# Standard Operating Instructions

Warnings

1. Do not touch the scanner and transducer unless you are allowed to.
2. Contact laboratory manager or director to replace tips or tip-area calibration.
3. If tip is exposed to excessive force, such as hitting on a surface, the motor controller initiates and Emergency stop, the icons will be disabled. In an emergency case like this, please reach out to the yellow knob (inside the chamber, above the assembly), turn it to "up" direction to disengage from the surface. Then, enable to motors and navigate to home position.

Sample preparation

1. Attach your sample to an AFM stainless steel disc with an adhesive, preferably with a super glue- such as Loctite.
2. Make sure the attached sample surface is completely horizontal.

Mounting samples on the stage

1. You will notice the aluminum and fused quartz standard samples are already on the stage. You may use the third dot (magnetic) to place your sample.
2. Attach your samples to stainless steel AFM discs (available to users) with a superglue. It is recommended you prepare your samples a day before the experiments.
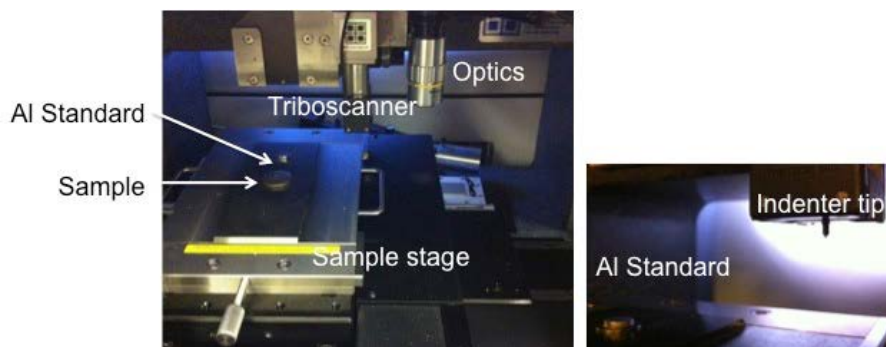
System Preparation

1. Turn on elements and electronics rack in the following order: fiber optic illuminator and vibration isolation control unit. Performech control module and motor driver should be on.
2. Start the Hysitron Triboscan software (image). The initial window will ask "Analysis Only" or "Cancel", cancel if you'd like to initiate the system controllers.
3. When "Transducer Select" window opens, Slot 2 is selected "clear" and Slot 1 is selected "Standard" and Optics is checkboxed. Click OK to proceed.

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4. During system startup, the stage motor will be initiated, wait until it is settled. It will then perform a transducer check.
5. The software will ask to choose a load function, you may choose default load function to proceed.
6. This initiation process takes 2-3 min to complete.
7. When Dynamic Load Function window appears, select default.ldf to proceed.
8. After the initiation process, create a new workspace. Default Workspace icon is located at the top left corner, click and choose New Workspace. When the software asks whether you want to save the current workspace, "don't save" and create a new workspace.
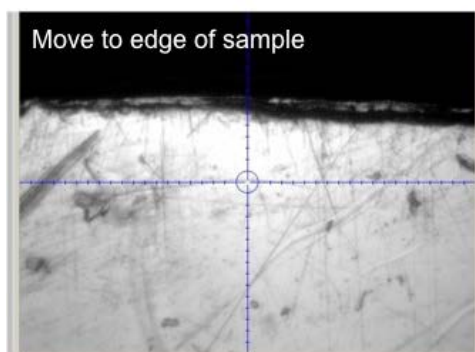
Sample Definition and Boundary Selection

