INTERNATIONAL STANDARD

ISO 11039

First edition
2012-02-01

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

Analyse chimique des surfaces — Microscopie par sonde à balayage — Mesurage du taux de dérive

Reference number
ISO 11039:2012(E)

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

Published in Switzerland

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 11039 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 9, *Scanning probe microscopy*.

Introduction

Scanning-probe microscopy (SPM) is a well-known microscopic technique for nanoscience and nanotechnology. Working at, or close to, atomic-scale resolution, it is recognized that the time stability of such instruments is very sensitive to their design, operating environment and usage. Among the many technical specifications of SPM, the drift rate is an essential parameter. A knowledge of, and minimization of, drift in the X-, Y- and Z-directions is required for designing many experiments. It is not only important for obtaining undistorted images and series of images throughout an experiment, but is also critical when, for example, measuring physical properties, monitoring dynamic behaviour, making micro/nanoassemblies, and manipulating materials at the nanoscale. Furthermore, a knowledge of the instrumental drift rate is also important when selecting an instrument for use. It is therefore desirable that manufacturers provide suitable information about the instrumental drift characteristics in a common way. Many manufacturers provide closed-loop scanners in their instruments. Unfortunately, drift is still present, although the magnitude of the drift rate is significantly reduced. Therefore, practical methods to measure and characterize drift rates of SPM instruments in the X-, Y- and Z-directions are required and are contained in this International Standard.

Two measures, the maximum and the average drift rates, are described for both the X-Y plane and the Z-axis. The maximum drift rate is given as the maximum observed, for reasons of economy, after a small number of fairly simple measurements. The maximum drift rate allows the user to design experiments that fall within the working zone available given the duration of the intended experiments; however, the maximum observed X-Y and Z-drift rates are based on a small number of observations and are less precise than the average drift rates determined. To deduce a working zone, a rule of thumb is to assume that the maximum is twice the value of the average. Clearly, in any population, the true maximum for a very large number of measurements would be very large, but here it is expected that the user only expects some 90 % of experiments not to require repetition as a result of the drift properties of the instrument. Depending on the importance of the measurements, users may, of course, set themselves any chosen margin of safety based on the data derived using this International Standard.

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

1 Scope

This International Standard defines terms and specifies measurement methods for characterizing the drift rates of scanning-probe microscopy (SPM) instruments in the X- and Y-directions and, for SPM instruments measuring topography, the drift rate in the Z-direction. Though the behaviour of the long-term drift rate might be nonlinear, both that and the behaviour of the short-term drift rate after a user-defined settling time can be characterized by either typical average or typical maximum drift rates.

This International Standard is suitable for evaluating the drift rate based on SPM images. It is intended to help manufacturers quote drift figures in specifications in a meaningful and consistent manner and to aid users to characterize the drift behaviour so that effective experiments can be designed. These measurements are not designed for image correction.

2 Normative references

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

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

3 Terms and definitions and abbreviated terms

3.1 Terms and definitions

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

3.1.1
drift
change in position of the probe tip, for a given positional setting by the instrument controller, relative to the test specimen

NOTE Drift occurs in all parameters (e.g. X-, Y-, Z-displacements, laser positioning on the cantilever, intensity in SNOM sources) but in this International Standard the term drift is restricted to the unintended change in position of the probe tip for given scanner X-, Y- and Z-coordinates, relative to the test specimen.

3.1.2
drift rate
quotient of the linear displacement of the probe tip, for a given positional setting, relative to the test specimen over a given time interval by that time interval

NOTE 1 The time interval is usually chosen to be the time between successive images.

NOTE 2 The drift rate may be given for each of the X-, Y- and Z-axes separately or as the magnitude of the resulting vector.

3.1.3
average drift rate
average of appropriate **drift rates** measured during a specified period of time

NOTE 1 The average drift rate may be given for each of the X-, Y- and Z-axes separately or as the magnitude of the resulting vector.

NOTE 2 The average drift over a long period might be low if, by chance, the test specimen returns to its original position whilst the average drift rate, being measured non-vectorially between successive images, remains high.

