# Agilent 630 Cary FTIR

Explainer

IR spectroscopy is a powerful tool for analyzing chemical structure, because it enables understanding the vibrational modes of molecules. The frequency of molecular vibrations depends upon the types of chemical bonds present in a molecule. The frequencies of certain vibrations are characteristic of functional groups or specific bonds; stronger bonds will vibrate at higher frequencies than weaker bonds (i.e. C-O bonds vibrate at lower frequency than C=O bonds), and bonds to less massive atoms will vibrate with higher frequency than bonds to more massive atoms (i.e. C-H bonds will vibrate at higher frequency than C-C bonds). Given the dependence of vibrational frequency on chemical structure, we can utilize IR to determine functional groups of molecules in a sample.

Given the power of IR spectroscopy, we have the Agilent Cary 630 IR to enable obtaining IR spectra. The Agilent IR has two modules - an ATR module for liquids and solids, and a "Dial Path" module for absorbance spectra of liquids, films, and gels.

Running a Sample

1. Check that the instrument is on by looking at the power button on the top of the instrument. The LED should be green. If it's red, turn it on by holding the power button down for 2 seconds.

2. Check that the appropriate module is installed - we have two modules, the ATR and DialPath module. If the module is not appropriate, then switch the module following the instructions in the section below.

3. On the computer, open the "MicroLab" software
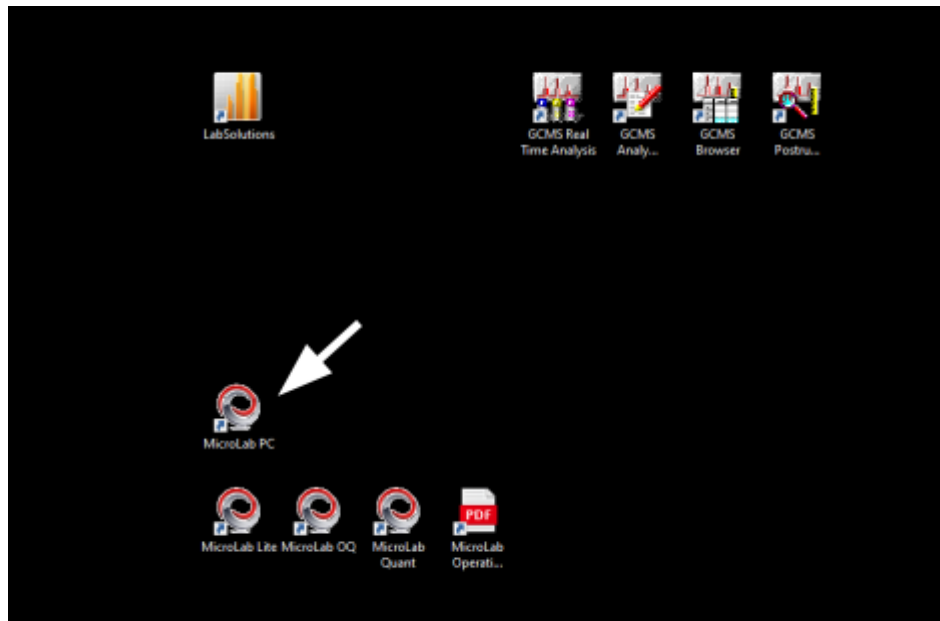
4. If it asks for a username and password, enter McNeil-Group and #gr8dientcopolymer for each. Otherwise, the window should look like:
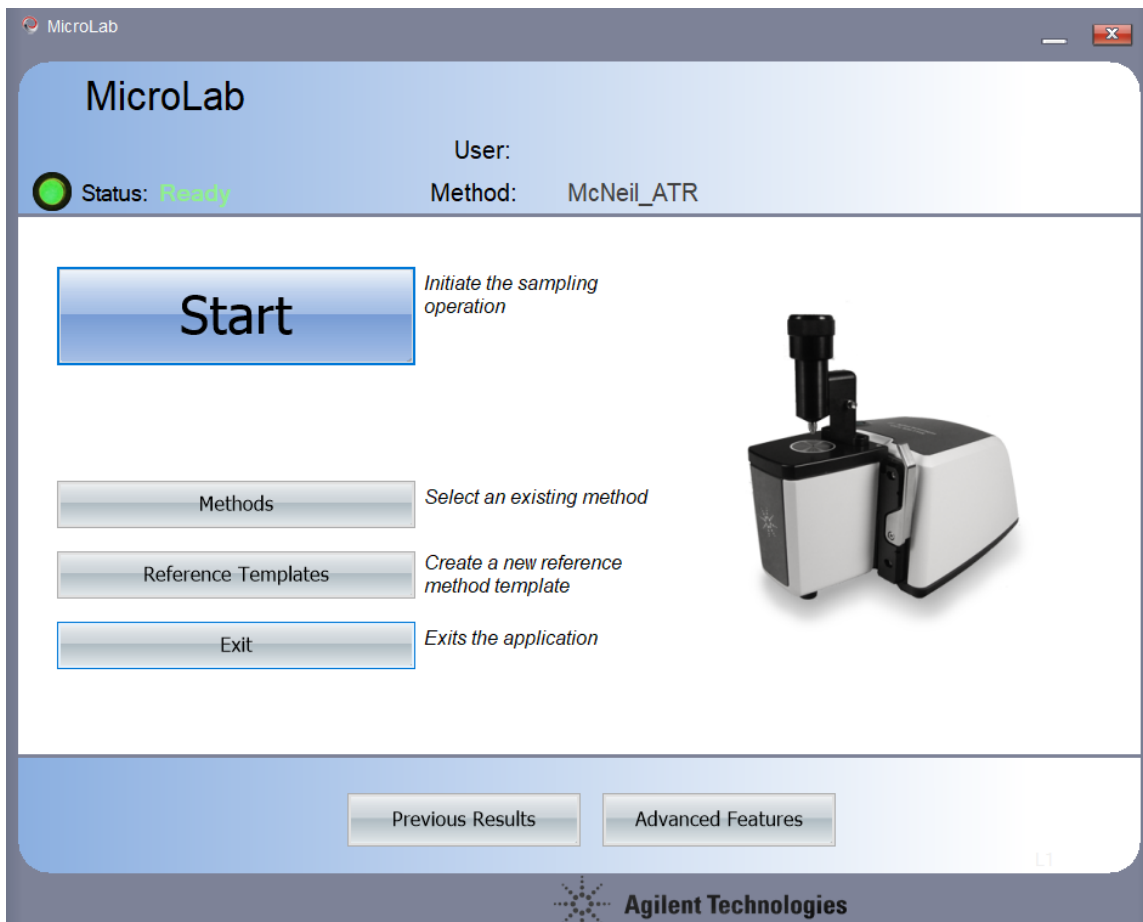
5. Check that the module on the screen matches the module installed on the instrument. If it does not, reattach the module on the instrument, or reload the software.

