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International Interlaboratory Comparison of AFM spring constant calibration and force-distance curves - Protocol For Analysis

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ABSTRACT

This report describes the protocol for analysis for an interlaboratory comparison of AFM spring constant calibration and force-distance curves. Two AFM cantilevers and one silicon sample are provided for this study, along with this protocol and a draft ISO standard, which contains simple instructions for the analysis.

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Approved on behalf of the Managing Director, NPL
by Dr Neil Harrison, Quality of Life Division

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1 OBJECTIVES

The measurand in this study is the normal spring constant, k_z, of each of 2 cantilevers and the associated force-distance curves on a 'infinitely stiff' surface, here, silicon in order to (i) to evaluate the current comparability of cantilever spring constant measurement at the nanoscale, (ii) to evaluate the repeatability of the spring constant and the linearity of force-distance measurements.

2 TIME TABLE

You should complete the analysis for this work by **14 August 2009**. If you cannot do so and need extra time please inform charles.clifford@npl.co.uk.

3 THIS PACKAGE

This package, unless you have otherwise specified, contains this protocol and one small gel-pack box containing two AFM cantilevers. Inspect the packaging to check if it has been opened by customs and if the integrity of the samples has been compromised. Please notify us that everything is received in order.

☐ I have emailed NPL that all is OK with the samples on / / 2009

4 THE MATERIAL

The materials for analysis in the standard set comprise two rectangular AFM cantilevers with trapezoidal cross-sections. The cantilevers are arranged with the more compliant cantilever on the left and the stiffer cantilever on the right when the box is facing you so that the hinges are at the back. The cantilever chip holders are arranged so that the cantilevers are mounted on the edge nearest the hinge of the gel pack box.

Your sample codes are given in the covering letter in a completed table of the form:

Serial Numbers of Your Samples

Cantilever	
C **(0.1 to 0.9 N/m)**	**S** **(15 to 60 N/m)**

Also included in the table are nominal ranges of the spring constant of each cantilever and given as a guide to the order of magnitude of k_z of each cantilever in case you need to pre-select instrumental options or parameters. The actual spring constants of the cantilevers could fall anywhere within the ranges given above.

Do not unpack the cantilevers until you are nearly ready to analyse them. Before doing so, read the rest of this protocol so that you are clear about the next steps. If any steps are unclear please contact me as soon as possible for clarification.

Also, supplied is a piece of silicon, approximately 1cm x 1cm. This is supplied **face down** in a separate container.

5 SAMPLE AND CANTILEVER HANDLING

The following comments should prove helpful in handling samples.

If you intend to analyse the cantilevers one by one by mounting them on the same sample holder, it is good practice to keep the cantilevers in their containers and remove them only when you are ready to mount them. If you mount them together on one sample holder, ensure that their identities are maintained.

You may have appropriate sample handling procedures as routine in your laboratory. Keep to those procedures but ensure that they are always superior to the minimum level given below.

You may need to handle the cantilevers and samples using uncoated clean stainless steel tweezers. If so, grip the cantilever chip holder away from the cantilever end and use round nose tweezers rather than sharp tweezers to minimise damage to the cantilever chip. Avoid breathing or speaking over the cantilevers. Use polythene gloves, or gloves of a higher quality, to avoid contaminating the tweezers or your cleaning equipment with finger grease. Do not use moulded gloves, for example vinyl, which may be covered with highly contaminating release agents.

6 PRACTICAL ISSUES IN MEASURING k_z AND FORCE DISPLACEMENT CURVES

Do not use any cleaning method on the cantilevers as this may affect the results. If there are any dust particles on the cantilevers these can be *gently* blown away using an inert gas [use, for instance, pure dry inert gases such as Argon or nitrogen to remove any particles. Do not use gas from pressurised cans that include a propellant or from compressed air lines that may

contain oil vapour, do not use a method likely to contaminate the sample].

Ensure that any necessary instrument calibrations are up to date. For AFM this may include the *z* piezoelectric scanner calibration. For issues relating to scanner calibration, please use the methods that you normally employ. If in doubt, some methods are given in reference [1-2]. In your results please state the z calibration method used and when it was undertaken.

The silicon sample is provided for calibrating the cantilever deflection sensitivity constant and to undertake force-distance curves. To mount the silicon sample in your AFM, in order of preference, the following methods are recommended: 1) use of a vacuum stage or a very secure mechanical holder, 2) epoxy glue (e.g. araldite), 3) cyanoacrylate glue (superglue). Sticky tape should be avoided if at all possible. Please note the holding mechanism in your report.

Undertake the calibration of spring constant first as described in section 6.1, and then undertake additional force-distance curves on silicon as described in section 6.2.

For force-distance measurements on silicon, try to ensure that your analyses are within the central regions of the samples in a featureless area of the sample. Do not acquire data within 1 mm of the sample edge.

Record all of the instrumental parameters used. Record the temperature and humidity of the laboratory at the time of the experiment and whether this was similar or noticeably different to the temperature and humidity in the 2 days leading up to the experiment.

