scan direction parallel to one of the scan axes, angle 0°);
 - at the scan speed usually used for the scan range concerned.
4. Repeat the measurements using the maximum scan range under identical conditions until a suitably stable state is reached, i.e. until two successive measurements of the structural spacings are the same.

Calibration:

5. The last measurement from item 4 is taken as the calibration measurement.
6. Make measurements using smaller scan ranges, e.g. half, one-quarter or one-eighth of the maximum scan range. Depending on the stability reached in item 4, carry out at least one prescan to ensure that a suitably stable state is reached.
7. When using a 2D measurement standard, the following cross-check is recommended:
 - rotate the measurement standard by 90°;
 - use the fast/slow scan axis as before;
 - make two measurements over the last measurement range used in item 6;
 - compare the two measurements, as follows:
 - statement on the stability after repositioning of the measurement standard;
 - compare the measurements in item 7 with the last measurement made in item 6;
 - check whether identical calibration factors are obtained;
 - check the angles formed by the two scan axes.
8. When using a 1D measurement standard^[21]
 - rotate the measurement standard by 90°;
 - use the fast/slow scan axis as before;
 - repeat item 4 and item 6 to calibrate the slow scan axis.

7.4.6 Extended calibrations (scan speed, angle, and eccentric measurements)

In many cases, it will be beneficial for the user to also investigate quantitatively the dynamic behaviour of the scan system, as the scan speed frequently has a greater influence on the selection of the calibration factor than the size of the scan range.^[17]

Particularly for systems of category B, it is advisable to state the factors C_x and C_y as a function of scan range and scan speed and possibly to add the factor C_{xy} for the coupling between the two lateral scan axes. This ensures that the user reaps optimum benefit from the position sensors.

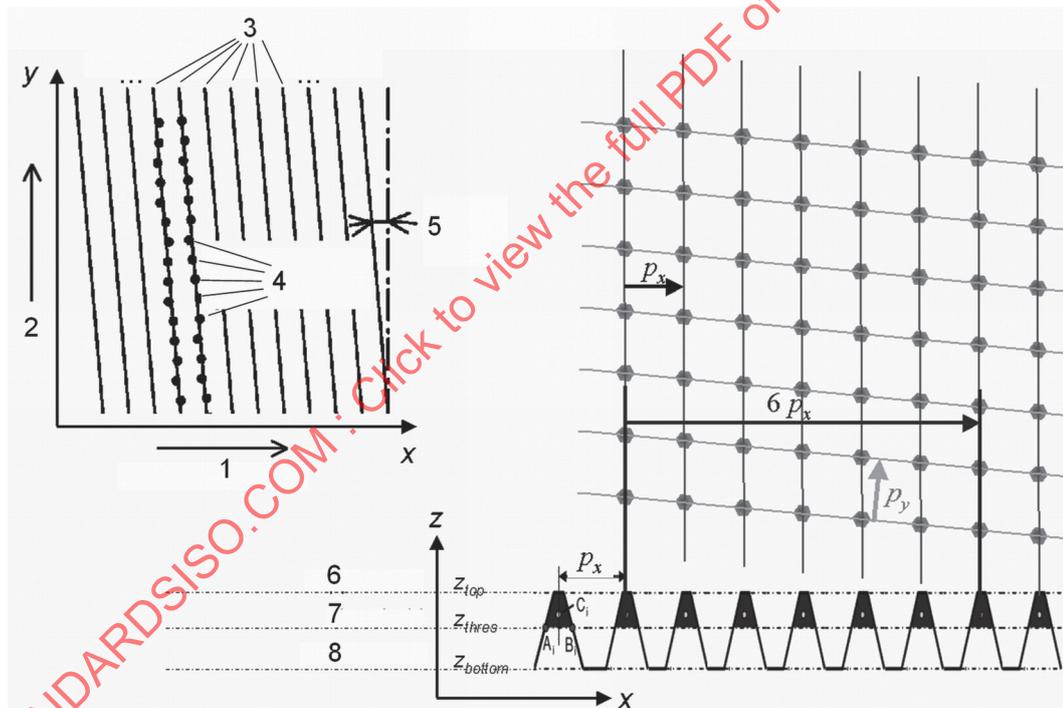
In many cases, the instrument or evaluation software supports the application of such corrections to the measurement data. With position-controlled scanners as are available commercially today, the

remaining uncertainty can thus be reduced to a few 10^{-4} so that it is smaller than that usually offered by the pixel resolution.

7.4.7 Evaluation

Different methods are used here. Table 3 gives the methods most frequently used as well as their significance as regards calibration factors and non-linearities.

1. After alignment along distinctive structural features (e.g. structure edges, peaks), the position of the individual structures can be determined
 - by the centre-of-gravity method in the local space (see Figure 10), [27][33][34] or
 - where Fourier-based methods are used, by cross-correlation. [37][40] This is done by calculating the cross-correlation of a grating element (mesh) cut from the total image with the total image, determining the centre-of-gravity coordinates of the resulting correlation peaks, and plotting the centre-of-gravity coordinates thus determined against the desired positions resulting from the respective reference value of the grating. In the case of 2D measurement standards, carry out these operations separately for the x -coordinates of the centres of gravity with p_x , and for the y -coordinates of the centres of gravity with p_y .



Key

- 1 fast scan direction
- 2 slow scan direction
- 3 rows of the 2D grating
- 4 centres of gravity of structures constituting the grating
- 5 angle of rotation (in this example, against the y -axis)
- 6 upper level
- 7 threshold
- 8 bottom level

Figure 10 — Evaluation in the local space

NOTE Application of a simple centre-of-gravity method by rotating the measured image until the columns of grating markings stand vertically in the image, followed by averaging over all lines of the measured image, and reading an integer multiple (here the sixfold multiple) of the measured grating period from the averaged profile. Note that the lower part of the figure shows the introduction of a threshold line (mathematically more complex) and determination of the centre of gravity of each grating marking.[33][34]

2. Carry out an orthogonality check (only if a 2D measurement standard is used)
 - in the local space: by alignment of two straight lines, each parallel to the grating directions and determination of the enclosed angle. For averaging, this is repeated in several regions of the image, and
 - in the Fourier space: by determination of the angle between the two vectors describing the grating.

The measured angles are compared with the reference value γ_{xy} of the measurement standard. Where provided for by the software, deviations should be compensated for by a calibration factor, taking into account that such a modification has effects on the calibration factors for the axes so that the calibration measurements in question (items 4 to 6) will need to be repeated.

3. Determine the calibration factors C_x, C_y in the local space, as follows:
 - Fit two regression lines g_0 and g_n through centres of grating structures in one column close to the left-hand and another column close to the right-hand edge of the image (see [Figure 10](#)) under the condition that both lines are parallel.
 - Raise a vertical s_x to the two straight lines g_0 and g_n .
 - Calculate the average pitch by determining the distance between the points of intersection of s_x with g_0 and g_n , and dividing by the number of enclosed grating periods n .

The quotient of the reference value p_{xref} and the measured value p_{xmes} of the grating period is the calibration factor C_x ; if need be, so-called cosine errors can be taken into account, i.e. a pitch or specimen inclination correction $\cos\theta_i$, for a pitch angle θ_i and a rotation correction $\cos\varphi_i$ if the straight lines g_0 to g_n form an angle φ_i with the slow scan direction.

C_y is determined in an analogous way to C_x , using regression lines through lines of grating structures close to the upper and lower image edges.

NOTE This method is, for simplicity, based on the first and last rows of structures only. Consequently, deviations of the positions of these two rows from their normal positions will affect the calibration result. This might be a disadvantage, as it needs to be noted that distortions are often particularly pronounced close to the edge of the scanned image, i.e. in these two rows. A more secure method that also takes into account the positions of all intermediate rows as described in [7.4.8](#) is therefore recommended.

4. Alternative determination of the calibration factors C_x, C_y in the Fourier space (see [Figure 11](#)).

Determine the measured mean grating periods by measurement of the position of the appurtenant peak in the FFT image. Bear in mind that a pure FFT possibly does not furnish the accuracy necessary. Many types of evaluation software therefore use a so-called refined FFT[33][34] or, subsequent to the FFT, calculate the cross-correlation of a unit cell of the grating with the total image in order to determine the centres of gravity of the resulting cross-correlation peaks in each individual grating mesh (see also [Figure 12](#)). These centres of gravity allow the structural spacings, and thus the calibration factors C_x, C_y , to be very precisely determined.[37]