6. Click the methods button. General methods for the group are labeled "McNeil" followed by the module name. Other methods have been developed by the group or came with the instrument.
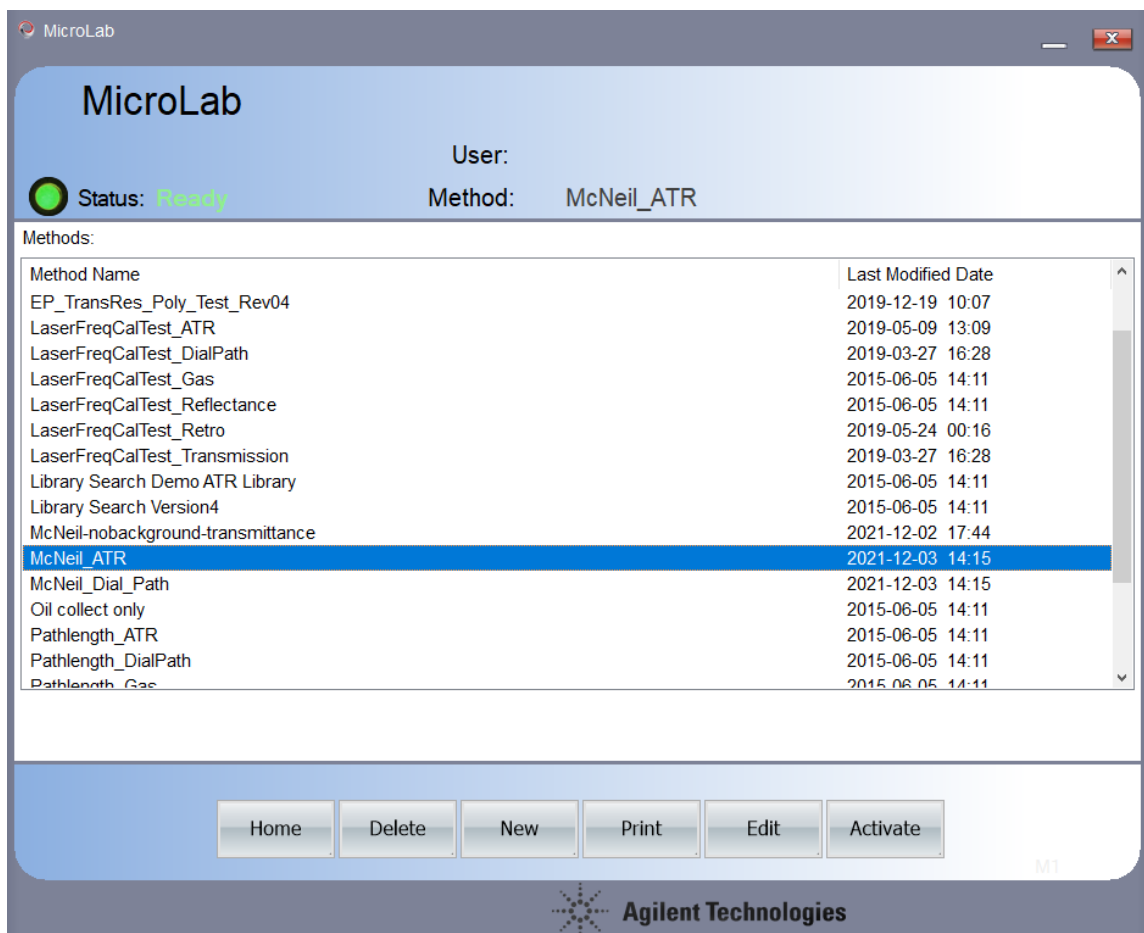
A good default method is McNeil_ATR or McNeil_Dial_Path (depending on which of the two modules you are using). The only difference between these two is the module specified in the parameters, and that the ATR data is plotted in **transmission** mode, while DialPath is plotted in **absorbance** mode.

After you find an appropriate method for your sample, click on the method and then choose "Activate" to bring you back to the home screen.