The sample stage can be moved in x, y and z directions and controlled by the navigator. The speed and direction are selected by clicking a location on the navigation window. The speed increases as the clicking location is away from the origin.
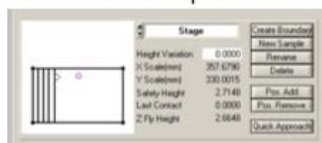
1. Using the x and y navigators (red marks in blue area), bring the Al standard sample under the optics, and focus on the Al standard. The focus is around Z = ~21. **Important: While moving the sample by controlling the navigator, simultaneously observe the sample on the sample stage. If you stage or the optics don't move, click on the X-Y Safety Disabled or Z-Safety Disabled dial to activate.**
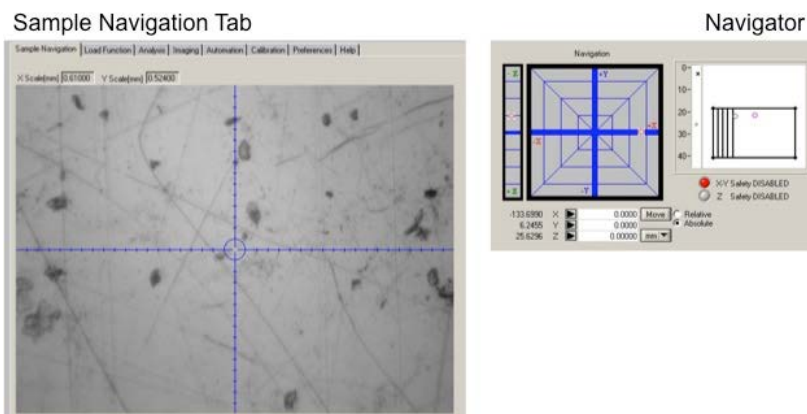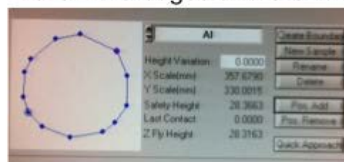

2. Move the stage until you see the edge of the Al standard, click New Sample on the Samples area. The sample name will be automatically assisnged, such as Sample 2. You may give a new sample name if you'd like. Define the sample boundary by scanning the edges of your sample. As you move around the edge, click on "Pos. Add".

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Move to edge of sample

Click New Sample and Name

Follow the edges and click Pos. Add

3. After you define the Al standard sample and boundary, move the stage by using the navigator and bring the Quartz standard under the optics and focus (Z=~22). **Important: While moving the sample by controlling the navigator, simultaneously observe the sample on the sample stage.** Define the other samples accordingly.
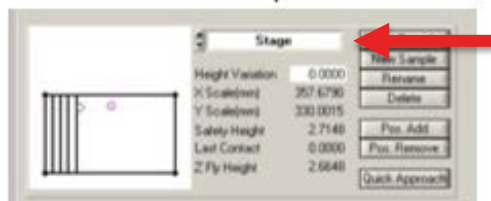
4. You may also define an area of interest on a given sample by using the navigation tool and focusing on the sample surface. An alternative way to create a testing area is to use "Create Boundary" option, which automatically creates a rectangular area in the visible area of the sample surface.

5. If the stage doesn't move in the X,Y direction or Z direction, it could be because stage safety is enabled. This action prevents the stage from moving. To enable the stage movement, make sure X,Y Safety DISABLE or Z Safety DISABLE radio buttons are selected and indicated as RED. This section is located at the top right corner.
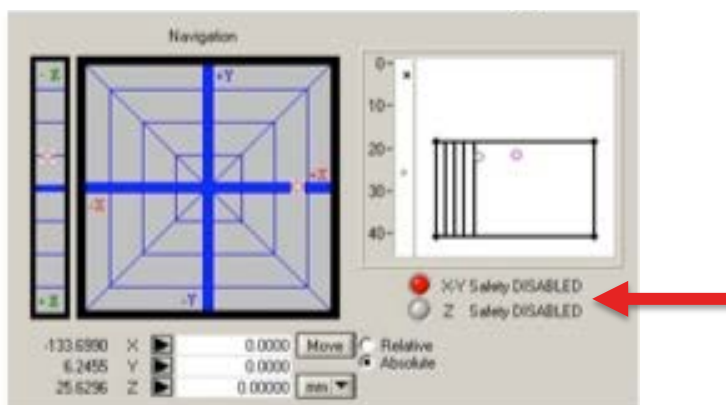

6. When all sample boundaries are defined, you may toggle between samples using the drop-down menu. On a given boundary, you may right-click to a point to bring this position under the optics.