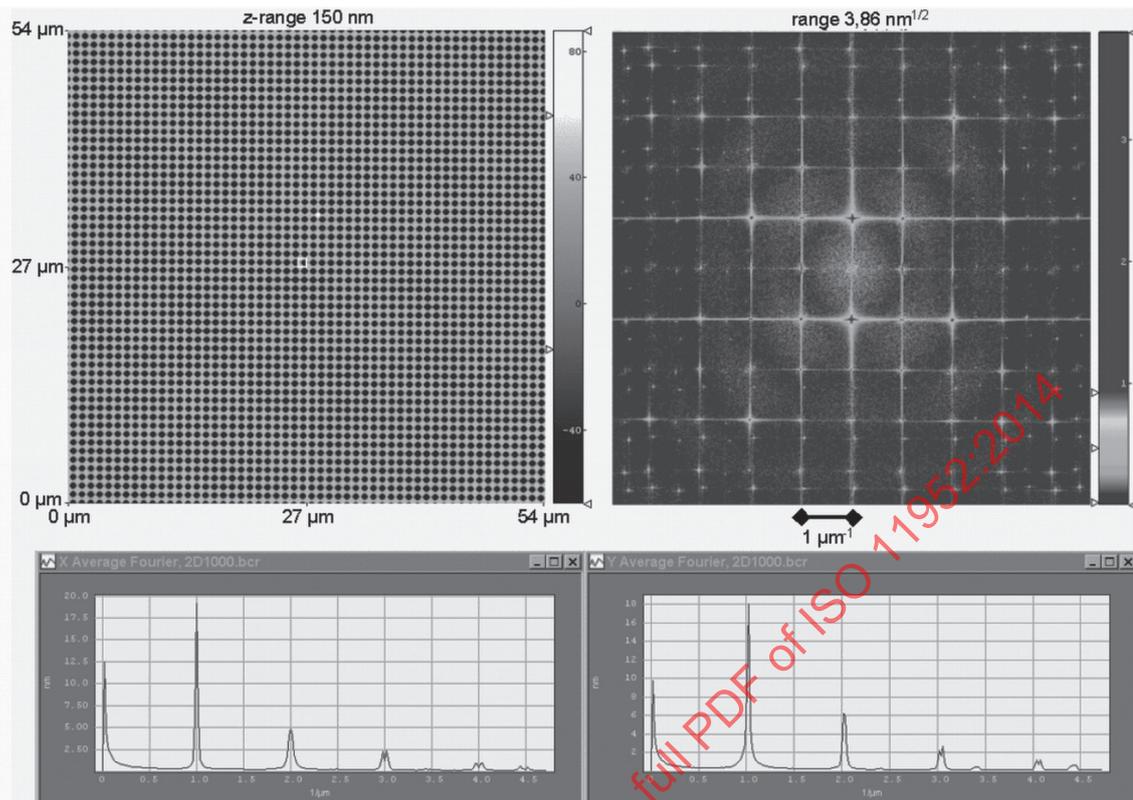


Figure 11 — Evaluation of a measurement on a 2D1000 measurement standard[37]

NOTE 1 Top left: AFM topography measurement, image size $54\ \mu\text{m} \times 54\ \mu\text{m}$. Top right: The appurtenant FFT analysis very clearly shows the peaks belonging to pitches p_x and p_y (each of nominal value $1\ 000\ \text{nm}$); from their centres of gravity, the mean measured grating period can be determined. Bottom: Averaging of the FFT measured by lines (left) and columns (right); by taking account of the error in the grating alignment with respect to the scan axes, the main FFT peak can be used to determine the measured mean pitches $p_{x\text{mes}}$ (left) and $p_{y\text{mes}}$ (right), respectively. For y , a slight deviation to the right of $1\ \mu\text{m}^{-1}$ can be seen and thus a pitch value somewhat below $1\ 000\ \text{nm}$.

7.4.8 Extended evaluations: non-linearity of the x - y -axis

Determination of the guidance deviations x_{tx} , y_{ty} (linearity of the axes).[18][37]

Prerequisite: A good grating has to be used for this, i.e. the standard deviation of the pitch value should be small. This standard deviation limits the determination of the non-linearity of the scanner.

- Fit parallel regression lines g_0 to g_n through centres of the grating structures in each column from the left-hand to the right-hand edge of the image (see Figure 10). The individual lines can be fitted to the points using the least-squares method.
- In good gratings, the mean values of the pitches of all the straight lines are a good approximation and should be used for further evaluation. If this is not the case, the parallelism of the straight lines is to be forced by fitting as above.
- Raise a vertical s_x to the set of straight lines g_0 to g_n .
- Determine the points of intersection S_0 to S_N of the vertical s_x with g_0 to g_n .
- Plot the measured points of intersection against the actual points as given by the reference value $p_{x\text{ref}}$ of the grating.

Fit a regression line $f(s_j) = ms_j + b$ through all the points of intersection j .

- The inverse of gradient, m , is the calibration factor C_x based on all structures used in the calculation.
- The deviation of the plotted positions of the regression line is a measure of the distortions xtx occurring along the x -axis. This plot further allows the distortion function to be approximated which in most cases can already be well described by a polynomial fit of the second or third degree (example in [Figure 15](#)).
- If the stability is appropriate, this polynomial can be used to correct the distortions.
- The deviations determined are to be compared with the scatter of the positions of the individual structures stated for the measurement standard in the calibration certificate. If the deviations determined agree within the scope of the uncertainty of measurement, the scanner behaves linearly.

To determine yty , in analogy to xtx , regression lines through lines of grating structures from the upper to the lower edge of the image are to be used.

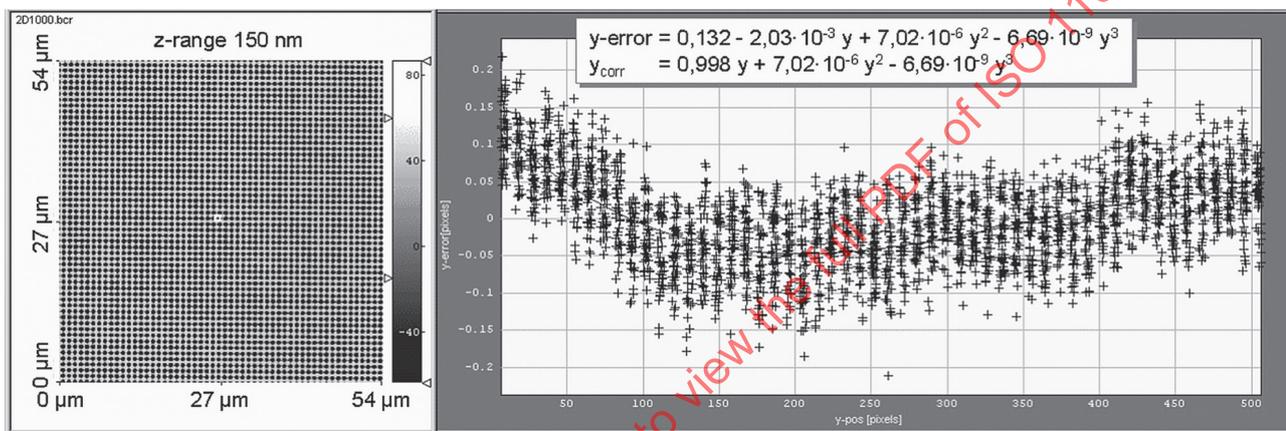


Figure 12 — Investigation of the non-linearity in the y-direction for the topography measurement of [Figure 11](#)^[37]

NOTE 1 Plotting of the “y-error” deviation of the measured y -coordinates of each grating marking (dimple) from the respective positions of an ideal undistorted grating with the same pitches. So up to about the 80th column (80 pixels from the left, y -position 8 μm), the measured y -coordinates lie somewhat too far to the left, in the middle of the image somewhat too far to the right, and finally again slightly too far to the left. This distortion can be approximated by a third-degree polynomial (upper formula and regression curve); it can be excluded to rectify the image (lower formula).

NOTE 2 In the case of clear deviations of the specimen temperature (e.g. in deep-temperature applications) from the reference temperature 20 °C in particular, for which the calibration of the measurement standard is valid, the thermal expansion is to be accounted for.

7.4.9 Summary

At the end of these investigations, the calibration factors C_x and C_y as well as the rectangularity ϕ_{xy} of the two lateral scan axes with respect to one another are to be retained, the selected measurement parameters such as scan ranges, scan speeds and pixel numbers being also to be stated.

Particularly for the initial calibration of an instrument, the calibration measurements should be so extensive that the user gets an idea of how strongly the calibration factors C_x and C_y and possibly also ϕ_{xy} depend on the measurement parameters. Based on this, the user can decide whether it might therefore be reasonable to repeat the calibration for different parameter settings. Also, a statement on the magnitude of distortions is to be aimed at in order to decide whether a systematic correction for distortion (as in [Figure 11](#)) is reasonable and necessary or whether the distortion is possibly to be included in the uncertainty budget for all lateral measurements.

7.5 Calibration of the z-axis C_z , ϕ_{xz} , and ϕ_{yz} , and determination of the deviations ztz , zwx , and zwy

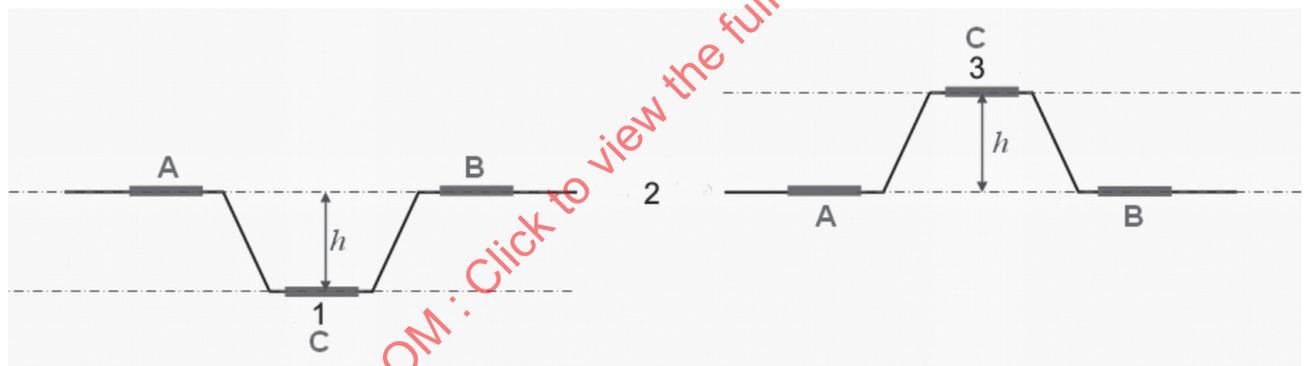
7.5.1 General

In many respects, the z-axis is calibrated in a similar way to the lateral axes, so the effort required is comparable. The tip shall follow the changes in the specimen topography which can be rather abrupt. Unlike lateral measurement standards, this requires fast reaction to deviations and high dynamics. Control parameters (such as proportional unit, P , and integration time, I) directly influence the dynamic behaviour of the SPM in the z-direction and, prior to the respective measurement, need to be thoroughly adjusted by, for example, analysing and carefully minimizing the error signal of the control.

