This quick guide provides brief instruction on handling the nanoindenter, model Hysitron TI 950. For more details, the user is encouraged to visit the manual on the official Bruker webpage.

NanoIndenter - Hysitron TI 950
Analysis Mode: Indentation-Quasistatic

Warnings:
- Transducer and tip are the most delicate parts of the instrument, so never touch them.
- The user is not allowed to perform any calibration other than the Transducer Calibrations (Cal. Air Indent).
- In case of any unexpected dangerous situation, click on the EMERGENCY STOP button (up-right corner).
- Make sure fo follow the orders when switching on/off the instrument.
- Do NOT insert any insecure flash drive or external hard disk to the Hysitron system.

Switching on the instrument
1. To access the controllers, open the lower door, and turn on the controller.
2. Toggle Vibration Isolation system to the Enabled mode and push the red button to the illumination of green and orange LEDs.
3. Power on the CCD light source (optical microscope illumination). For automation or long period measurements, power it off.
4. Turn the vacuum pump on (on the PC case), in case you need to mount the sample on the vacuum chucks.

PC and software
1. Usually, the PC and the controller is on, otherwise, turn the PC on (for username and password look at the PC monitor leg). Then, on the desktop, open the **TriboScan** software.
2. In the popup, click **Analysis only** if you need to analyze the result, otherwise, **Cancel** or wait until the window disappears.
3. In the **Transducer Select** window, select proper configuration based on the mounted transducer. Check through the window.
4. The stage is initializing and X, Y, and Z-axis are homed (0,0,0 position). Notice that the tip and optical microscope are moving in the Z-axis, while the stage is moving in the X and Y-axis.
5. Keithley controller (used for nanoECR mode) message pops up. Confirm the message by clicking OK and then click NO.

Mounting the sample
1. First, check what is placed on the stage and ensure the clearance of the moving parts (Do NOT touch the **Al** and **FQ** samples).
2. Click on **Pause TriboScan** to suspend the system to avoid any violations or damage during the sample mounting.
3. Mount the sample on the stage. You can use magnetic pucks, vacuum chucks or clip (see the file **Useful Information**)
4. Arrange the samples based on the height (shortest on the left and tallest on right) with a 3 cm distance between them.
5. When the mounting is done, click the **TriboScan continue** to activate the system again.

Transducer calibration
1. In the Transducer Calibration\Indentation Axis click **Calibrate**. It will navigate you to Load Function\Indentation. **Peak Force** value must be ~ 1500 µN for 1D transducer.
2. There, click **Cal. Air Indent,** and in the Single Indent popup window click **Start**. This is for the calibration of the transducer.
3. After calibration, a Curve Fit Plot will show the **ESF vs Displacement** profile which in an ideal case it should be linear, representing a sharp tip. The red plot is data and the blue one is the fit. The RMS value should not exceed 1.8×10^{-5}.
4. If it is so, click **Yes** in Air Calibration Indent popup window.
5. You are navigated to Analysis\Quasi\Quasi. The **Force vs Displacement** profile is plotted which exhibits a hysteresis behavior.

Sample navigation
1. The **Sample Navigation** tab consists of three main areas; Live video, Stage control, Sample boundary. The **Stage Controls** area allows the user to move the stage in X, Y, and Z-axis with proportional speed, represented by four segments. The speed increases as moving to outer segments. The **X-Y Safety DISABLED** and **Z Safety DISABLED** buttons should be activated (red) when you need to create a sample boundary, where the stage can move. Generally, this safety feature is designed to prevent any likelihood of a probe crash. After the sample boundary has been created the stage will be updated to show an outline of

the current sample spaces in blue. The optics is represented by a pink circle and the nanoindentation probe is represented by a red diamond.

2. Move the stage until you see the sample under the optical microscope. **Warning:** Focus on the edge of the transparent and middle of the opaque sample. To focus, +Z moves the probe and optics down (closer to the stage), and -Z moved them up (away from the stage). When you are in focus, activate the Z Safety DISABLED (red) to lock the stage movement in Z-axis.

Sample boundary

1. Once again, check if you are focused on the surface of the sample (be careful with the transparent samples). Go to the **Sample Navigation** tab and open your **Workspace**. If this is the first time you work with Hysitron, click on the roll-down button and create **New Workspace**, then next time(s) you will work on this workspace. The Workspace should be saved as your surname_user number (i.e. Kolar_12345). Now you can find your sample name if you click on the up/down arrows in the down-left corner of the window.

2. You see the live video of the surface. It is possible to create both regular or irregular boundaries on the sample. Click on **Pos. Add** to create boundary point by point all around the sample or just a part of that. The **X-Y Safety DISABLED** must be deactivated to allow the stage movement in those axes. Right-click inside the created boundary to move the optics to the corresponding location. Create a boundary for each sample.

3. Double-check if the sample surface is in focus, then click **Quick Approach** and Ok for each sample. This is to define the tip to sample distance, though, the stage starts to finely move down until it contacts the surface.

Indentation mode: Quasistatic partial unload

After setting the indentation area, go to Load Function\Indentation**Automatic Save** and click **Enable** to save the results as a file. Find your folder and click Done without typing a filename. Now in the popup window type your filename.

Single Indent: In Standard Load Functions click on **QS partial-Unload** and check the load function setup parameters, especially Peak Force to be correct (1200 µN for 1D transducer is recommended). Click **Perform Indent** and then **Start**. It will take about 3 minutes for drift correction and then indentation starts. After indentation, you will be navigated to Analysis\Quasi\Quasi, where you can analyze the Force-Displacement curve. Click on the analyze icon and **Add** the file you've created and 📊 then hit **OK**. To update the graph after each fit, click **Yes** and then insert the number of segments in each load-unload part (for QS usually it is 3). You will see that for each curve in the Force-Displacement profile after fitting, the value of **Er** and **Hardness** are changing. After fitting, a window of Modulus and Hardness vs Contact Depth will appear. If the Zero point in Load-Displacement profile is not in Zero, click in a particular point which seems to be zero, and then click Displ. Offset to make that point the zero value of displacement. Click on the icon Hardness and modulus plots and select the generated TXT file to see your analysis results. Also, you can extract this file from your directory. You can also export Force-Displacement raw data. Remember that the software will generate the TXT raw data with the same name as the HYS file. Then, to rescue your Hardness and modulus plots, move them to a new folder. Go to Analysis\Quasi\Quasi\File and click **Export Multiple Text Files**. Select HYS file, Add, and then Ok.

Multiple Indents: Open your workspace where you see the live image of the sample surface. Create boundaries for each sample. To perform an array of indents, go to Automation\Methods and in **Patterns** tab create your desired pattern after focusing on the surface. In **Setup** tab click the New Method, then name your method to use it for future projects and save it through Drive & Directory just in your own folder. Method Type is Indent and Base File Name will be numbered as the filename000 and on. In Use Pattern select the previously created pattern in Patterns tab. Check Patterns Using Positios in and select the position. Finally, click Load Function and choose a standard load (recommended) or modify your own load function and click OK and save workspace.

Switching off the system

Follow based on the order. Firstly, close the TriboScan software by clicking on ❌ and then in the popup window, **Yes**. You may want to save the workspace for future use, then click **Save**. You will see the stage is homing. Turn onn the monitor. Push the red button to see the illumination of green and orange **LED**s is fading away, then, toggle the **Vibration Isolation** system to **Disabled** mode. Power off the CCD **light source** and **vacuum pump**. Never turn off the PC and the piezo/transducer **control unit** unless there is a extended luckdown, otherwise both PC and controller should be on.