Standard Operating Procedure of Triboindenter (Hysitron TI 950)

I Sample Loading and Preparation

DO NOT TOUCH the bottom of transducer and optical microscope. Always place the tall samples on the most right location and in the corner. Sample should be placed on the metal pucks and be super glued. Just wiggle around your sample just to ensure that it is not moving freely.

II Setting Safety Limits and Defining Sample Boundaries

Under the "**Sample Navigation**" tab, using the stage controls area, move the X, Y and Z axes of the sample stages to locate a corner or edge (for transparent samples) or center of the sample (opaque samples) and adjust Z-axis for the corner or edge of the sample to be in focus. **CHECK the X-Y SAFETY DISABLED. DO NOT CHECK THE Z-SAFETY.**

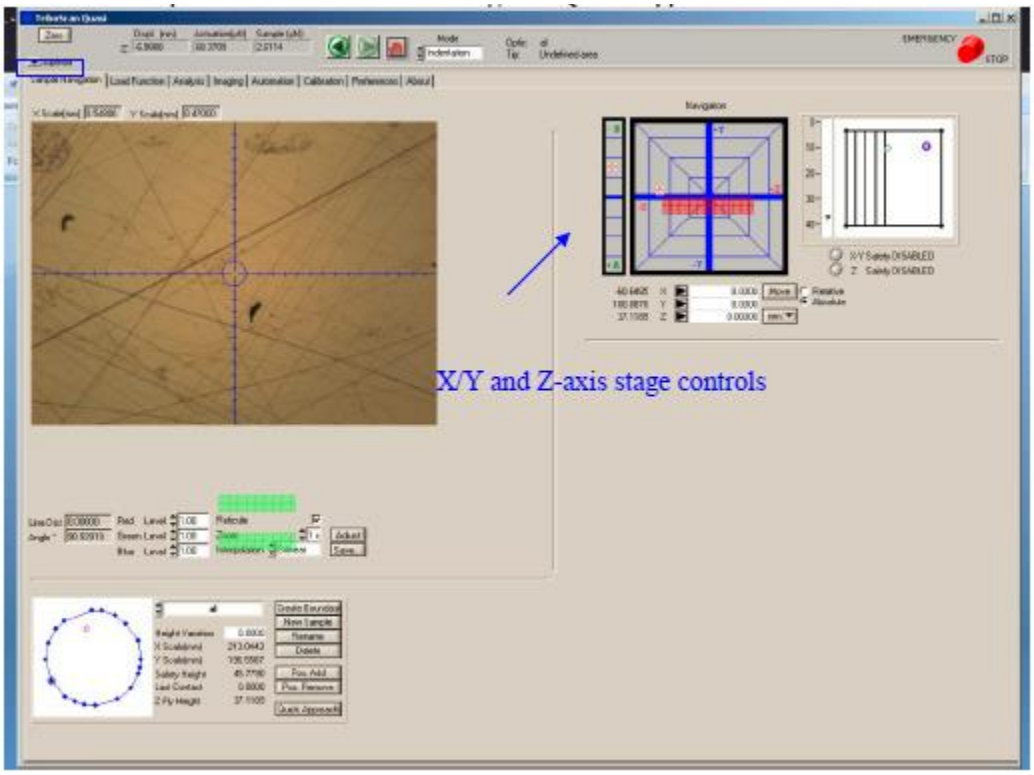
It is important that **the surface of the sample be in focus when the boundary is created**, especially when the sample is translucent as the bottom surface can easily be mistaken for the upper surface resulting in tip damage. Once focused, just go little bit outside of the corner or edge and **click the "New Sample" button**. **Select a name for the sample and click OK**. Click the "Pos. Add" button to add a position to the sample boundary. Square/rectangular shaped samples boundary can be defined by four corner points while circular samples boundaries should be defined by multiple points.

Using the X/Y-axis stage controls, move the optics over a different corner or other edge location and click "Pos. Add" until the entire sample boundary is created. Repeat the steps for each additional sample or sample boundary area. **Right-click within the sample boundary area to move the optic to the desired location.**

ANOTHER option of defining the safety limits both for smooth and rough surfaces: For smooth surface, instead of defining the corners, just use the optics and bring the sample field of views where you want to indent and click on **CHECK BOUNDARY**. It will create the small area where you can indent within. If you have ROUGH sample and focus is changing from position to position across the sample, try to bring area in field of view and click on CHECK boundary.

NOTE: **Transducer is coming from left to right, so if your sample is non-uniform in height, please make sure that higher end of rough side remains on your right**.

Uncheck the X-, Y safety DISABLED. If testing will be performed from the optic position, the actual height of the sample MUST be measured by adjusting the sample to be in focus and clicking the "Quick Approach" button.