The basic calibration relates to an operation of the z-scanner in which the latter is operating symmetrically around half its maximum range (Figure 14). Suitable series of calibrations for other setpoints of the z-scanner can be performed in an extended calibration.

It needs to be noted that contamination has even more serious consequences on step height measurement standards than on lateral measurement standards (Annex D). The exclusion of contaminated regions from step height analysis often cannot be done automatically and is thereby time-consuming. Furthermore, contamination is in practice usually the largest contribution to the uncertainty of step height calibrations.

7.5.2 Definitions of the step height



Key

- 1 groove
- 2 plane surface
- 3 elevation

Figure 13 — Cross-sections of two alternative types of step height measurement standards

NOTE 1 Left: Embodiment of the step height above an indentation, right: above an elevation with respect to the surrounding plane surface.

NOTE 2 For the evaluation only the measurement values for regions A, B, and C are to be used.

On a plane surface there is either an elevation (bar) or an indentation (groove, trench) whose middle part is also plane and, in addition, oriented parallel to the plane surface (Figure 13). In this way two plane-parallel planes are defined whose spacing embodies the step height. On both sides the step is surrounded by the surface. One sided steps are not suitable for z-calibration. For application, these planes are to be arranged in the instrument so that they are parallel to the x-y-plane; any remaining tilts are to be eliminated when the evaluation is started. Measurements are always performed across two complementary steps (i.e. either upwards-downwards or downwards-upwards combination) between the two reference planes. For single steps other procedures have to be used.^[40]

7.5.3 Measurement strategy

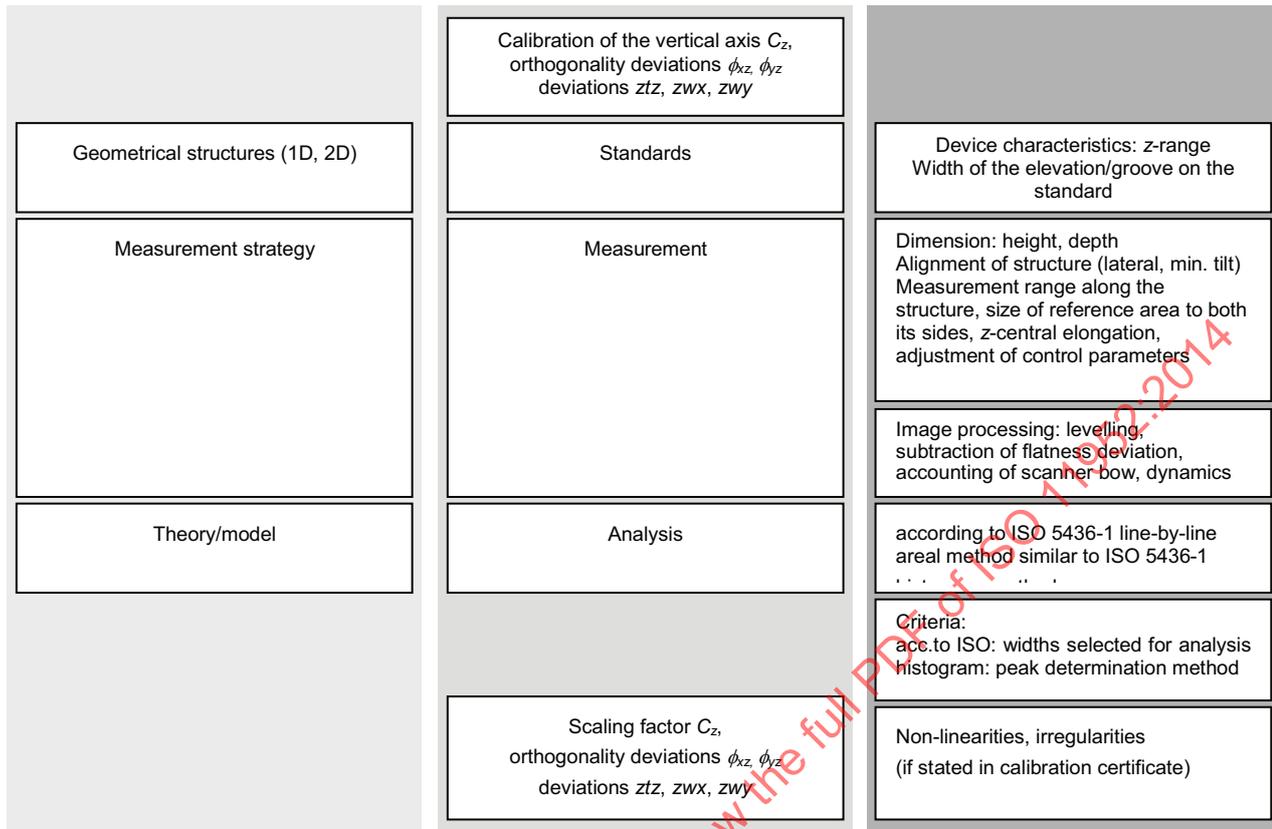


Figure 14 — Flow diagram for z-calibration[35]

7.5.4 Selection of step height measurement standards

The step heights of the measurement standards are selected as a function of the specific measurement purpose (see Section 6.1) and the heights and depths to be measured. For further characterization of the z-axis, a set of measurement standards with different step heights is to be used. Another criterion for the selection of the measurement standards is the lateral scan range of the SPM to be calibrated, which requires suitable structural widths of the elevation(s) and indentation(s) (recommendation: the scan range should be four times the structure width).

If the elevated or indented regions of the measurement standard consist of different materials, differences in the tip-specimen interaction can occur which give the deceptive impression of an additional topography contrast. It is therefore recommended to use measurement standards with the same material all over the reference field, or to have the measurement standard coated.

Furthermore, it cannot be precluded that the individual SPM indicates slightly different values for the step height depending on which mode of operation is used (e.g. “contact” or “non-contact”) and which setpoint has been selected for distance control. This can, for example, be a measurement artefact which is due to the design of the detection system.[20]

7.5.5 Basic calibration — Adjustments and measurements

z-position of the scanner: It is first to be adjusted such that the z-scanner operates symmetrically about the central position its z-range (Figure 15, left).

Lateral positioning: The structure to be measured on the measurement standard should be positioned in the centre of the x-y-scan range and preferably show no disturbing particles or defects.

Alignment: In the normal case it is advisable to align the line or the groove (in the case of measurement standards with one-dimensional structures) or one edge of the structure (for measurement standards with two-dimensional structures) perpendicular to the fast scan direction, as this allows any subsequently required line-by-line background subtraction to be most safely carried out.

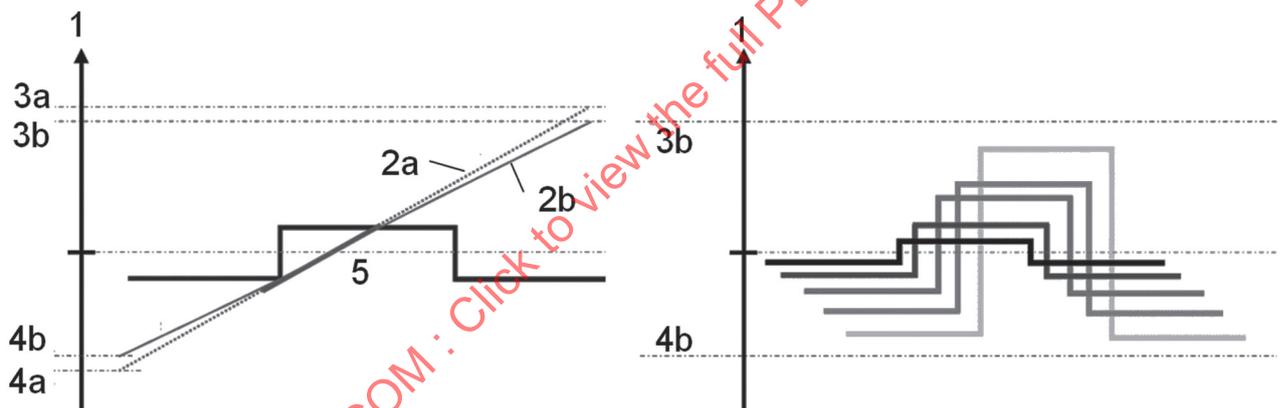
Levelling: The step height measurement standard should be so aligned that its surface preferably lies parallel to the x - y -scan plane.

Measurement values: It is to be ensured that a sufficient number of measurement values are obtained on the plateau of the elevation or on the bottom of the indentation. The range of measurement has to be at least four times the line or groove width w , respectively.

Adjustment of the control parameters and of the scan speed: The controller deviations (error signal) at the edges of the structure should preferably be small, i.e. overshoot and rounded images are to be avoided as otherwise the evaluation might be falsified. Similarly, phase jumps are to be avoided (see important note below).

Probe: For the measurements a conventional probe with a stable tip is to be used. Indications of potential changes in the tip shape, e.g. a conspicuous broadening of the measured structure widths are to be noted; if need be, the measurement is to be repeated with a probe of greater stability.

Disturbing influences of the detection system are to be noted (e.g. for optical detection systems: disturbing interferences which are due to reflections from the specimen surface).



Key

- 1 z-scanner elongation
- 2a extrapolated z-scanner behaviour based on measurement at one step height measurement standard
- 2b actual true z-scanner behaviour
- 3a extrapolated maximum position of z-scanner
- 3b actual true maximum position of z-scanner
- 4a extrapolated minimum position of z-scanner
- 4b actual true minimum position of z-scanner
- 5 calibrated range

Figure 15 — Calibration of the z-scanner about its mean deflection

NOTE Left: When calibration is carried out with only one step height measurement standard, conclusions regarding the behaviour of the z-scanner over its total deflection range are insufficient. Right: For a basic calibration a set of step height measurement standards is to be used whose reference areas are each symmetrical about the mean z-scanner elongation.

