INTERNATIONAL STANDARD

ISO 11952

First edition
2014-05-15

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

Reference number
ISO 11952:2014(E)

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Published in Switzerland

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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*.

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.

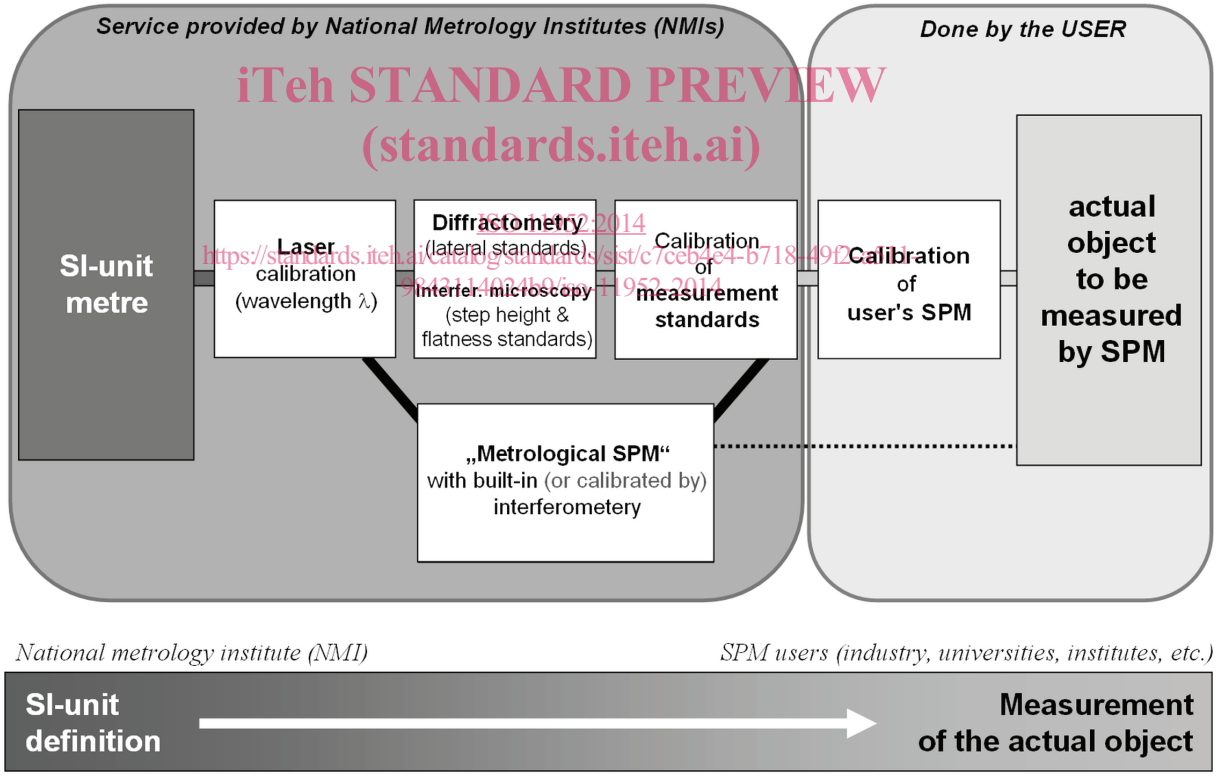


Figure 1 — Traceability chain for scanning-probe microscopes

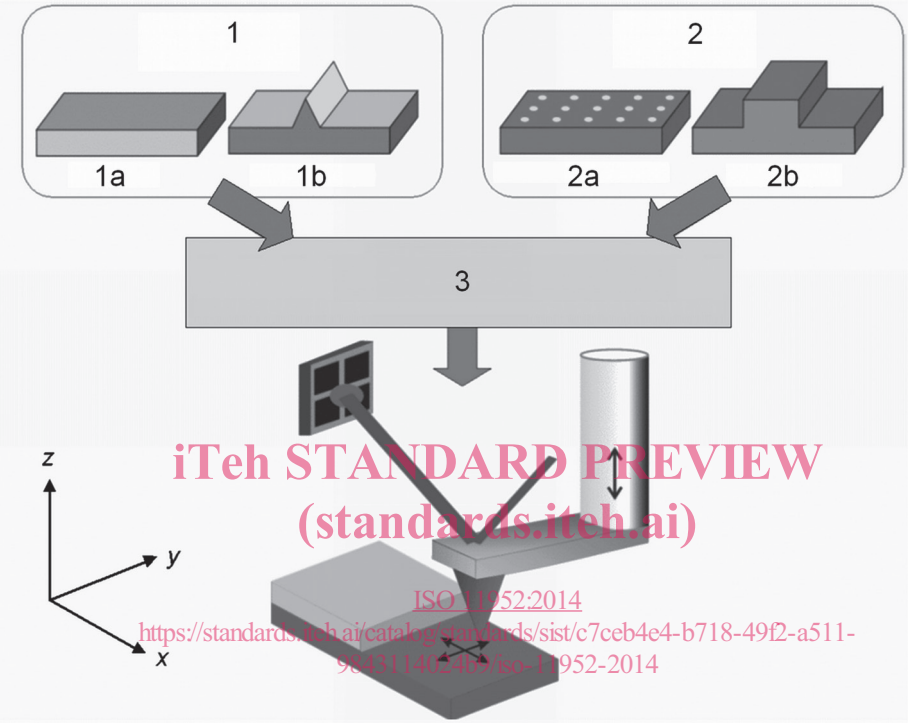
NOTE The calibration of a user's SPM by means of traceably calibrated measurement standards is the object of this International Standard (done by the user).