NOTE 3 The average drift rate obtained here is intended for designing experiments so that the effects of the drift can be minimized or eliminated so that e.g. the important region of the test specimen remains in the field of view. Thus, the user may multiply the average drift rate by a factor of 2 and add some safety factor to ensure that a certain fraction of the field of view is maintained during the experiment.

3.1.4
maximum drift rate
maximum of the **drift rates** measured during a specified period of time

NOTE 1 The maximum drift rate may be given for each of the X-, Y- and Z-axes separately or as the magnitude of the resulting vector.

NOTE 2 As in any set of measurements, the true maximum drift rate measured might increase slowly with the number of measurements. The maximum value obtained here is intended for designing experiments so that the effects of the drift can be minimized or eliminated so that e.g. the important region of the test specimen remains in the field of view. Thus, the user may add some safety factor and a very accurate value of the maximum is not required.

3.1.5
settling time
time after selecting the area of the test specimen or point on the test specimen for measurement and the commencement of the measurements for which the drift data are relevant

NOTE Settling times are often chosen to be from 5 min to 60 min for convenience.

3.2 Abbreviated terms

AFM atomic-force microscope

NPG non-periodic grating

SPM scanning-probe microscopy

4 Measurement method

To characterize the drift behaviour of an SPM instrument, it is important to recognize that drift occurs as a result of many processes, as shown in Annex E, and each of these processes causes an onset of drift that might slowly reduce with time. Thus, after switching on the final part of the instrument, a drift behaviour may be observed that, after a suitable waiting period, will generally be lower than that initially obtained. After inserting a new test specimen into the measurement position, a similar behaviour occurs. After moving the specimen to a new point using the specimen stage controls, a further drift will be initiated. Finally, after moving the SPM probe to a new region of the specimen using the piezoelectric scanner, a fourth drift behaviour is seen. Each of these behaviours might occur in a different direction and be of different magnitude. Indeed, each time this process is repeated, all these might change in magnitude and direction. Nevertheless, after deciding on a certain protocol for operating the instrument, typical average and maximum drift rate behaviours can be established. Important in this protocol is the settling time, i.e. the period during which the instrument is allowed to stabilize after selecting the area of the specimen or point on the specimen for measurement and the commencement of the measurements for which the drift data are relevant. The average and maximum drift rates permit the user to decide what influences the drift behaviour and hence what actions need to be taken to ensure that the drift performance is suitable for the user's requirements. This is described in Clause 6 and Annex E. Subclause 6.1 describes an initial check to see if there is a significant drift behaviour that might need further investigation. If the instrument is adequate, the investigation may cease. If further investigation is required, a

basic characterization is described in 6.2 to evaluate an appropriate settling time. For those interested in a fuller characterization, the effects of changing operating conditions are evaluated in 6.3.

For the drift rate measurement, the following three methods are specified in this International Standard:

— image correlation method (see Annex A);

— characteristic-marker method (see Annex B);

— non-periodic grating method (see Annex C).

To facilitate the selection of the drift rate measurement method, guidance is given in Annex D.

5 Requirements

5.1 Instrument requirements

5.1.1 The SPM instrument shall have the capability of measuring and recording digital images of the specimen surface, as a function of time, throughout the work.

5.1.2 The instrument shall maintain its dimensional calibration throughout the work.

5.2 Environment requirements

5.2.1 The instrument should, if possible, be operated under the required, or better, environment conditions specified in the manufacturer's documented instructions.

5.2.2 It is recommended that the measurement be performed in controlled conditions with the temperature stable within ±1 °C and the relative humidity preferably less than 50 %. The laboratory environment should be clean, with levels of electromagnetic interference, ambient vibration and ambient noise which are sufficiently low that they do not influence the characterization of the instrument. The measured data will relate to the instrument used in whatever operating conditions are selected and might or might not be relevant to any other operating conditions. Suggested ways to improve the operating conditions, likely to lead to improved drift characteristics, are provided in Annex E.