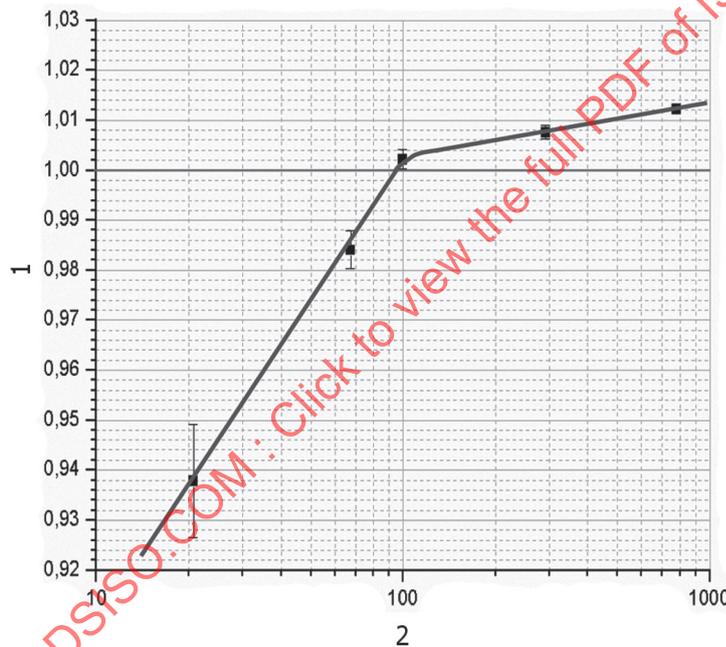
To account for the non-linearities of the z-scanner ztz , these measurements are to be carried out on a set of measurement standards with different step heights covering the deflection range of the scanner (Figure 18, right). It is thus possible to calculate individual calibration factors, $C_z(h)$ for the different

(step) heights, h .^[17] Considering the associated uncertainties (see 9.2), the calibration factors thus determined allow appropriate calibration factors to be determined by interpolation for heights lying in-between (Figure 19).

If the subsequent measurements on the user’s objects are also carried out about this central position, this basic calibration will be sufficient.

Important

When operating in “non-contact” or “intermittent” AFM mode, abrupt phase jumps can occur at the edges that translate into apparent, false topographic jumps. It is therefore mandatory to record, in addition to topography and error signal, the phase signal simultaneously, i.e. the phase difference between the alternating electric voltage applied to drive the cantilever in (or near) its resonance frequency, and the mechanical response of the cantilever as detected by the detection system. Abrupt jumps in the phase image are an indication for false heights in the topography image. Any images with abrupt larger phase jumps are to be excluded from step height analysis. Counter-measures: Sometimes a more robust operation of the cantilever (i.e. increase of drive voltage and thereby cantilever oscillation amplitude, or lowering of control setpoint) will help, but this in turn leads to stronger tip/specimen wear. Alternatively, a change of the cantilever is recommended.



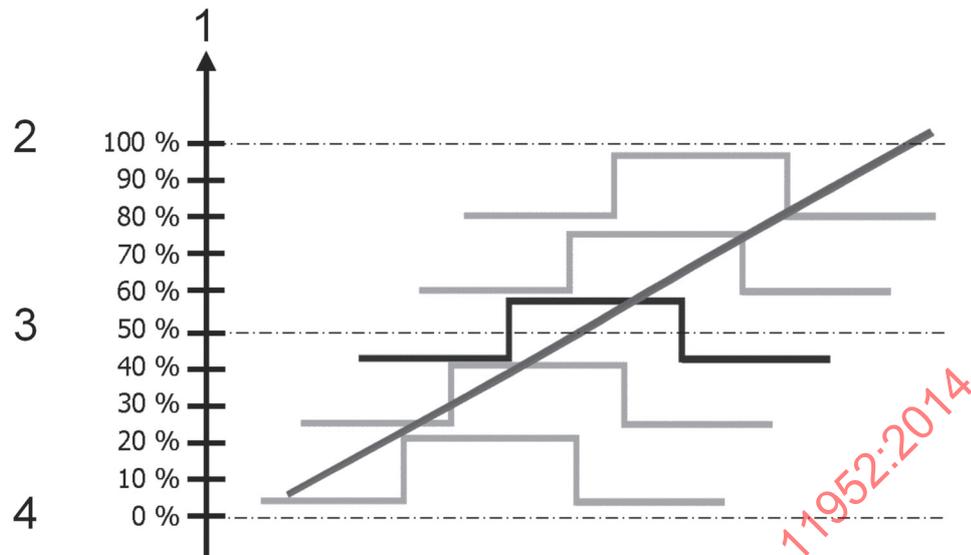
Key

- 1 z-calibration factor, C_z
- 2 reference step height/nm

Figure 16 — z-calibration factors, $C_z(h)$ determined with a set of step height measurement standards for five different heights measured symmetrically about the mean z-scanner deflection (example)

7.5.6 Extended calibrations

If the user actually intends to measure with the z-scanner operating around another mean elongation significantly differing from half elongation, further series of calibrations on the measurement standards need to be carried out in analogy to 7.5.5 around further positions in the z-deflection range (e.g. about 10 %, 30 %, 70 %, and 90 % of the maximum deflection, see Figure 17) to determine separate calibration factors C_z for the different z-deflections, and there each time possibly also for several step heights.

**Key**

- 1 z-scanner elongation (deflection)
- 2 maximum deflection
- 3 medium (central) deflection
- 4 minimum deflection

Figure 17 — Extended z-calibration

NOTE Repetition of the measurements at systematically varied z-deflection, here, for example, measurement about 10 %, 30 %, 70 %, and 90 % z-deflection, in addition to the basic calibration about 50 % deflection in [Figure 18](#).

7.5.7 Evaluations

Two different methods are suitable for data analysis: in analogy to ISO 5436-1 (see [Figure 18](#)) and the histogram method ([Figure 19](#)). For simple, smooth measurement standards, the two methods furnish similar values^[17] (for an example, see [Annex E](#)).

It is a prerequisite for the application of the two methods that the background which results from the inclination of the specimen ϕ_i and the guidance errors of the scanner is appropriately allowed for. The guidance errors can be eliminated subsequently if the measurements in question have been carried out under identical conditions on a traceable flatness measurement standard (see [7.3](#)). Otherwise, the results of the preliminary characterization of the measuring system ([Clause 6](#)) valid for the conditions of measurement selected here are to be taken into account when subtracting the background. The remaining inclination of the specimen ϕ_i is to be excluded by first-order plane subtraction.

Noise influences on the step height evaluation will be minimized in both methods if averaging over a great number of data points is carried out.

In step height evaluation, the handling of contaminations and wear phenomena is most critical; surface regions which have demonstrably undergone a change since the calibration of the measurement standard should preferably be excluded from the evaluation.

7.5.7.1 Profile evaluation in analogy to ISO 5436-1

This method has been derived from the step height determination in profiles performed according to ISO 5436-1 ([Figure 18](#)) such as it has been established long ago for contact stylus instruments. Therefore the procedure is line by line. It is based on the fit of two parallel straight lines in three user-defined areas A, B, and C of a scan line:

For the one straight line - besides the parallelism requirement - the determination uses only an area C in the middle of the indentation or elevation whose width can be selected by the user; it is usual to select one (according to ISO 5436-1) to two-thirds (Figure 18) of the total width w of the indentation/elevation.

Taking account of the parallelism requirement, the second straight is selected through two areas A and B which lie symmetrically about the indentation/elevation and usually show the same width as C. The distance of A from the left edge and of B from the right edge of the indentation/elevation is, as a rule, to be selected equal to the distance between C and the left or right edge, respectively, i.e. A and B lie symmetrically about the middle of the indentation/elevation and A and C lie symmetrically about the left edge and B and C about the right edge of the indentation/elevation.

As to the mathematics, the determination of the step height, h , is reduced to the calculation of only one regression line by appropriately shifting the points area by area by $+h/2$ and $-h/2$, respectively.

- For an indentation, the points are lowered by $h/2$ in areas A and B and raised by $h/2$ in area C.
- For an elevation, the points are raised by $h/2$ in areas A and B and lowered by $h/2$ in area C.

This line-by-line evaluation is to be carried out successively for many (normally all) lines (as indicated in Figure 18, left) to achieve a stable mean value for the measured step height, h . The variation of the step heights, h_i , of the individual scan lines provides information about the stability of the step height determination; it is to be accounted for in the uncertainty analysis.

Important

If the evaluation reveals that the plane area within the indentation/elevation is narrower than the selected width for area C, the width of C is to be selected so small that C lies completely in the plane area as otherwise the evaluation is not valid.

In analogy, the following holds for the distance of areas A and B from the structure edges: If offshoots of the edge transition or control artefacts such as overshoots project into area A or B, their distance from the edge is to be increased, or the width of A and B is to be reduced, until they lie completely in the plane area.

In many practical cases (e.g. often for steps with trapezoidal cross-sections), the widths for the sections in C on the one hand and for A and B on the other hand cannot be set identical. However, the widths in section A and B need to be identical to allow for a balanced analysis.

It is recommended to zoom into critical sections to better identify plane deviations and to reduce the widths accordingly. The selected widths for section C as well as for sections A and B are to be entered into the result report.