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*),[11] 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*, *ytz* 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 $j = 1,..., Nj$)
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
xty	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

Rqx noise in the *x*-direction

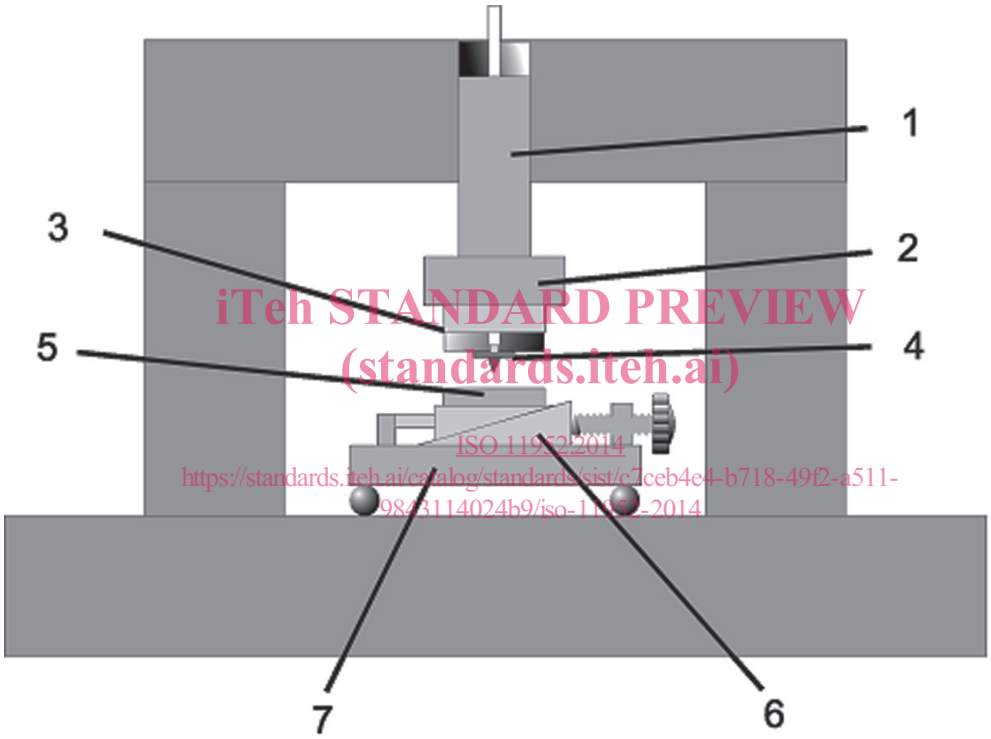
Rqy noise in the *y*-direction

Rqz (Sqz) 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.