6 Measurement procedures

6.1 Initial check

6.1.1 Select the probe in accordance with the manufacturer's documented instructions.

6.1.2 Operate the instrument in the manner and operating mode for which drift data are required. For low-drift performance, keep as much of the instrument operational continuously and switch on remaining items more than 1 h before conducting measurements.

NOTE Different operating modes might lead to different amounts of drift if different amounts of power are supplied to different parts of the instrument.

6.1.3 Prepare the test specimen together with any reference material required for the measurement method. When using the image correlation method of Annex A, it is preferable to have obvious features within the field of view. For the characteristic-marker method of Annex B, there will need to be at least two sharp features at a distance of approximately a quarter of the image size in from two opposite corners of the field of view. For the non-periodic grating method of Annex C, obtain a suitable grating. Ensure, as far as possible, that these items are clean in order to avoid probe tip contamination. Particulate matter might move during analysis, causing

erroneous drift determination. Particulates can be removed by washing in, for example, high-purity iso-propyl alcohol with ultrasonic agitation. Ensure that any solvents used do not adversely affect the specimen.

6.1.4 Mount the specimen in accordance with the instrument operator's manual or in-house documented procedures.

NOTE Poor specimen-mounting methods might increase the drift behaviour.

6.1.5 Optimize the image acquisition parameters in accordance with the instrument operator's manual or in-house documented procedures.

NOTE High tip loads will cause tip wear and this might lead to imprecision in the drift measurements.

6.1.6 Set the scan field of view to a suitable value to define the drift behaviour. If uncertain about the behaviour, 5 µm is a suitable value for initial studies. Select a region of the specimen with at least two sharp features at a distance roughly a quarter of the image size in from two opposite corners if using the characteristic-marker method of Annex B or obvious features across the field of view if using the image correlation method of Annex A. Scan the specimen with the frame time used for the studies for which this characterization is required (e.g. 5 min to 10 min).

6.1.7 Record two successive images. Determine the X-, Y- and Z-drift rates in accordance with the selected method listed in Annex A to Annex C, using images that all have the same X- and Y-scan directions.

NOTE Some instruments scan with the slow scan firstly in one direction and secondly in the reverse direction. There might be a shift between these images. By restricting the analysis to one direction, issues associated with this shift are removed.

6.1.8 If the measured drift rates are significantly smaller than the minimum drift rates required to conduct the required experiments, the instrumental operating conditions are satisfactory and no further drift characterization will be required. The initial check may be considered complete and the evaluation terminated. If the measured drift rates are not significantly smaller than the minimum drift rates required to conduct experiments, or if further characterization is required, proceed to 6.2. If the measured drifts exceed 20 % of the scan size in the X-, Y- or Z-directions, increase the relevant scan size to satisfy that condition and repeat 6.1.7.