Undertake the following experiments:

6.1 MEASURE k_z

The units for measuring the normal spring constant is N/m. For each cantilever, measure k_z by whichever method(s) you wish to use. Repeat this method 5 times for each cantilever, although this may not be possible for the more time consuming methods. You may use as many methods as you like.

Some example methods that you may wish to follow are detailed in the attached draft ISO standard 11775. Describe the essential parts of the methods you use and any divergence from the attached draft. You may diverge from the draft, if for instance, you have a better method or if you cannot realise a part of the method. A note as to why you have diverged will help us revise the draft. If you are using a completely different method, please provide a detailed method.

Record your 5 values of k_z for each method, along with the uncertainty of the result, if at all possible. This may be expressed as the mean value ± the standard uncertainty or the expanded uncertainty at 95% confidence. Express the results in the way you feel is best but please describe what you have done, e.g. components to the uncertainty budget and the manner of their combination.

If possible measure the resonant frequency of the cantilever using your AFM either using thermal motion or using mechanical oscillation or preferably both methods.

6.2 MEASURE FORCE DISPLACEMENT CURVES

Depending on the method, as part of the k_z calibration process, you may need to calibrate the deflection sensitivity (sometimes called InVOLS). This should be done either by following the method outlined in the attached draft ISO standard and using the supplied silicon wafer or using your own method. This should be undertaken within the *linear region* of the detector. Please describe which method is used.

After you have completed the k_z calibration, please test the linearity of the detector system over a wider range of movement. Reset the deflection signal (A-B) to as negative a value as possible. On most instruments this is likely to be around –9.5 to -10V.

Land on the supplied silicon surface in contact mode at an appropriate set force and undertake 5 force-distance curves at a rate of 0.2 Hz up to the maximum deflection value (e.g. ~10V). If possible, use closed loop z mode for the z piezo scanner and record the data in terms of the closed loop sensor signal (in nm) versus raw cantilever deflection (in Volts).

When all analyses are complete, return the cantilevers and sample to their containers. Please retain them in case we find any anomalies between the data sets.

7 REPORTING AND CALCULATING THE RESULTS

Calculate and report the k_z. Also, supply Charles Clifford with an electronic copy of the original force versus indentation depth data, calibration data, temperature and humidity and the calculations that you have made. Include details of all values along with their uncertainty values that you have you used to calibrate k_z. As a reminder, values and data to report may include:

1) Z scanner calibration of AFM (when and how)
2) Method(s) used to calibrate k_z values and any deviations from draft ISO standard
3) The 5 k_z values for each method and associated uncertainties (including the values and uncertainties of the ancillary measurements)
4) All measurements and data associated with 3), this could include temperature, humidity, dimensions of cantilever, force-distance curves along with values of deflection sensitivity, frequency measurements including spectrum
5) Resonant frequency of cantilever plus the frequency spectrum recorded in the format described in section 8.2
6) The 5 force distance curves for the full range of the photodiode as per section 8.1
7) Any general comments on draft ISO standard

8 DATA REPORTING FORMAT

8.1. FOR FORCE-DISPLACMENT CURVES

In decreasing order of preference, the preferred digital data formats are:

(1) Three column ASCII format as shown in Table 1.
(2) Manufacturer's text data format.
(3) A simple binary format – with details of structure supplied.

Table 1 - Format for force-distance curve data using mechanical oscillation

Closed loop z piezo signal (nm)	Cantilever deflection (V)	Time (s)
0.113	0.000	0.000
0.325	14.881	0.014
0.654	29.763	0.029

8.2. FOR FREQUENCY MEASUREMENTS

In decreasing order of preference, the preferred digital data formats are:

(1) Two column ASCII format as shown in Table 2.
(2) Manufacturer's text data format.
(3) A simple binary format – with details of structure supplied.

Table 2 - Format for Frequency measurements

Frequency (Hz)	Cantilever deflection (V)
0	0.000
10.01	0.881
20.02	0.763

For, thermal vibration measurements, the format is the same as Table 2, with suggested units of $nm^2/Hz^{0.5}$, making sure to note in your report the value of the dynamic deflection sensitivity used.

8.3. GENERAL DATA REPORTING

Send your report and data preferably electronically or if not hardcopy to:
C A Clifford, National Physical Laboratory, Hampton Road, Teddington, Middlesex TW11 0LW, UK, email: charles.clifford@npl.co.uk
(Tel: +44 20 8943 6620; Fax: +44 20 8943 6453)

9. ANY DOUBTS

If you are unsure of anything or need advice please contact Charles Clifford, email: charles.clifford@npl.co.uk.

10. FUTURE PLANS

The study will be used to develop the ISO standard. All methods will become part of an ISO technical report on spring constant determination, with the 'best' methods forming an ISO standard. In addition, if all goes well, the results will be published.

ACKNOWLEDGEMENTS

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REFERENCES

[1] Guideline VDI/VDE 2656 Part 1: 2008-06, Determination of geometric quantities by Scanning Probe Microscopes – Calibration of Measurement Systems
[2] Haycocks J and Jackson K 2007 *Meas Sci Technol* **18** 469