

AFM probe calibration

Intro:

Why to calibrate an AFM probe?

Without calibration, you don't know the force that the tip presses sample. The system takes nominal values for the cantilever and uses those to calculate the force. However, there is a certain spread of these parameters, so in order to know the real force spring constant of the cantilever and deflection sensitivity of the cantilever + optical path system has to be calibrated. Calibration also allows you to gather quantitative information from the QNM channels.

With some techniques, such as nanoindentation, you must calibrate the probe before doing the measurements. However, you can do normal PeakForce scanning without calibration if you don't care what the exact PeakForce is, or if you don't need quantitative information from the QNM channels.

How the peakforce is determined?

$$F = \text{spring constant} \cdot \text{deflection sensitivity} \cdot \text{measured voltage}$$

F = PeakForce, spring constant = cantilever spring constant, deflection sensitivity = tells how to how much the laser spot position at the photodetector changes in volts when the cantilever flexes. The unit of this is nm/V. Measured voltage = the measured photodetector signal.

Example:

You might have noticed that when you use Icon and engage with Scanasyst Air probe the PeakForce setpoint is automatically set to 3.6 nN during the engage. This is because the system calculates the force from nominal values for that probe.

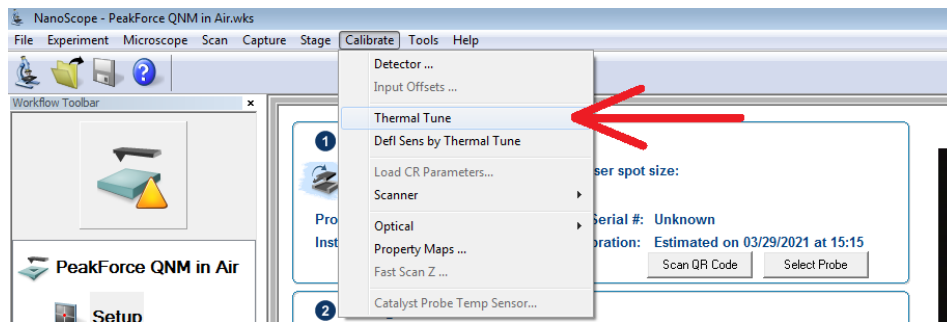
$$F = 0.4 \text{ N/m} \cdot 60 \text{ nm/V} \cdot 0.15 \text{ V} = 3.6 \text{ nN}$$

Calibration procedure

1. Open the software, load the probe and the samples and do the normal laser alignment
For calibration you need a hard sample. If you want to do optional tip radius measurement, you also need a sample with pointy features. Shelf over the controllers has samples that can be used for this. Figure below shows a very hard sapphire sample and titanium sample for (optional) tip radius measurements. They are in the cabinet above the controllers. Tip radius is needed to calculate some of the QNM channels.