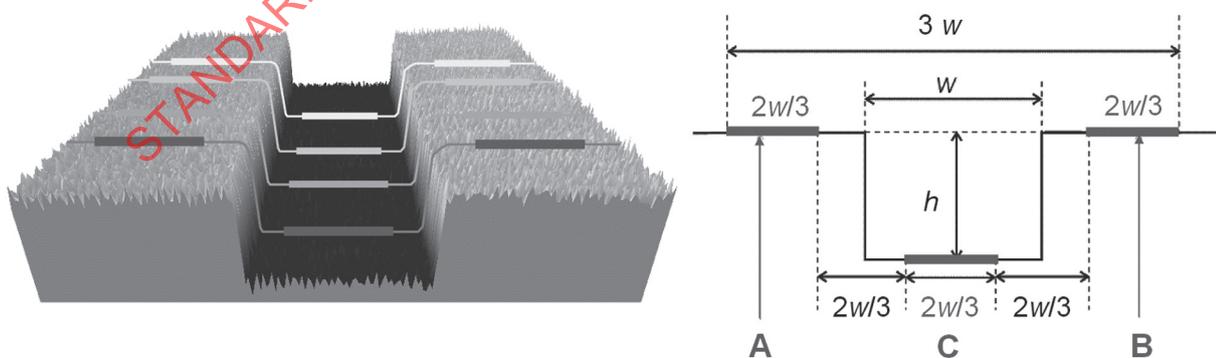


Figure 18 — Step height determination according to ISO 5436-1 (left: step height measurement standard 6 nm)

7.5.7.1.1 Alternative areal evaluation

If a line-by-line evaluation in analogy to ISO 5436-1 cannot be performed or can be performed only at unreasonably high expense, it is possible, as an alternative, to carry out an areal evaluation over the same three rectangular areas (Figure 19) which would be used in an evaluation successively carried out line by line. If need be, a line-by-line background subtraction is to be made prior to the calculation taking account of the preliminary investigations as regards the handling of line skips (Section 6).

It is also advisable not to include the first and last lines in the evaluation as the guidance deviations here are, as a rule, greatest.

To follow the procedure of ISO 5436-1 as closely as possible, a plane is to be fitted through areas A, B and C, the points being shifted - as in the fit of straights - area by area by $+h/2$ or $-h/2$, respectively. The general conditions — especially deviations from the procedure described here - are to be recorded.

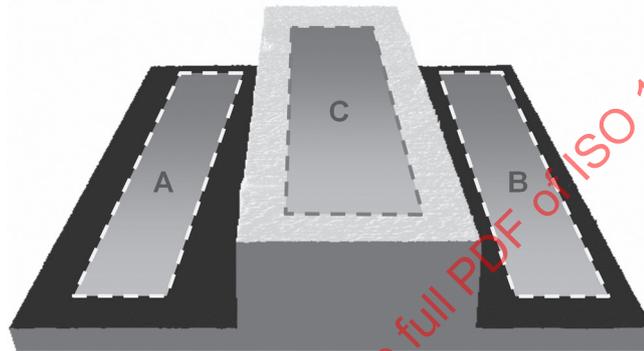


Figure 19 — Alternative areal evaluation in as close agreement with ISO 5436-1 as possible (Account is taken of the areas delineated by a dashed framing line)

7.5.7.2 Histogram method

The histogram method comprises a representation of the frequency distribution of the measured height values. For step height measurement standards, two peaks are thus obtained: one with the height values of the substrate and one with the height values of the step (indention/elevation) itself. These peaks shall be identified to determine their respective centres of gravity. For the determination of the centre of gravity of each of these two peaks, it is expedient to set a lower threshold value (e.g. the half value) in the frequency distribution in order to minimize the influence of outliers and any irregular edges (Figure 20). The difference of the height values determined for both centres of gravity correspond to the step height, h .

A careful background subtraction as described at the beginning of 7.5.7 is absolutely necessary before the histogram is made as otherwise the histogram peaks are broadened or split and the step height value determined is incorrect.

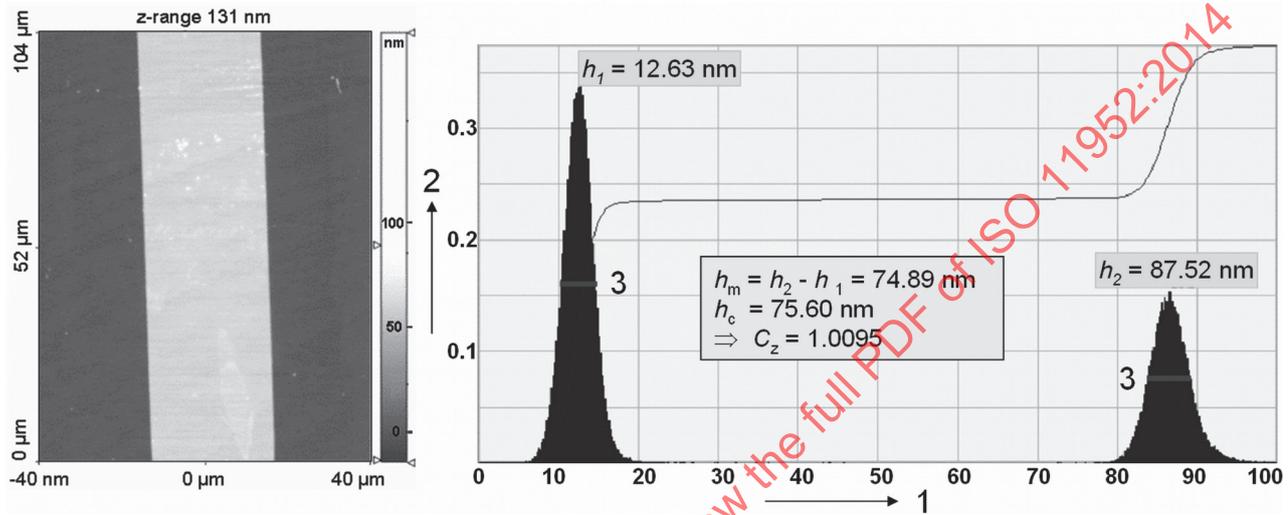
7.5.7.3 Comparison of the evaluation methods

In practice, the selection of the evaluation method depends in most cases on the evaluation procedures available or easier to realize and possibly also on the shape of the indentation/elevation on the measurement standard used.

Compared to the histogram method, evaluation according to ISO 5436-1 has the advantage that the edges themselves are not accounted for. Apart from edges that preferably show contaminations and roundings, control errors at the steep transitions are substantially greater than in the plane areas and sometimes produce non-symmetrical histogram peaks. A tilt of the measurement standard which has possibly not been subtracted completely has no significant influence on the result. As experience has shown, evaluations according to ISO 5436-1 have a tendency to show smaller deviations than evaluations by the histogram method (Annex E). A drawback of the ISO 5436-1 method is that it is rather sensitive to

contamination. It is therefore recommended to exclude contaminated regions carefully and/or exclude whose lines from further consideration that show a step height significantly deviating from the most frequent step heights determined for cleaner lines.

It is an advantage of the histogram method that it can be applied in the same way when step height measurement standards with two-dimensional structures with plateaus of any shape (i.e. including shapes with non-straight edges) are used, whereas the application of ISO 5436-1 is restricted to one-dimensional structures (line, groove) or two-dimensional structures with straight edges parallel at least in one direction. Particular care is, however, to be taken to ensure as complete a background subtraction as possible, as a tilt of the measurement standard as well as potential guidance deviations falsify the result directly.



Key

- 1 height, in nm
- 2 frequency of the measured height values
- 3 thresholds

Figure 20 — Histogram method (example) — Setting of thresholds to half-value height, determination of the centre of gravity above the threshold

NOTE The continuous curve is the integral of the frequency distribution. h_m measured step height, h_c reference value from the calibration certificate of the measurement standard.

7.5.8 Summary

At the end of the investigation of the z-axis, a set of calibration factors, $C_z(h)$, for measurements at different heights is available, operation taking normally place symmetrically about a mean deflection of the z-scanner. For the interpolation of the calibration factor to be applied, a diagram similar to that in [Figure 16](#) can be set up.

7.6 3D measurement standards for alternative and extended calibration

7.6.1 General

As an alternative or, where applicable, in addition to the lateral and step height measurement standards described in [7.4](#) and [7.5](#), 3D measurement standards can be used.^[41] They allow all three scan axes to be calibrated simultaneously but require an evaluation software specifically created for this task.^{[42][43]} Basically, this type of measurement standard is even suitable to determine some additional calibration factors which, using the measurement standards described in [7.4](#) and [7.5](#), cannot be determined at all or only at great expense.

At the end of this section, the advantages and disadvantages are dealt with in detail.

7.6.2 Requirements for 3D measurement standards

3D measurement standards show simple geometric bodies (usually pyramids) on their substrate surface. They serve as supporting structure for markings on different planes of the multi-step pyramids. The supporting structures can either be positive (elevations, see e.g. [Figure 21](#)) or negative (i.e. groves), or both. While the heights and widths of the supporting structure are not meant as measures for calibration, the markings act as reference points for this kind of calibration and will in the following be referred to as nanomarkers.

The nanomarkers need not lie in a regular grating or at discrete step heights, respectively, as the three spatial coordinates of each nanomarker are used for the calibration (so-called landmark-based calibration). For this procedure a clear nomenclature is needed allowing each individual nanomarker to be uniquely identified.

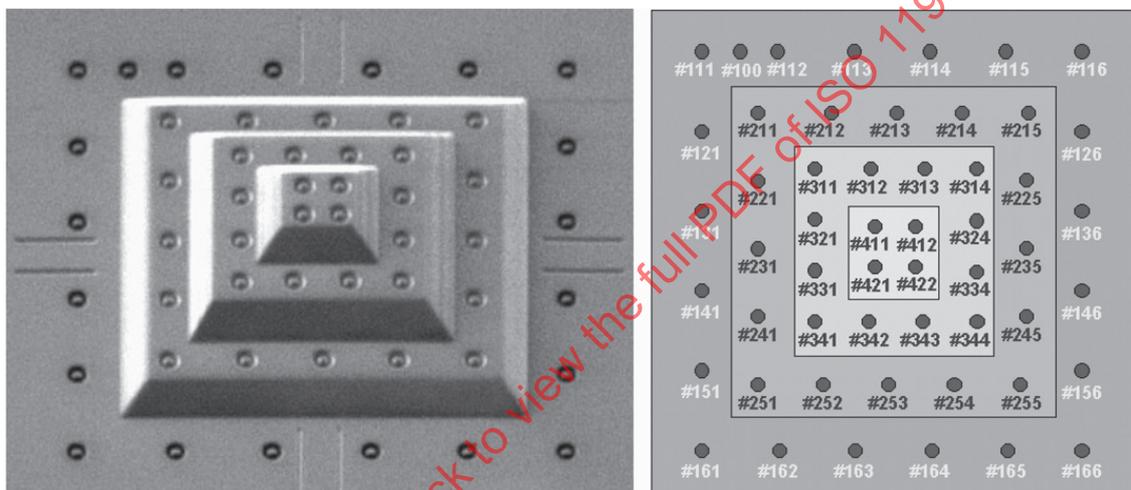


Figure 21 — Example of a 3D measurement standard SEM micrograph

NOTE Left, size: approx. $24\ \mu\text{m} \times 24\ \mu\text{m}$, height of pyramid approx. $3\ \mu\text{m}$, i.e. about $1\ \mu\text{m}$ per step and nomenclature of the punctiform nanomarkers (right).