III Transducer Calibration

Open the **Tip calibration** file (C:\Hysitron\triboscan\Area Function\), and load the specific tip file.

"System Calibration" click "**zero**", to make z axis zero.

"System Parameters" click "**Tare Update**".

Under the "**Calibration" tab, "System Calibration**" sub tab, click the "**Transducer Calibration**", "Indentation Axis Calibrate", click the "**Cal Air Indent**" button and the reminder window will pop up, click "**Start**" to continue. When the calibration process is done, click "Yes" when asked if you want to keep this as your Air Calibration Indent data.

IV Stage Calibration, make H pattern

Sample Navigation. When you perform focus step, first click –z make z distance between 5-7, then click +z to do focus. Decrease the z speed if you see the light is coming, which means focus is close. After you finish the focus, move the x and y axis to the sample interest. It is helpful for focus if change 1x to 3x and 5x. Disable X and Y (No red), then create a new boundary.

Go to the "**Calibration**" tab, "**Stage Calibration**" sub tab
"**Tip to Optic Calibration**"button (near the bottom left) to make a New "H" Pattern button. Click OK.

The software will prompt the user that the **Z Safety is disabled** and will move over

a defined sample space. Click OK.
When prompted, enter a peak force of 8,000 μN and click OK. The system will move the sample under the probe.
When prompted, manually lower the Z-axis until the probe is approximately 2mm above the sample. And Click OK.
The system will automatically approach the surface and make seven indents in the shape of an H. After the pattern is done, the system will move the location back to under the optics.

Locate the H pattern that is recently made using the X/Y-axis stage controls, and ***place the cross hair (x) on the center indent of the H. Click OK. The system have now proper tip-optics calibration.***

Go to the "**Load Function**" tab and "**perform Indent**" sub tab to define the load function. After finish, save as file 8.

Add curves, multi-curve analysis.

III Performing Indentations and Imaging
There are 4 different ways to perform indentation tests. A single indent can be made either from the optic location or from the center of an imaging scan window. The through optic indent is generally for the samples that are homogeneous while the imaging indent is useful when user wants real time image of the indented location. Multiple indents can be made by setting up automated methods or piezo automation. The latter are always set up and executed from the imaging position when the probe is in contact to the surface while the latter from the optical position.

(1) Single Indentation on Optical Position

Move the optics over a desired sample location. Do "**Quick Approach**" first.

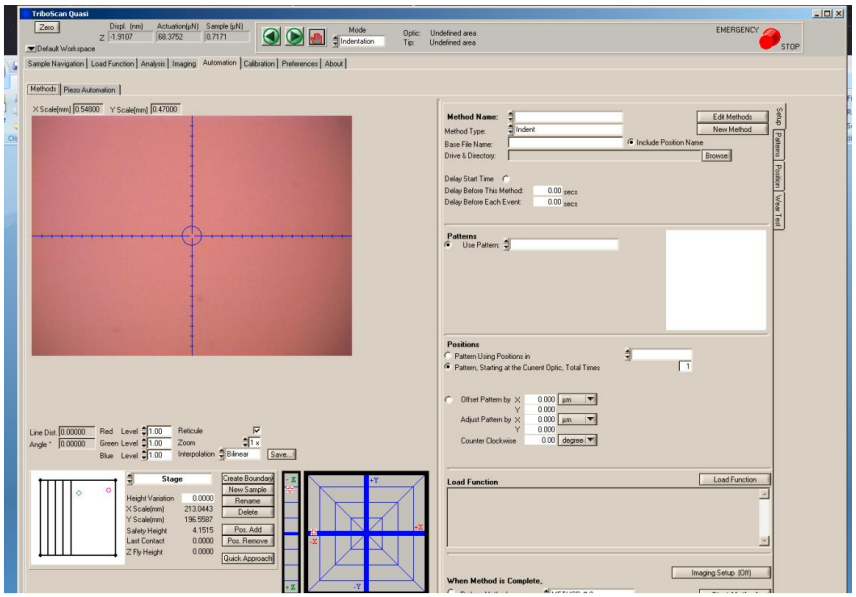
Go to the "**Load Function**" tab and **perform "Indent"** sub tab to define the load function.

Open the trapezoid load function file and modify the parameters as desired by left-clicking the segment.

Click the Perform Indent at the bottom of the load function window to start the test.

Save the result file. Click **Execute Fit** to calculate the reduced modulus and hardness from unloading data.

(2) Making Array of Indents on Optical Position
Automated methods are always configured and executed from either current or predefined optical positions. This automated section can be found under the "**Automation**" tab and "**methods**" sub tab. The left side of the "Methods" sub tab is nearly identical to the "Sample Navigation" tab to allow the user to navigate the sample space, define samples and perform other functions without the need to toggle between tabs. The right side of the "Methods" sub tab consists of four side tabs (Setup, Patterns, Positions and Wear Test) that are useful for performing multiple automated tests.