7. Once you're on the home screen, click start. The instrument will walk you through the process, first by having you clean the crystal.
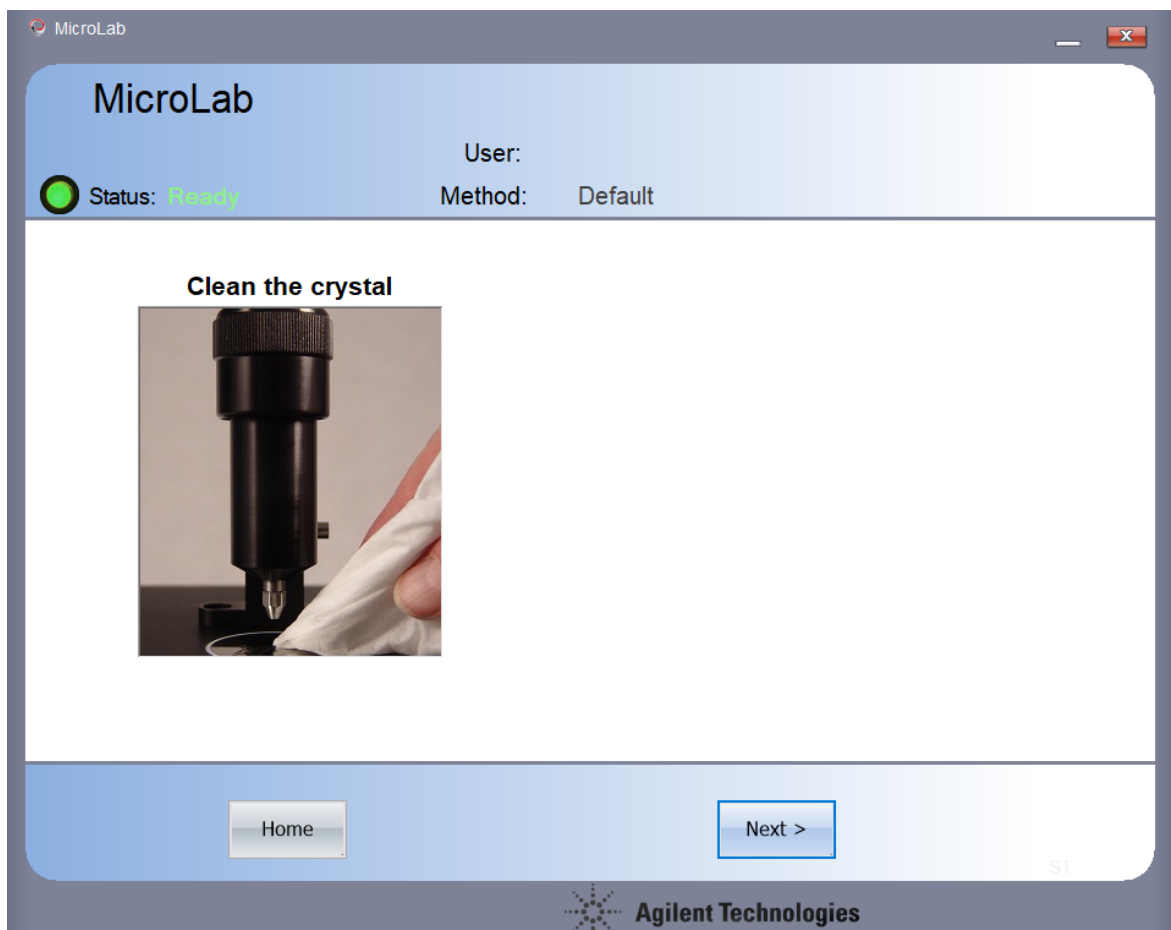

Any solvent may be used for cleaning the ATR, since the window is made of diamond (therefore, it is chemically resistant). The DialPath has less chemical resistance (because its window is made of ZnSe), therefore, only use solvents that are compatible with ZnSe optics.

After cleaning the ATR, leave the module in an open position (do not turn the anvil down).

After cleaning the DialPath, turn the knob to any path length (a longer path length will give a better background).

Click "Next". It will then verify that the crystal is clean and then (depending on the method) take a background.