The calibration of the SPM takes place by comparison of the coordinates measured for each nanomarker with the respective reference data which have been determined in the (certified) calibration of the measurement standard.

From these statements, the following requirements are obtained for 3D measurement standards:

- substrate surface equipped with one or several bodies tapered upwards and preferably with smooth side faces and without sharp edges (so as to avoid control problems during scanning as far as possible and allow the probe to easily follow the shape of the body);
- substrate surface and body from the same material (to avoid material-induced contrasts, with cross-talk to the measured height value);
- bodies shall show several (largely) flat areas at different height levels above the substrate surface in which the nanomarkers are located;
- nanomarkers shall lie at an appropriate distance from the edge of the flat areas referred to, have a characteristic shape (e.g. indentation in the form of a hole, a cross or a ring) differing clearly from other structures to be found on the surface, and be very homogeneous in their form over the entire measurement standard (ideal case: congruent).

It shall be possible for the measurement standard with its bodies and nanomarkers to be uniquely mounted as regards its orientation (unique markings/shape for left-right and top-bottom orientation), or the evaluation software needs to automatically determine the orientation of the measurement standard in the instrument by asymmetric features in its reference field.

The measurement standard shall be distributed or made available together with the following documents:

- nomenclature for the nanomarkers (unique designation of each nanomarker);
- for each nanomarker, all three reference coordinates shall be available (calibration values for each nanomarker), at least in a digital data format supported by the specific evaluation software;
- the method used in the calibration of the measurement standard to determine the reference coordinates shall be stated or described, respectively;
- form and size of the nanomarkers shall be stated.

For the evaluation, a specific evaluation software distributed or made available together with the measurement standard and the reference data can be used.

7.6.3 Selection of the 3D measurement standards

- The reference surface or body with nanomarkers should preferably be entirely covered by the lateral scan range of the SPM. It needs to be noted that the method performs best if all nanomarkers are measured. Consequently, the reference field should be slightly smaller than the maximum scan range to allow for navigation/positioning inaccuracy.
- The height of the bodies with nanomarkers shall not exceed the maximum z-deflection range of the scanner; furthermore, it is to be ensured that the measurement standard can be positioned in the z-deflection range so that both elevated and indented areas are actually accessible for the probe (question of offset adjustment for specimen/probe holder).
- The nanomarkers should be covered with a minimum number of typically 9×9 pixels when the necessary reference range is scanned (question of maximum number of pixels and their conversion to the necessary size of the range of measurement). Certain shapes of nanomarkers might require a higher number of pixels for safe evaluation; this should be indicated in the manual of the measurement standard.
- It shall be possible for the measured data to be reliably converted to a format supported by the specific evaluation software; if need be, conversion programs are also to be obtained.
- The evaluation software shall have been certified or have otherwise been provably tested for correct function.

7.6.4 Carrying-out of the basic calibration

- To avoid influences by wear of the tip: mounting of a probe with dimensionally stable tip which is still sharp enough (possibly already slightly used so that the phase of initial wear is already over).
- Mounting and alignment of the 3D measurement standard ensuring that the reference range is completely covered by the following scan. The specimen shall be positioned in such a way that both elevated and indented areas are actually accessible to the tip (suitable offset adjustment of specimen/probe holder); if need be, the approach is to be repeated in an area of the specimen lying at mean height.
- Adjustments for measurements: A sufficient number of pixels (at least 9×9 pixels per nanomarker) are to be ensured; as a rule, the greatest number of pixels is to be selected. Measurement with the conventional scan parameters (control parameters optimized, scan speed as otherwise usual).
- If need be, a prescan is to be made, i.e. rejection of measurements performed under the influence of initial drifts until the instrument is stable according to the investigations of [Clause 5](#).

- The valid measurement is to be repeated at least three times. Images with sudden jumps are to be excluded from further analysis. Furthermore, it is recommended to calculate the height differences between subsequently recorded images to identify smaller jumps (e.g. due to pick-up of contamination particles) or remaining drifts and eliminate these images as well.
- Measurements to be repeated also for smaller scan ranges: repetition of the points above. The minimum possible scan range is reached when less than 25 nanomarkers lie in the scan range or when the distribution of the nanomarkers in the scan range is no longer homogeneous.

7.6.5 Evaluation of the measurements

The evaluation of the measured data is carried out using specific software furnished or already available which supports the subsequent evaluation steps. These steps thus also represent minimum requirements for the evaluation software.

If required for the evaluation, the measured data are first to be converted to a data format supported by the special software.

- Reading-in of the measured data into the nanomarker search program. This program serves first of all to determine the measured lateral coordinates of the nanomarkers which includes the following:
 - marking/elimination of nanomarkers in the image which are damaged or contaminated;
 - extraction of a zoom cutout with a nanomarker from the total image with the nanomarker having to lie in the middle of the cutout with subpixel accuracy; or use of an artificial template image of a nanomarker exactly centred in the template image;
 - cross correlation of the cutout with the total image;
 - setting of a threshold value for the cross correlation peaks, determination of the centre of gravity within the correlation maxima thus outlined, writing of the lateral coordinates into a table/database stating the designation of the nanomarker;
 - determination of the z-coordinate of each nanomarker: according to the form of the nanomarker, the accompanying documents, or the specific program directly, contain the definition of the z-value; determination in the same way as stated for the reference data.
- Comparison of the measured nanomarker coordinates with the reference data set
 - Alignment of the data sets: The program compares the two data sets and translates and rotates the measured data set such that optimum agreement with the reference data is reached (six parameters: three translational, three rotational ones).
 - Admission of three further parameters: C_x , C_y , and C_z (calibration factors for the three axes), another fit in which the values for C_x , C_y , and C_z are adjusted such that the deviations between the measured coordinates thus corrected and the reference coordinates are minimized; the program also states the mathematical uncertainty contribution.
 - Repetition of the evaluation for all three images measured under identical conditions; check whether the calibration factors are in agreement within their uncertainty; in the affirmative: the calibration factors are valid; in the negative: the measurements are again repeated three times until stability is reached, or a substantially greater uncertainty of measurement needs to be stated.
 - Evaluation of the images with smaller scan range.

At the end, a set of calibration factors for the individual scan ranges (at the selected scan speed) is available.

7.6.6 Extended calibrations

Measurements

The calibration measurements can be repeated for different scan speeds — in analogy to calibrations against lateral measurement standards in order to determine the dependence of the axis calibration factors on this parameter.

If the bodies on the measurement standard do not fill the entire z -range of the scanner, the scanner as described for pure step height measurement standards, can be positioned that the measurements are recorded at different z -deflections of the scanner. This allows non-linearities of the z -scanner to be accounted for.

Evaluations

It is to be checked whether the available data sets can be brought into significantly better agreement with the reference data by introducing further correction parameters. These are in their order of suitability including

- cross-talk of the lateral scan axes with one another (xty, ytx),
- cross-talk of the z -axis to lateral scan axes (ztx, zty), i.e. check to what extent the scanner swerves off in z -movements in the lateral directions, and
- introduction of axis calibration factors of higher order (to compensate for distortions).

These additional correction parameters can be uniquely determined only if a greater number of nanomarkers lie in the image. Furthermore, their introduction is appropriate only if they allow clearly better agreement with the reference data to be reached systematically, i.e. for all data sets to be taken into account.

7.6.7 Advantages and disadvantages of the 3D measurement standard

The use of the alternative type of measurement standard entails the following advantages and disadvantages.^[41]

Advantages:

- simultaneous determination of all three axis calibration factors C_x , C_y , and C_z ;
- coverage of the entire 3D measuring volume by distributing nanomarkers as well as possible over the entire lateral and vertical scan range;
- as a result, object to be calibrated closer to conventional measurement tasks on 3D topographies;
- Practically, the only type of measurement standard allowing the non-orthogonality of the z -axis with respect to the x - y -plane to be easily determined (ztx, zty);
- easy standardized handling and operation, as these are possible only with special software which carries out the evaluation for the user and largely precludes user-induced errors;
- application and evaluation less sensitive to contamination, as only contaminations of the nanomarkers can have disturbing effects — however, this holds true only if the contamination does not lead to sudden tip jumps;
- contaminated/damaged nanomarkers can be excluded from the evaluation without the result being falsified.

Disadvantages:

- requires, as a rule, additional specific evaluation software;

- newer methods, therefore not included in most of the available analysis software otherwise used in SPM.

8 Report of calibration results

The result report (for pattern, see [Clause 10](#)) contains the following statements on the calibration of the instrument:

- instrument that has been calibrated (manufacturer, type, serial number, inventory number);
- metrological category of the instrument (A, B1, B2, C);
- date of last calibration.