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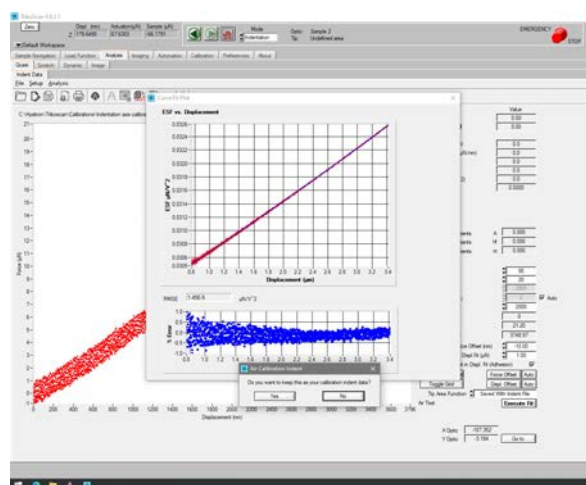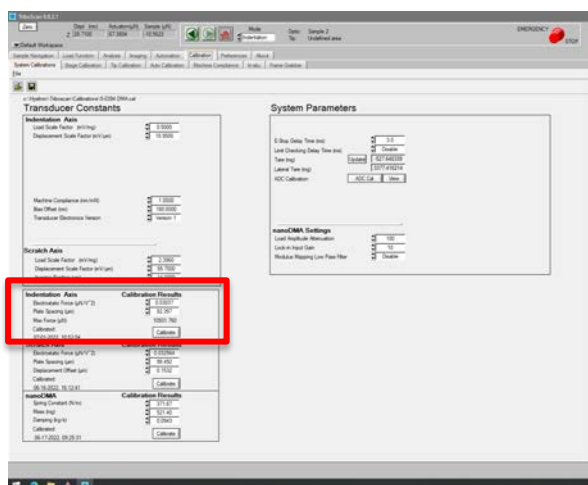Contact: Deniz Rende, PhD, rended3@rpi.edu

Transducer Calibration – Air Indent Calibration

This is to calibrate the spacing between the drive plate of the transducer and the center electrode. Before starting the calibration, make sure the transducer load is zero. Click on the ZERO button at the top left corner of the software.
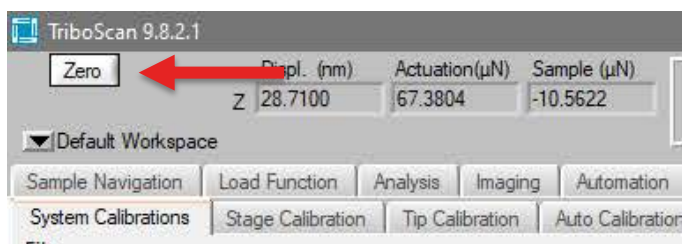
1. On the Calibration tab, click on calibrate on the Indentation Axis part. It will navigate to Load Function tab, as the peak force is set to 1300 uN. Click on Cal. Air. Ind. Button.
2. When the calibration is done, the electrostatic force vs displacement plot automatically appears. On this graph, the actual measured data (red) should be close to the fitted linear model (blue). Accept to keep the air indent data and close the window.
3. On the Calibration tab, the plate spacing under Indentation Axis part should read between 80-90 um.

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Optics-Probe Tip Calibration

This is to calibrate the spatial locations of the optics and the probe tip. The software must know the X, Y and Z distances between the sample stage and the indenter tip. **This calibration needs to be performed only when the transducer or the probe is replaced. Otherwise, you may proceed to the next steps.**