8. The next steps involve placing the sample over the window, and adjusting the accessory.
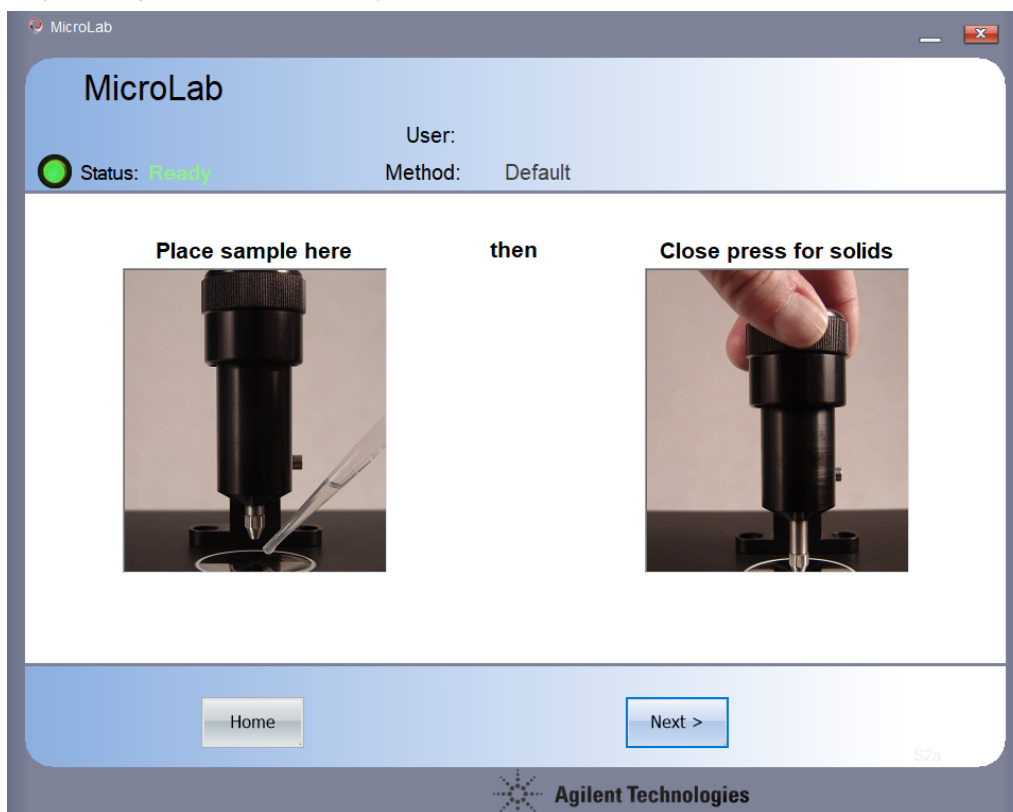

For the ATR:

- liquids and solids should go directly on the window.
- **for liquids, do not press the anvil down** - this will push the liquid away from the window and give you less signal
- **for soft solids, press the anvil down** by turning the knob at the top of the ATR. Turn the knob until you feel or hear a click.
- **for hard solids (e.g., glasses, large crystals)** grind the solid up into a fine powder using a mortar and pestle **before placing it on the ATR window**. The diamond window is very thin, and hard solids may cause the diamond to break (but they won't scratch it lol). After you have a fine powder, place it on the window and press the anvil down, **but only turn the knob until it makes contact with the sample.**

For the DialPath

- liquids and films should be placed directly on the window
- after placing the sample, turn the knob to the desired path length

After placing the sample, click "Next".