Equipment used

- kind and type of measuring head and scanner, including scan range (x-y-z) and relevant metrological category
- cantilever type used
- mode in which the calibration has been carried out
- specimen stage used
- measurement standards used
- evaluation software used

Statements on ambient conditions

- place of measurement (air-conditioning system, clean-room conditions, interference from neighbouring instruments)
- details of, for example, vibration damping system, protective hood, electromagnetic shielding
- temperature (for measuring chamber: comparison of inside/outside temperature)

From [Clause 5](#): Preliminary investigations

- waiting times after instrument is switched on (temperature regulation especially when measuring chambers are used, e.g. sound proofing hoods)
- procedure for specimen/probe changes, repositioning, and other changes as well as resulting waiting times
- details of the performance of prescans (prescan times) intended to contribute to stabilizing the instrument for the measurement proper
- procedure in the case of deviations from the conventional conditions of measurement

Furthermore, the result report contains the following measurement results:

- drift ([5.2](#));
- z-noise Rqz and Sqz (if determined);
- x-y-noise Rqx and Rqy (if determined).

From [Clause 5](#): Calibration — Details of measurement standards, scan range, and scan speed

- flatness: $P-V$ or Pt , respectively, third-degree polynomial ([7.3](#))
- flatness deviations: cross-talk xtz , ytz ([7.3](#))

- rectangularity of the lateral axes ywx (7.4)
- where applicable: rectangularity of the z -axis with respect to the lateral axes zwx, zwy (Section 7.6)
- calibration values C_x, C_y, ϕ_{xy} , and non-linearities of the lateral axes xtx, yty (Section 7.4)
- calibration value C_z and non-linearities of the z -axis ztz (Section 7.5)
- where applicable: further calibration values from measurements on 3D measurement standards (7.6)

Further statements

- performance of the basic calibration
- date of performance of the basic calibration
- unusual features, remarks regarding the internal quality management system

9 Uncertainties of measurement

9.1 General

Calculate the uncertainty of measurement in accordance with the “Guide to the Expression of Uncertainty in Measurement” (GUM) (ISO/IEC Guide 98-3). Take account not only of the relevant instrument characteristics and of the particular measurement details including the set-up employed but also of the properties of the measurement standard used, of the object to be measured and of the evaluation method applied.

To illustrate the procedure, the relatively simple estimation of the uncertainty in the determination of an unknown step height will be elucidated.

A more ample analysis of the uncertainty contributions is given in [Annex F](#), using the determination of lateral measurands as an example.

9.2 Vertical measurand (height and depth)

In the following the determination of an unknown step height will serve as an example. By repetition of the measurement it is possible to reduce uncertainty contributions which are based on random errors while systematic errors persist. Assume that the instrument has been calibrated against a step height measurement standard whose traceable step height, h_c , with its expanded uncertainty U_{95} is known (value as stated in the calibration certificate, not the nominal value). Assume further that the measurement standard has been selected such that the value of its step height corresponds more or less to that expected for the unknown step height.

For the uncertainty calculation a suitable model is to be selected; in our case, it is given by Formula (1):

$$h_x = \frac{h_c}{h_{cm}} \cdot h_{xm} + \sum \delta h_i \quad [1]$$

where

h_x value of the unknown step height;

h_c value of the step height of the measurement standard as stated in the calibration certificate;

h_{cm} measured value of the step height of the measurement standard;

h_{xm} measured value of the step height of the unknown object to be measured;

δh_i other contributions (to the uncertainty of measurement).

The other contributions stem from errors such as stability and quality of the measurement standard, remaining non-linearities of the scanner not to be systematically allowed for, cross-talk between axes, roughness and inhomogeneity of the object to be measured, evaluation method, drift effects during the measurement etc. At temperatures slightly differing from 20 °C, the thermal expansion can be neglected for step heights below 1 µm (for conventional materials).

The standard uncertainty, $u_c(h_x)$, can thus be calculated using Formula (2):

$$u_c^2(h_x) = u^2(h_c) + u^2(h_{cm}) + u^2(h_{xm}) + u^2(\delta h_{\text{non-lin}}) + u^2(\delta h_{\text{cross-talk}}) + u^2(\delta h_{\text{eva}}) + \dots \quad [2]$$

assuming that h_c approximately h_{cm} , approximately h_{xm} . The expanded uncertainty, U_{95} , is to be calculated from $u_c(h_x)$ by $k = 2$ for a Gaussian distribution. For detailed uncertainty considerations it is absolutely necessary to better know the instrument characteristics. See relevant literature. [27][29][31]

10 Report of results (form)

Report on the results of SPM axes calibration

according to ISO 11952 Surface Chemical Analysis – Scanning Probe Microscopy – Calibration of SPM scan axes at the highest level

General specification of the instrument							
Instrument (manufacturer, type, serial number, inventory number or similar, year of manufacture, lab location)					Metrological category <input type="checkbox"/> A <input type="checkbox"/> B1 <input type="checkbox"/> B2 <input type="checkbox"/> C <small>ref.SPM closed-loop open-loop no sensor</small>		
Date of previous calibration		Modifications of instrument and changes to the environment since the previous calibration					
Type of sensor head, type of probe-sample interaction detection (e. g. beam deflection, glassfiber interferometry, piezolever)							
Sample stage / sample mounting		Type of coarse positioning lateral		Type of coarse positioning in z		Probe type, scan mode (c, nc, etc)	
Ambient conditions and shielding							
<input type="checkbox"/> air-conditioning <input type="checkbox"/> cleanroom <input type="checkbox"/> UHV-chamber	Vibration damping, acoustic shielding, electro-magn. shielding, etc.			of lab	Temperature of chamber	of sample Humidity	
Detailed specification of scanner equipment for all 3 directions (chapter 5)							
Scanning Sample/Scanning Probe, actuator (e. g. piezo tube, piezo stack), nom. scan-range, metrolog. category (type of position sensor)							
x-axis							
y-axis							
z-axis							
Results of the preliminary characterisation (chapter 6)						Date	Time (from-until)
Procedure (e. g. idle time) when a) changing the probe b) changing the sample c) aligning d) opening / closing the measurement chamber							
remaining drift (after the specified idle time) lateral (ISO 11039) vertical (ISO 11039)		z-noise <i>S_{qz}, R_{qz}</i>	lateral noise <i>R_{qx}, R_{qy}</i>	Prescan-time before x-/y-calibration	Prescan-time before z-calibration	Other data	
Results of the flatness investigation (chapter 7.3)						Date	Time (from-until)
Standard (artefact) used (manufacturer, type, serial number, calibration certificate: number/date)				Software and method used for analysis			
Scan-range	Scan-speed	<i>P_t</i> or <i>P_v</i>	Approximation of the flatness deviation (e. g. polynomial of 3rd order), remarks				
Results of the lateral calibration x-axis and y-axis (chapter 7.4)						Date	Time (from-until)
Analysis method (e. g. gravity centre method automatised/manual, FFT-method, combined methods)					Analysis software		
Scan-range	Scan-speed	Standard (type, serial n ^o ., certificate)		C _x	C _y	Φ _{xy}	Non-linearity
Results of the calibration of the z-axis (chapter 7.5)						Date	Time (from-until)
Scan-range	Scan-speed	z medium elongation	Analysis software	Method (according to ISO 5436-1 linewise/plane, histogram)			
Standard (type, serial number, reference value, calib.certificate)			C _z	Remarks (e. g. on the kind of levelling)			

This report contains the most important results of calibration. Further calibration results, remarks, etc. are to be noted on a separate sheet (Annex)

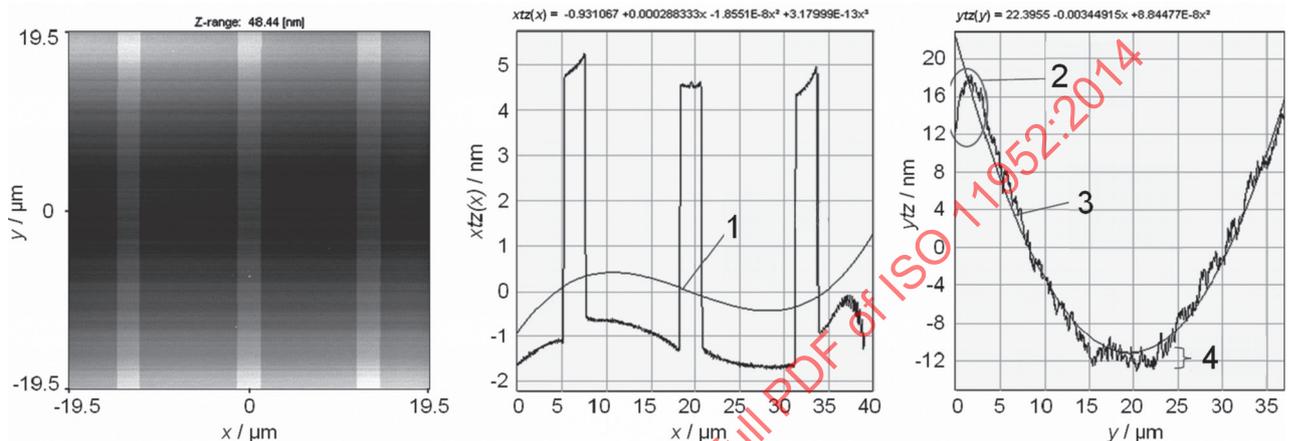
Enclosures: Results of calibration with 3D-standards (chapter 7.6) Diagrams for C_x / C_y Diagrams for C_z(h) _____

Remarks, reference to QM-System:

Date _____ Signature operator _____ Signature supervisor _____

Annex A (informative)

Example of superposition of disturbing influences in the topography image



Key

- 1 dynamic cross-talk of the fast scan axis x in z -direction (fit)
- 2 initial drift in the first scanlines
- 3 out-of-plane motion of the y -axis: crosstalk of y -axis in z -direction
- 4 scanline-to-scanline noise (jumps in z when reversing the scan direction forward \leftarrow backward)

Figure A.1 — Topography image taken after the warm-up phase has elapsed

NOTE Without levelling (left), appurtenant averaged x -profile (xtz , mean of all scan lines, middle) and a y -profile (ytz , mean of all columns, right) from the middle of the image.