1. Bring the sample under the optics by selecting the Al standard and right clicking on a place inside the boundary on the sample area. You will see many dots and patterns, pick a clean area on the Al standard surface using the navigator.
2. On the software, switch to Calibration > Stage Calibration tab > Slot 1 Tip to Optic Calibration button.
3. Select New "H" pattern, this will create a pattern composed of 7 points. Focus the optic.
4. Adjust the force to 8000 µN.
5. The instrument will now move the stage to bring the sample under the nanoindenter probe and stop. You will see a warning, "Use the Z navigation control to move the tip down approximately 1 mm above the sample". **Important: While moving the sample by controlling the Z navigator, simultaneously observe the sample. As you approach the sample, slow down the navigator by moving the red marker to center position on the Z-axis navigation bar. Very slowly approach to sample, when you see the reflection of the tip on the Al standard, stop.**
6. After adjusting the probe tip, click OK on the window, this will start the calibration.
7. During the calibration, the Tribodenter will automatically approach the surface. After the indents are complete, the stage will be moved back under optics. When OK is clicked, the software will calculate the position of the optics and position of the center indent.
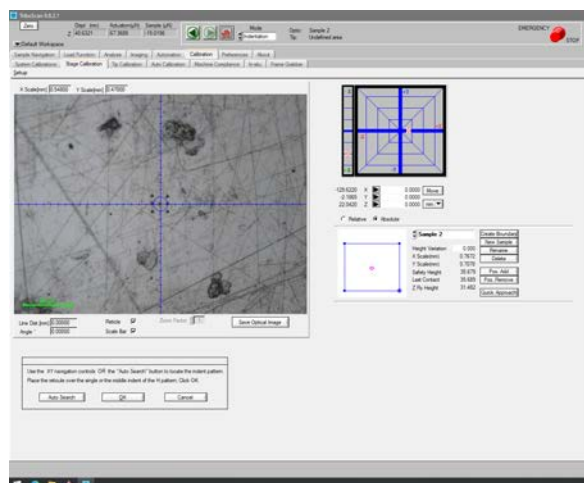
8. H-pattern is a symmetrical pattern, it is hard to understand whether the H-pattern is created by the current user or previous users, so as a rule, to indicate the H-pattern is used, after the calibration is completed, position the navigator to make one more single pattern.
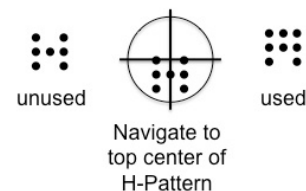
9. After you position the optics to a single point, Restart the optic-probe tip calibration, switch to Calibration > Stage Calibration tab. Click on the tip to optic button. Select "New Single Indent" button. Focus the optic.

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10. The instrument will now move the stage to bring the sample under the nanoindenter probe and stop. You will see a warning, "Use the Z navigation control to move the tip down approximately 1 mm above the sample". **Important: While moving the sample by controlling the Z navigator, simultaneously observe the sample. As you approach the sample, slow down the navigator by moving the red marker to center position on the Z-axis navigation bar. Very slowly approach to sample, when you see the reflection of the tip on the Al standard, stop.** After adjusting the probe tip, click OK on the window; this will start the single indent calibration.
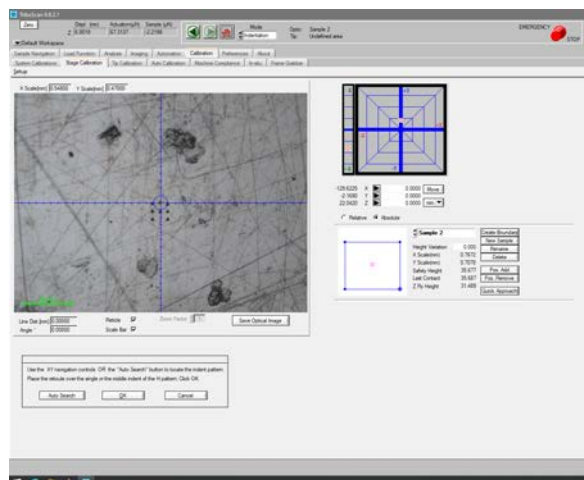
## Sample Testing – Single Point Testing

1. Go to the Sample Navigation tab and bring the sample under the optics by selecting the sample and right clicking on a place inside the boundary on the sample area.

2. Click "Quick Approach" button, this will direct the sample under the indenter, and automatically approach the surface. Upon touching and recording the surface position, it will retract back to the position.

3. Switch to Load Function > Indentation tab. You may either create a new load function or call a re-saved load function. Set up the load function parameters. Zero the transducer.

4. Click on Perform Indent button to start testing. During testing, the sample will be moved under the tip, the probe will automatically approach to sample surface, then the progress will take 60 s to settle the motor controller and 40 s for drift monitor.