9. The program will then switch to a live-view of the sample. On this window, you can:

- check that the peaks are appropriately shaped
- there are no contaminants (particularly pay attention to the 2300 cm-1 region, since that is where CO_2 appears)
- and that the background looks appropriate (if there are peaks with absorbance >100, or that are greater than the baseline around it, then you may want to retake the background).
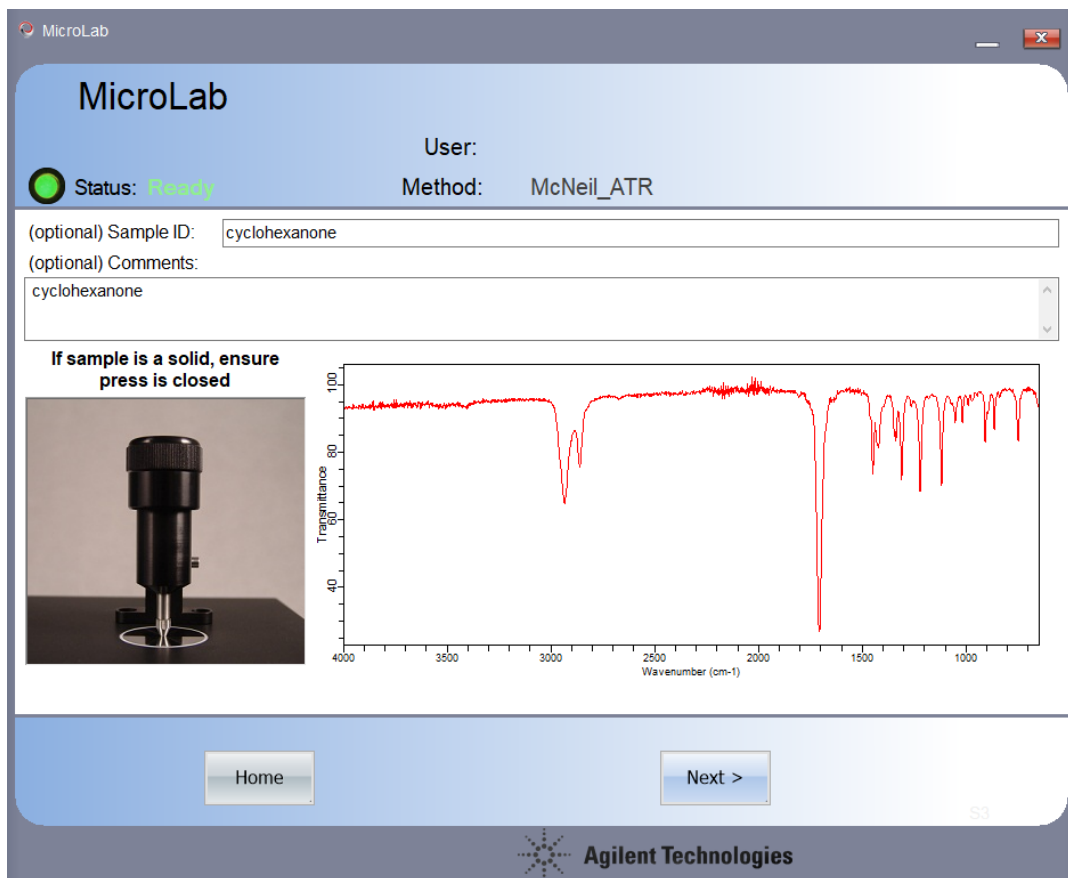