The example shows the superposition of various disturbing influences as can still occur after the warm-up phase of the instrument has elapsed; for the characterization of the instrument they are to be separated from each other by careful investigations so that they can be differentiated into temporary (initial), permanent and dynamic components and suitable countermeasures can be taken, if necessary. The fast scan direction is from right to left (x -direction) and the slow one (y -direction) from top (start of the measurement) to bottom (end of the measurement).

On the basis of further investigations (which are not shown here), the observations from this topography image can be interpreted as follows:

- At the beginning (left: in the first scan lines, right at the top of the image) another initial drift can be observed (diagram on the right side, at the far left) — a temporary effect which will no longer occur when the measurement is repeated.
- In the y -direction, a bowed guidance deviation ytz dominates which is to be attributed to shortcomings of the scanner design.
- Further, this individual y -profile shows the noise.
- In contrast, in the fast x -direction, dynamic guidance deviations xtz dominate. The higher the scanning speed, the greater these deviations.

NOTE According to the design and the operating conditions, the disturbing influences exerted on the instrument can be most different. So observations similar to those made in the measurement discussed here can also be due to other causes. In any case, further investigations need to be carried out to identify the causes for the observations.

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Annex B (informative)

Sound investigations: Effects of a sound proofing hood



Figure B.1 — AFM in a sound proofing hood consisting of a box of thin aluminium plates, the inside being lined with bitumen and foam combs

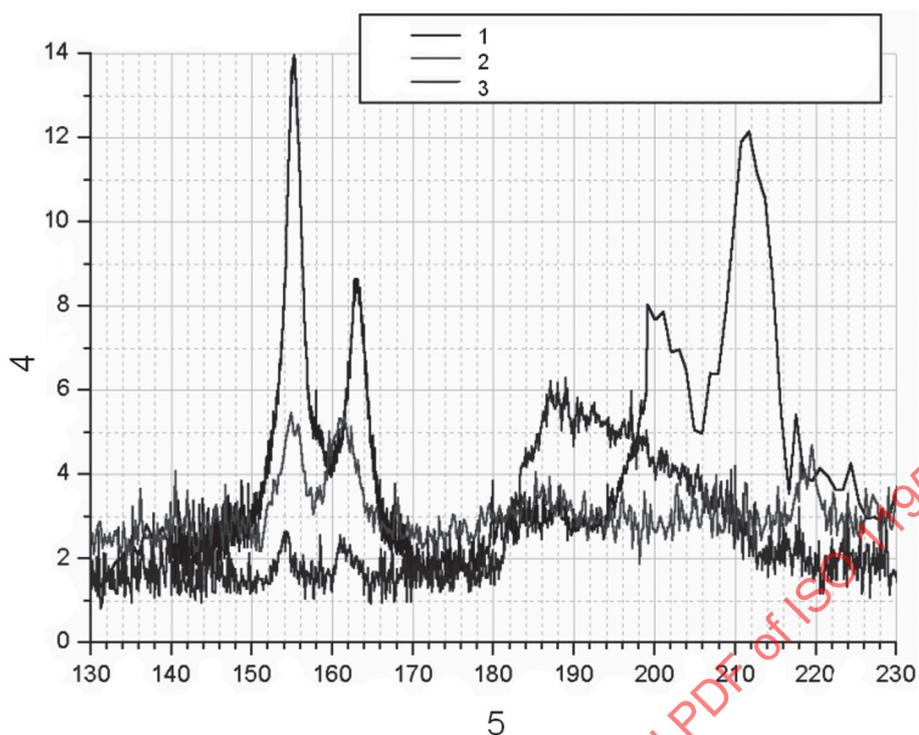
For vibration protection an active vibration damping stage is used on which the AFM is accommodated in a chamber (not to be seen here).

The sound proofing effect of the hood shown on the photo was investigated by exposing the AFM to an external sound source. As the measure of the influence of the sound the noise was selected; it was recorded with the probe approached and active force control in a simultaneous standstill measurement. During the measurement the frequency of the sound was varied.

In this way it is possible to derive frequency spectra for the sensitivity of the measurement to external sound from the standstill measurements. The diagram below shows three frequency spectra, the influences of the sound being reduced not only by the hood but also by a massive specimen holder ([Figure B2](#)).

Below and above the frequency interval shown, the measurements did not exhibit any significant sound influences.

NOTE The effectiveness of such hoods, measuring chambers and specimen holders depends on the set-up of the measurement circle and sometimes also on the adjustments of the measurement parameters.



Key

- 1 without the chamber, specimen on thin Al plate
- 2 within the chamber, specimen on thin Al plate
- 3 within chamber, photomask on mask holder
- 4 amplitude, in nm
- 5 frequency, in Hz

Figure B.2 — Sound sensitivity without and with sound proofing hood as well as with different specimen holders

NOTE Noise in a standstill measurement as a function of the frequency of external sound to which the instrument is exposed, as a measure of the spectral sound sensitivity of the practical measurement operation.

Annex C (informative)

Thermal isolation effect of a sound proofing hood/measuring cabin

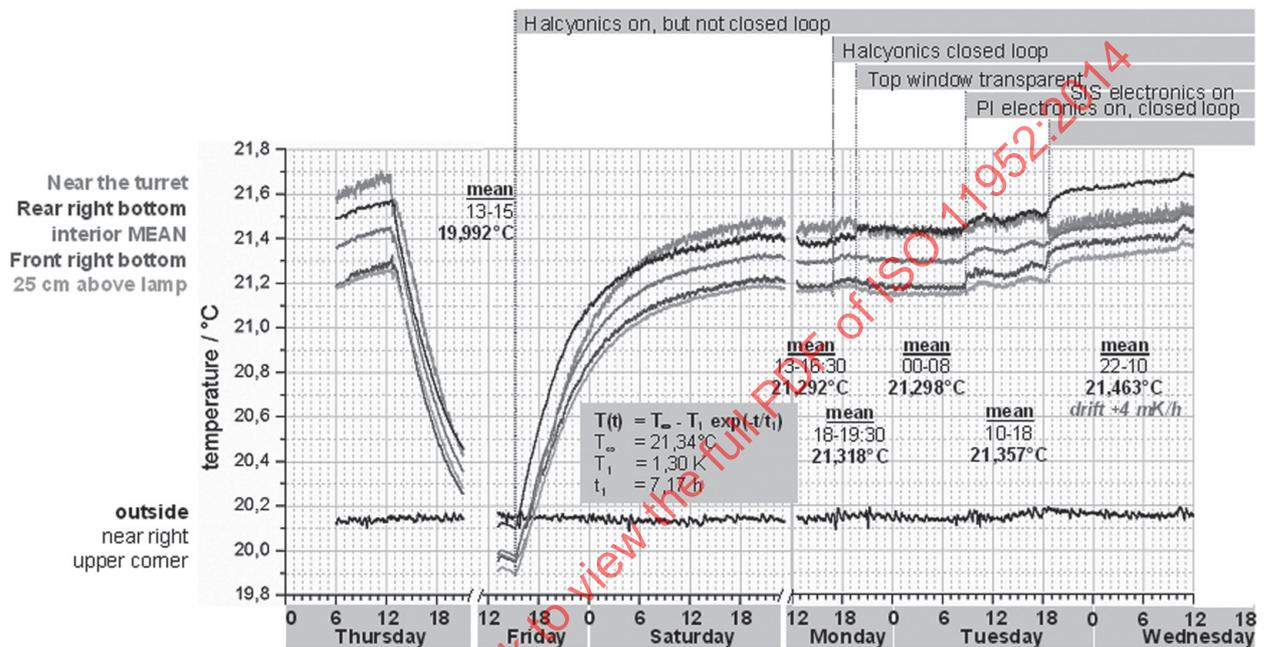


Figure C.1 — Temperature variation inside and outside the sound proofing hood

The diagram in [Figure C1](#) illustrates the temperature isolating effect of the sound proofing hood presented in [Annex B](#). The air temperature outside the hood (black curve) was compared with that at various places in the hood. When all components of the apparatus in the hood are switched on, a temperature is reached inside the hood which is a little less than 1,5 K above ambient temperature.

In order to identify the causes of this warming, all heat sources in the chamber were switched off (in this example, Thursday, 12:00 h) and a waiting time of a little more than one day was observed for the system to cool down to ambient temperature. Subsequently, the potential heat sources were successively switched on again. The active vibration damping stage with integrated power unit proved to be the strongest heat source (87 %) while the measuring head and the scan stage with capacitive sensors contribute only little to the thermal load ([Table C1](#)).

NOTE In such cases, the individual heat sources and the time constants of the temperature variations need to be determined for highly quantitative measurements in order to appropriately account for their influence; this can be achieved, as shown in this example, by observing a defined waiting time after the chamber is opened. It is also possible to take appropriate structural measures, e.g. to keep and design the measurement circle as compact as possible and to use materials with small thermal expansion so that the position of the measuring probe in relation to the specimen does not vary with temperature.

Table C.1 — Thermal loads

Heat source in the box	Thermal contribution approx. in K	Heat component in %	Warm-up period approx. in h
Vibration damping stage, switching-on	+1,30	87	24
Measuring head, electronics on	+0,11	7	3
Scan stage, electronics and control on	+0,06	4	2
Vibration damping stage, control on	+0,03	2	2
Sum	+1,49		

From the temperature variation conclusions can be drawn for the waiting times to be observed: After prolonged, extensive work on the instrument with the hood door opened (e.g. specimen/probe change with adjustments), the waiting time to be observed is approximately half a day (e.g. adjustments on the evening before, start of the measurements in the next morning); temporary opening for a few minutes is not to be regarded as critical considering a time constant of about 7 h.

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