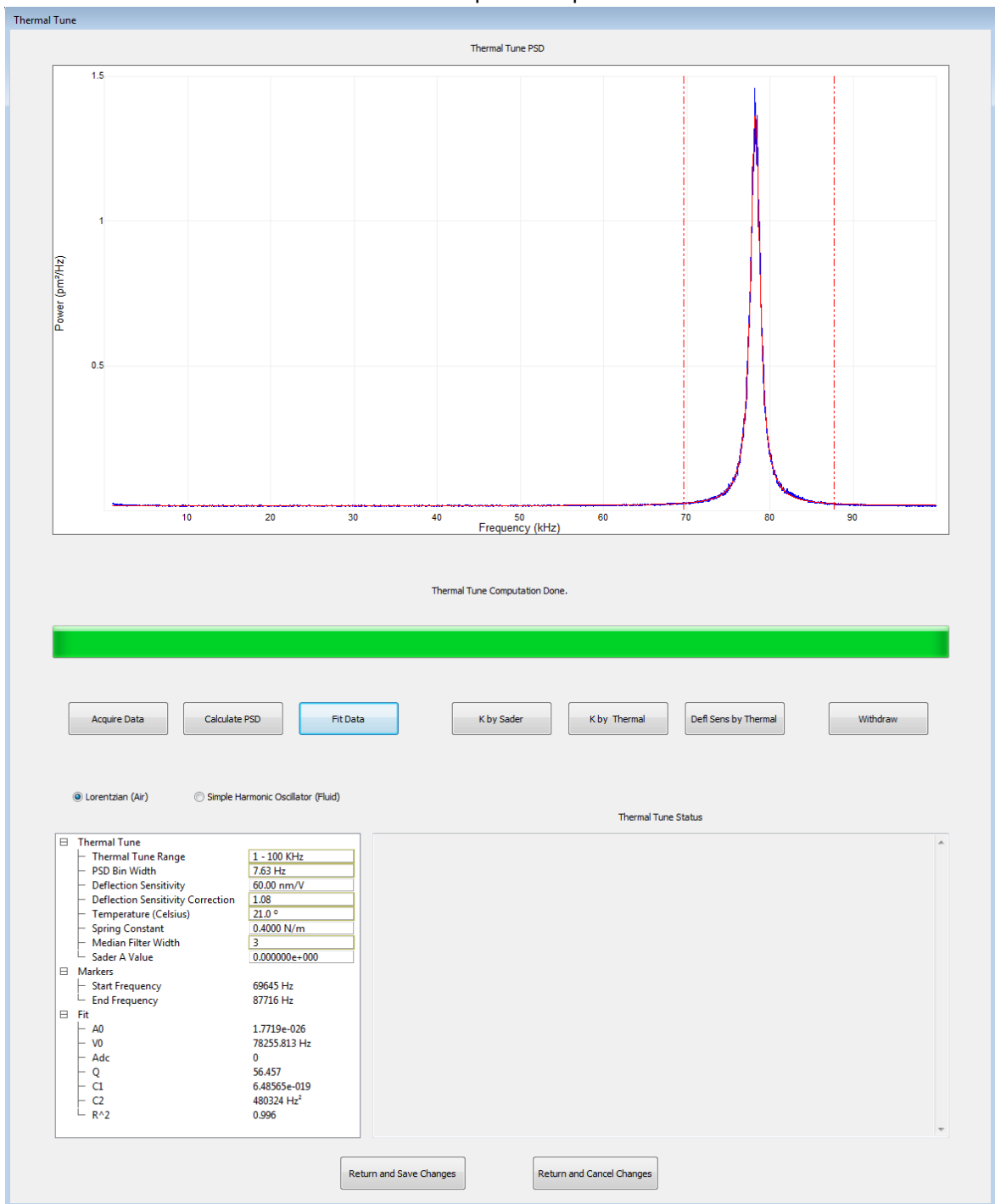


2. Thermal tune. Open Thermal Tune window, shown in the figure below.



3. Press Acquire data and wait for the system to gather the data.

4. Draw cursors on the both sides of the resonance peak and press Fit data



5. You can press K by Thermal and Defl Sens by Thermal to get initial values for them. Make a note of the resonant frequency (V0), Quality factor (Q) and Spring constant. You will need these later.

6. To get a better estimate of the spring constant, using Sader method is advised. This is a standard and well documented method to calibrate the cantilever. Check <https://sadermethod.org/> for more information and to login to the system. In order to use the site you need to register, but it is free.



 sadermethod.org



Calibration of Atomic Force Microscope Cantilevers
Sader Method - Global Calibration Initiative

User Login

Overview

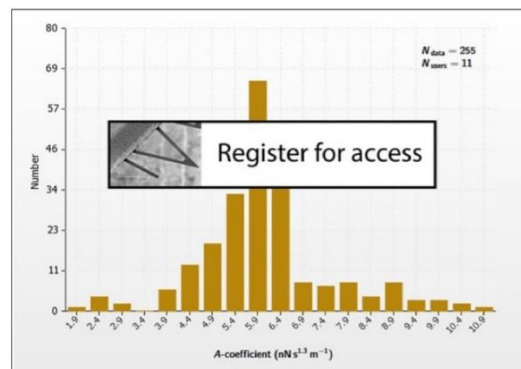
The Global Calibration Initiative will determine the spring constant of your cantilever and, for the first time, standardise it against measurements from the entire AFM community.



AC240-R3

Frequency (kHz): Quality factor: Normal k_{thermal} (N/m):

RESET CALCULATE Normal k_{Sader} (N/m):



Simply enter measurements you already take using your AFM thermal tune.

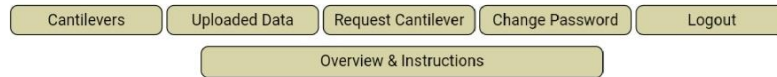
Obtain your free login via the "Register for access" link above.