If the live view looks okay, then give the sample and ID and comments (Note: The ID and comments do not have to match like above). Then click "Next". The instrument will then collect the data.
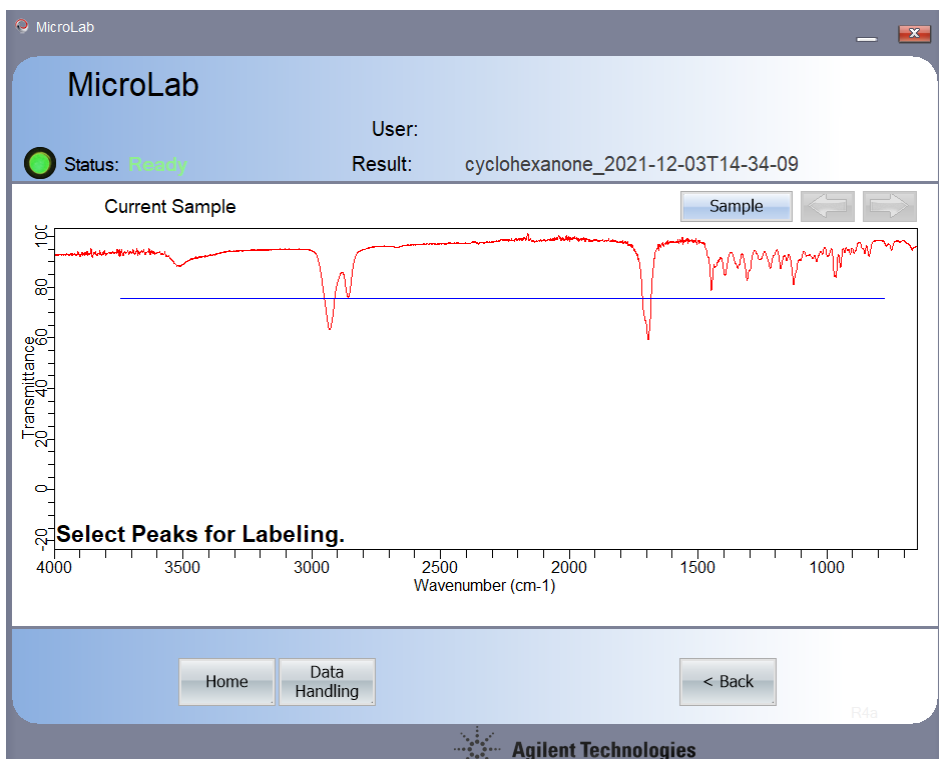
10.   After data collection, the spectrum will be displayed. Note: If the whole spectrum