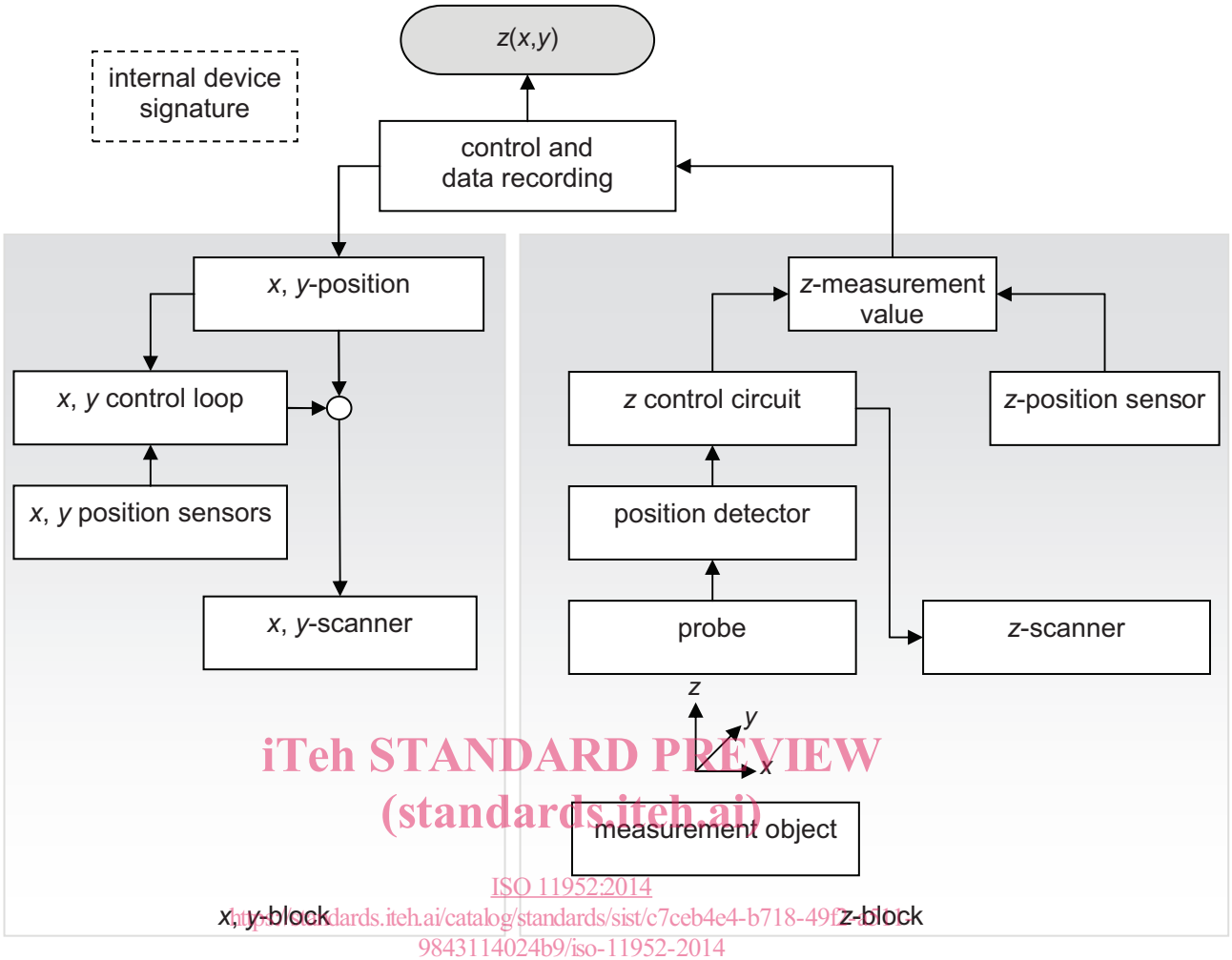


Figure 4 — Block diagram of a scanning-probe microscope

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:

a) investigation of the waiting time after putting the instrument into operation (warm-up, initial drift, etc.) (see 6.2);

b) 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);

c) 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);

d) the noise of the instrument (see 6.3 and also Table 1);

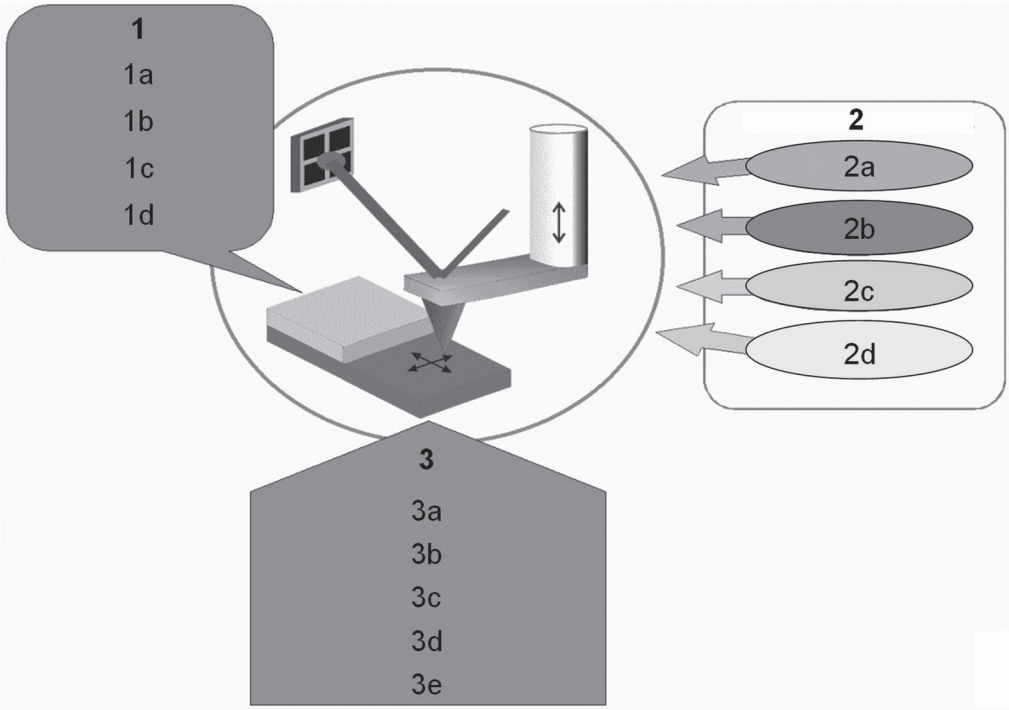
e) *xy*-scanner/*z*-scanner-guidance deviations (cross-talk from one scan axis to other axes, which can, at times, be detectable only by repeated measurements) (see 7.3);

f) 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 (P) and integral (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