TO ENABLE FORCE STANDARDISATION, REPORT

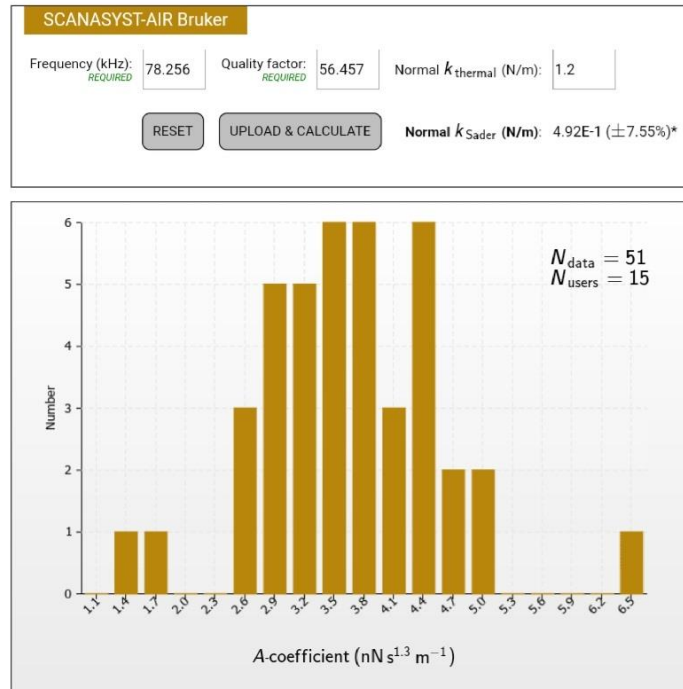
1. Spring constant – k ... that you use!
2. Cantilever model – e.g. Olympus AC240TS-R3
3. Resonant frequency in air – f_R
4. Quality factor in air – Q

* This allows standardisation of ALL force measurements, regardless of your choice of k

7. Login to the site, pick your cantilever and type in the resonant frequency, Q-value and spring constant that you determined during the thermal tune. Press upload & calculate.



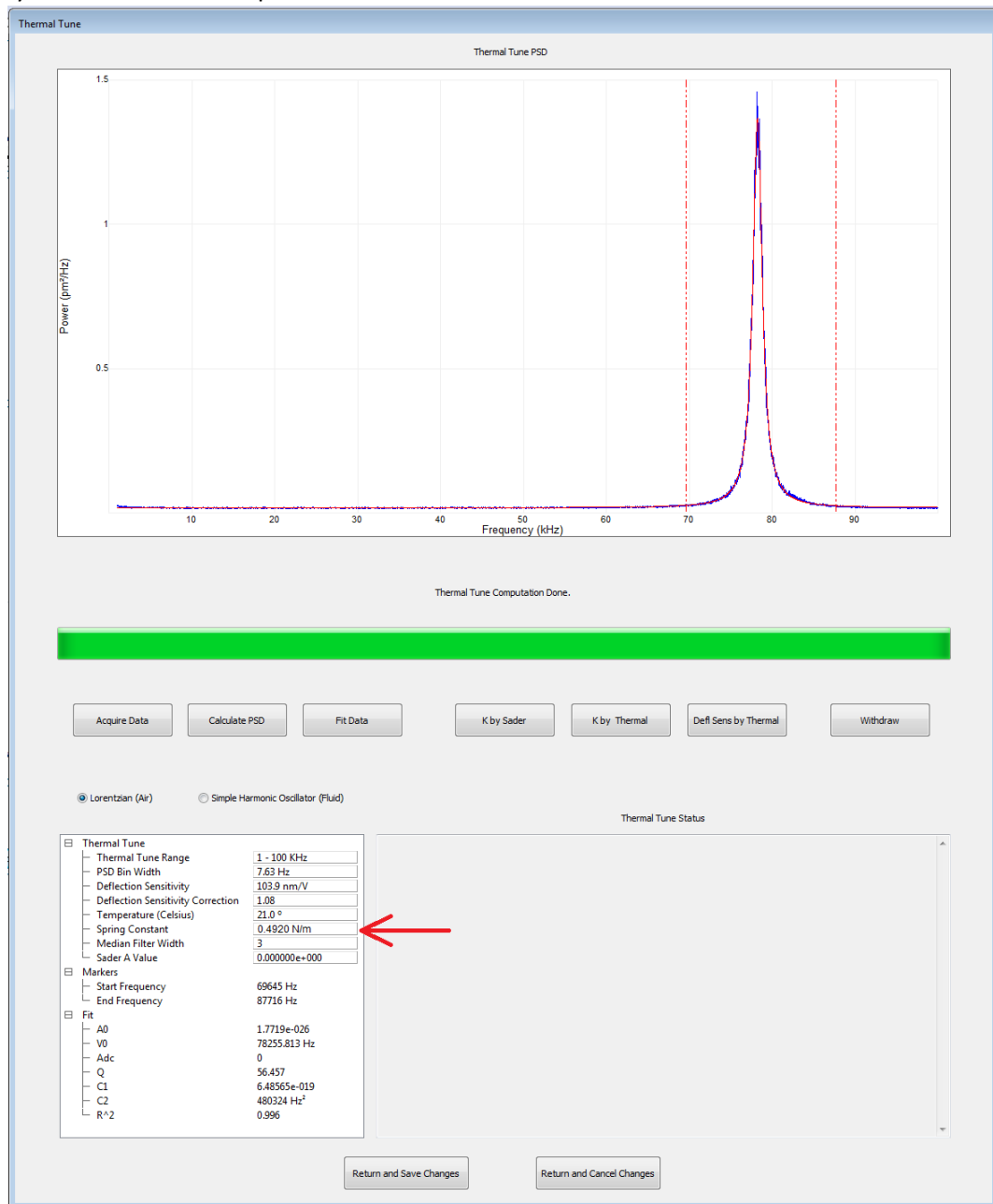
Measure fundamental flexural mode in air at least 100 μm from surface.