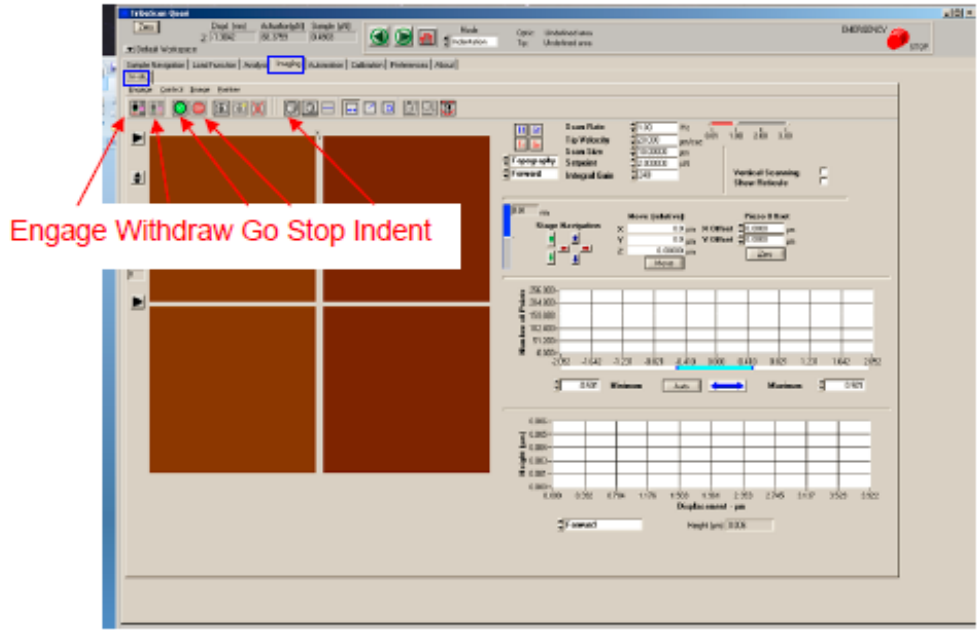


- Go to the "**Automation**" tab and "**Methods**" sub tab. Set the array of indents by clicking on the "Patterns" side tab.
- Select Grid and choose the number of indents in X and Y and then click Create. Assign a name to the pattern when prompted.
- If the pattern will be performed at more than one location, select the "**Position**" side tab, create a new Position Group, move the X/Y-axis stages to define Positions where the pattern will be performed *(not required if performing the pattern at one location)*.
- Select the "Setup" side tab. Click the **New Method button**, select a name for the method. The Method Type is Indent.
- Set the Base File Name for saving the files. Each file will automatically have a number assigned at the end of the file name starting at 0000. Select the directory to save the files by clicking the Browse button.
- Select the Pattern that is previously created.
- Under the Position section, in case there are multiple positions for the pattern to be made, select "Pattern Using Position in" and choose the group you want to test. If only the current optical position to be performed, select "**Pattern, Starting at the Current Optic**", total times 1, and make sure the optics are located over the area of interest.
- Click the **Load Function button**. Load the desired load function by selecting the Select load function button. Choose how to modify the load or displacement over the range of tests with the radio buttons and start/end load values. Click OK.
- Save the Workspace by clicking the small down arrow in the upper left of the main window. This saves all automated methods, positions, and patterns.
- Click the Start Method button and follow on-screen instructions.

(3) Single Indentation during Imaging

- Optically locate the area of interest within a defined sample boundary.
- On the "**Imaging**" tab click the **Approach** button (See Figure below) or from the menu bar click **Engage** Approach. The system will move the stages to bring the probe into contact with the sample surface at the default set point value of 2µN.

- When the Progress window closes, click the **Go** button (See Figure below) or from the menu bar click **Control Start Scan** to start scanning the surface.
- 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.
- During scanning, click **Image Background subtraction** and select **Linear Regression**.
- 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 you want.
- You can play with offsets to shift the image if current location is not good.
- DON'T use sample navigation arrows here.
- 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.
- Save the file when done. Click **Execute Fit** for reduced modulus and hardness calculations.
- After indent is complete, tip automatically scans the surface and you can view the indentation mark and capture the image. Images can be saved by clicking the Camera icon or Image Capture from the menu bar. **MAKE SURE that you define your capture directory here.**
- To leave imaging mode, **click Stop button to stop scanning and withdraw** the probe from the sample surface by clicking the **Withdraw** button.



Every time when the user finish, magnification with 1x, and delete boundary.

Turn off v-Lux 1000, always make Table Stable on.