6.2 Basic characterization and the settling time

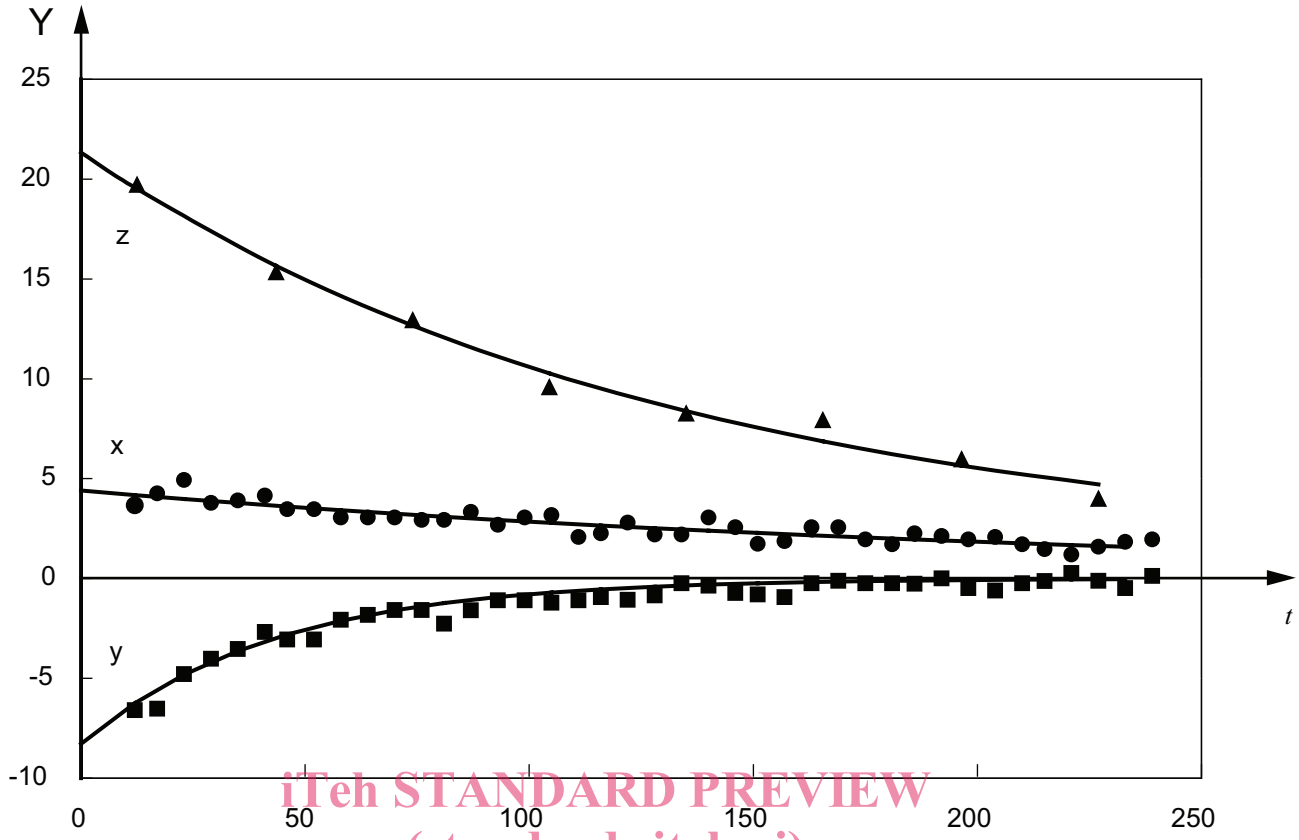
6.2.1 Continue from 6.1.8 and record a series of successive images for 2 h. For measuring the drift rate performance, the time at which the specimen position has been set and the image area has been selected is taken as the origin of time. For each change in specimen position or imaged area, a new time origin is established.

6.2.2 Determine the X-, Y- and Z-drift rates as in 6.1.7.

6.2.3 Observe the variation of the drift rate with time and decide a practical time after setting the specimen in position and addressing the selected point on the specimen for which the drift rate is adequate for the work intended. This is the settling time. If the drift rate after 2 h is too high for the intended use of the instrument, continue for a longer period or consider operational improvements to the instrument or use the equipment as suggested in Annex E, and repeat 6.1.1 to 6.2.2.

NOTE 1 An example of such measurements is shown in Figure 1. In that example, if 9 nm/min is an acceptable drift rate in the X-Y plane, further data could be recorded with no delay after identifying a new point on the specimen for study. If 4 nm/min is acceptable, a settling time of 1 h is required after starting with a new specimen.

NOTE 2 Occasionally, it might be found that the drift rate increases with time as the several independent contributions can partially cancel near the start of the measurements. Extending the settling time is then unhelpful and operational improvements will have to be checked.



Key

t time, in minutes

Y drift rate, in nm/min

Figure 1 — Measured drift rates in the X-, Y- and Z-directions as a function of time at a new point in an instrument in which everything except the laser is on continuously and the laser has been switched on for 16 h[1]

6.3 Further characterization and fresh image areas

6.3.1 Next, establish a grid of four points near the extremities of the X-Y scan range of the piezoelectric scanner to which the probe tip will be addressed, as well as the central point, as shown in Figure 2. These five points define the centres of five image areas with fields of view sufficiently large to cover any drift likely to occur.