* Error estimate (95% C.I.) for the spring constant, k_{Sader} , is derived from the histogram.

This error reduces systematically with increasing number of data-points, N_{data} .

8. Type in your k_{Sader} to Nanoscope.



9. Return and Save Changes

10. Drive to a hard sample. You can use the sapphire reference sample or your own substrate, if it is hard. If you use the sapphire sample, be careful when lowering the scanner, since sapphire is transparent and it might be tricky to find the right surface focus.

11. Go to setup. Under Probe Calibration, click Advanced.

Thermal Tune

For fluid measurements, the calibration environment should match the measurement environment.
Find an appropriate location on the sample. When ready, engage the system.

Move XY Stage Speed: 0%
Move Scan Head Speed: 3%
Engage
Withdraw

Deflection Sensitivity
Calibrate Deflection Sensitivity and Spring Constant

Use Ramp | Use Spring Constant

More Settings
Ramp Size: 300.00 nm
Ramp SetPoint: 0.10 V
Num. Of Curves: 5
Ramp

Current Values
Deflection Sensitivity: 103.92 nm/V
Spring Constant: 0.4920 N/m

Calculated Values
Deflection Sensitivity: 91.94 +/- 0.26 nm/V
Spring Constant: 0.6809 N/m
Update Current Values

Deflection Error (mV) vs Height Sensor (nm) graph showing a linear ramp with a sharp peak at the end.

12. Check that you are in good focus and that the spot is clean and engage.
13. When engaged, ramp the sample a few times. With soft cantilevers (like Scanasyst Air) use deflection setpoints around 0.1-0.2. With stiffer cantilevers use lower setpoints.
14. Make sure that the cursors are at the linear part of the ramp curve. Uncheck the spring constant checkbox. Since you have calibrated the spring constant with the Sader method, you should not let the system to change that. Click update current values to update the deflection sensitivity. Press Ramp again to check the result. If the numbers are red, the values are out-of-tolerance and you need to do the procedure again. When the values show up black, they are ok.
15. Next you can do PeakForce QNM calibration, if you want. Use similar or a bit lower PeakForce setpoint as before and drive amplitude that is half of the ramp size you used before. Click Calibrate.

Calibrate PeakForce QNM
Calibrate Sync Distance QNM and PFT Amplitude Sensitivity for each desired frequency.

PFT Frequency: 2 kHz

More Settings
Drive Amplitude: 150.00 nm
PeakForce SetPoint: 0.08 V
Num. Of Curves: 5
Calibrate

Deflection Sensitivity Convergence Comparison
Defl Sens Target: 91.94 nm/V
Defl Sens from Cur PFT Ampl Sens: 103.03 +/- 1.02 nm/V

Current Values
Sync Distance QNM: 0.00%
PFT Amplitude Sens.: 0.00 nm/V

Calculated Values
Sync Distance QNM: 30.84% +/- 0.01%
PFT Amplitude Sens.: 658.72 nm/V
Update and Verify Current Values

Force (mV) vs Z (nm) graph showing a noisy ramp with a sharp peak at the end.

Finish
Return and Save Updated Values | Return and Cancel Updated Values
Status: Engaged

16. Click update and verify current values, until you don't get red values to Sync Distance QNM and PFT Amplitude Sens
17. Return and Save Updated Values
18. (Optional) You can measure tip radius of your probe by scanning the Ti roughness sample. This is pretty well documented in Nanoscope help. Search for tip radius measurement.
19. Happy scanning!