5. Upon the completion of testing, you will be prompted to save the measurement file. Each user has a folder under C:/Hysitron/Triboscan/Users/username. Please create a folder under your full name or username and use this folder to keep your data.

6. On the measurement graph, you may click on Execute Fit button to calculate Er and H.

Sample Testing – Automated Testing

Automated methods are used to generate high-throughput data and allows users to get complete characteristics from the samples.

1. To initiate an automated method, go to Automation tab and bring the area of interest under the optics.

2. Create a New Method and call a previously recorded method.

3. Create an array pattern by providing parameters such as indent spacing, number of points in rows and columns. Click on Update grid.

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4. Select the Target Position at the current optic.

5. Select a predetermined load function, save the workspace and start the method.
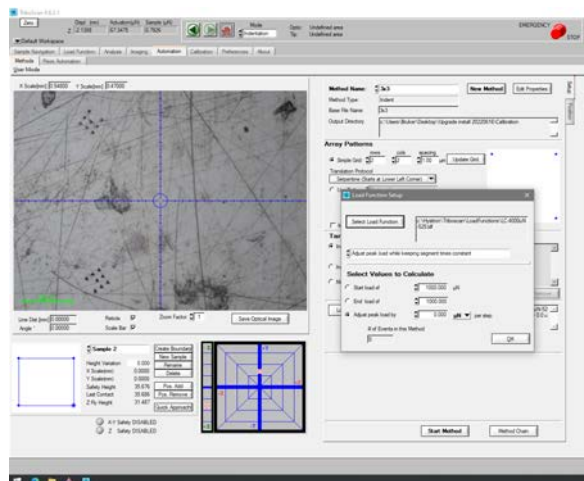
6. When the method is completed, you may plot and analyze all measurements under Analysis tab.

Sample Testing – Imaging

1. Optically locate the area of interest within a defined sample boundary.
2. On the "Imaging" tab click the Sample Approach button. The system will move the stages to bring the probe into contact with the sample surface at the default setpoint value of 2µN.
3. When the Progress window closes, click the Start Scan / GO button to start scanning the surface.
4. Image parameters such as scan size and scan rate can be changed while scanning. 0.5Hz and 10 micron scan size is good to start with. Don't change the set point force and integral gain values.
5. To adjust the image contrast, select the image that want adjusted by clicking one of the image types: Tf (Topography forward), Gf (Gradient forward), Tr (Topography reverse) and Gr (Gradient reverse) and click and drag the dark blue bars below the histogram plot. Click and drag the light blue bar to move the range. Do this for each image that you want to use.
6. You may save images by clicking Capture Image / Camera icon.
7. To perform an indent, set up the desire load function in the "Load Function" tab
and in the "Imaging" tab, click the indent icon and system will perform indentation exactly in the center.
8. When the imaging is completed, Stop the scan and withdraw from the sample.

Shut Down Procedure

1. Exit from the software, do not save the workspace.
2. Turn off fiber optic illuminator and vibration isolation control unit.
3. Remove your samples.
4. Log out from computer
5. Fill the logbook.

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Quick Testing Guidelines

1. Log on to the computer using your RCS ID and password.

2. Turn on the fiber-lite and vibration isolation controller.

3. Place your sample on the stage
(Unsure? Go to Mounting samples section on the stage on page 2).

4. Initiate the Triboscan software
(Unsure? Go to System Preparation steps on page 2)

5. Describe your sample boundary, move your sample under the optics, using create boundary, create a sample testing area.
(Unsure? Go to Sample Definition and Boundary Selection on page 3)

6. Go to Calibrations tab, Stage Calibration subtab and complete Indent Axis calibration.
(Unsure? Go to Transducer Calibration – Air Indent Calibration on page 4)

7. Go to Load Function tab, and make a "quick approach" on your sample
(Unsure? Sample Testing – Single Point Testing on page 6)

8. Once the sample is back under optics:
>   a. For a single test: select a new nearby position and setup load function parameters or open a load function.
>   b. Automated Run: Go to Automated Testing Tab and setup an automated run.
>   c. Piezo Automated Run: Go to Automated Testing Tab > Piezo Automation and setup an automated run (this option limits the testing area to 10um x 10um)