is not displayed, it is likely displaying a "Component" based on what the method specified. To see the whole spectrum, click   the   "Sample"   or   "Component"   button.
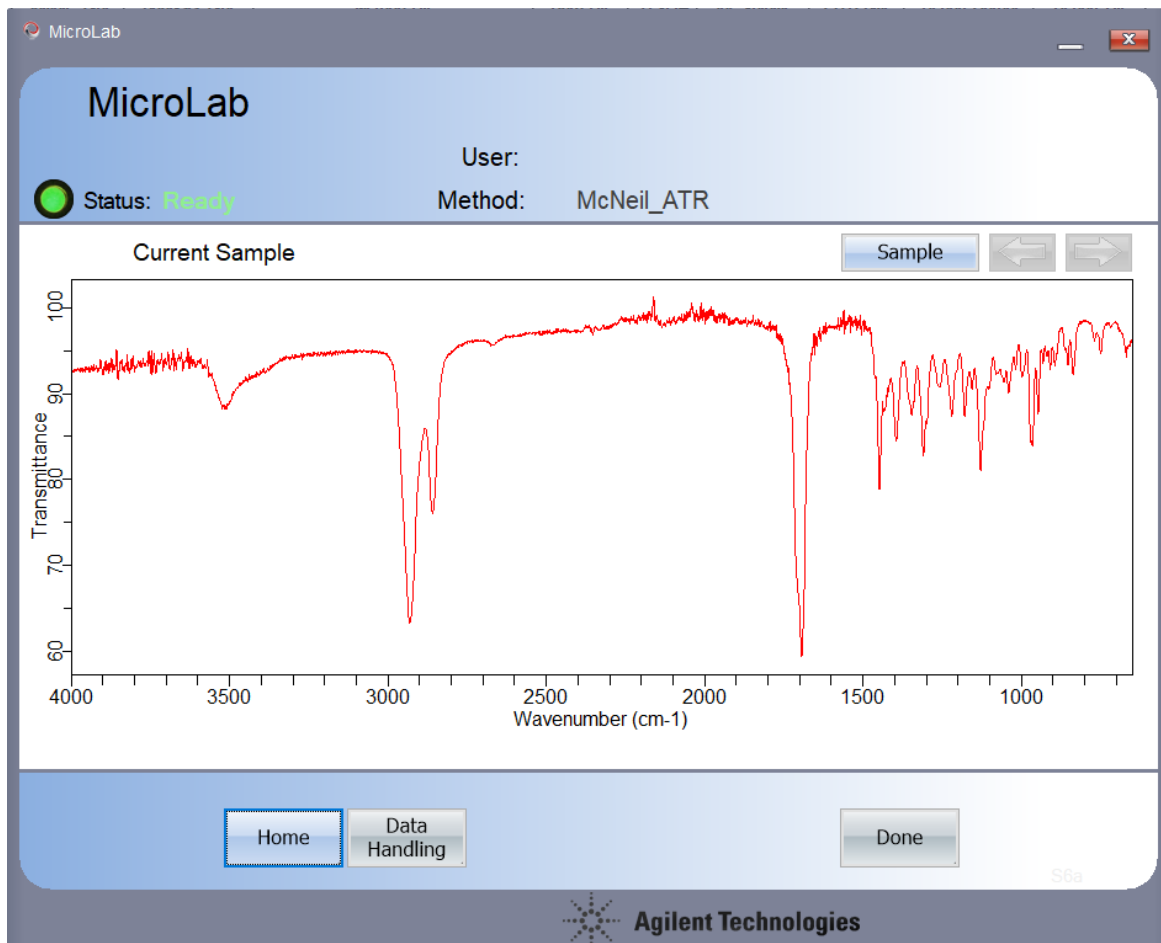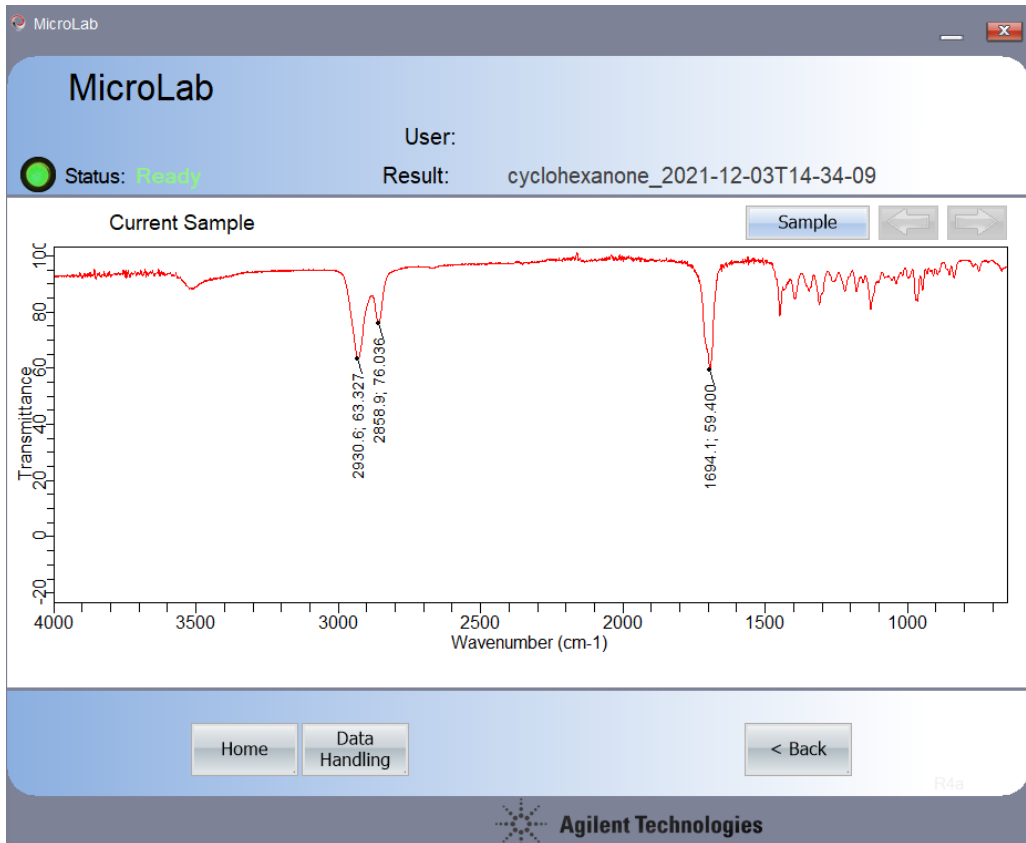

To pick peaks, right click on the spectrum and select "pick peaks".
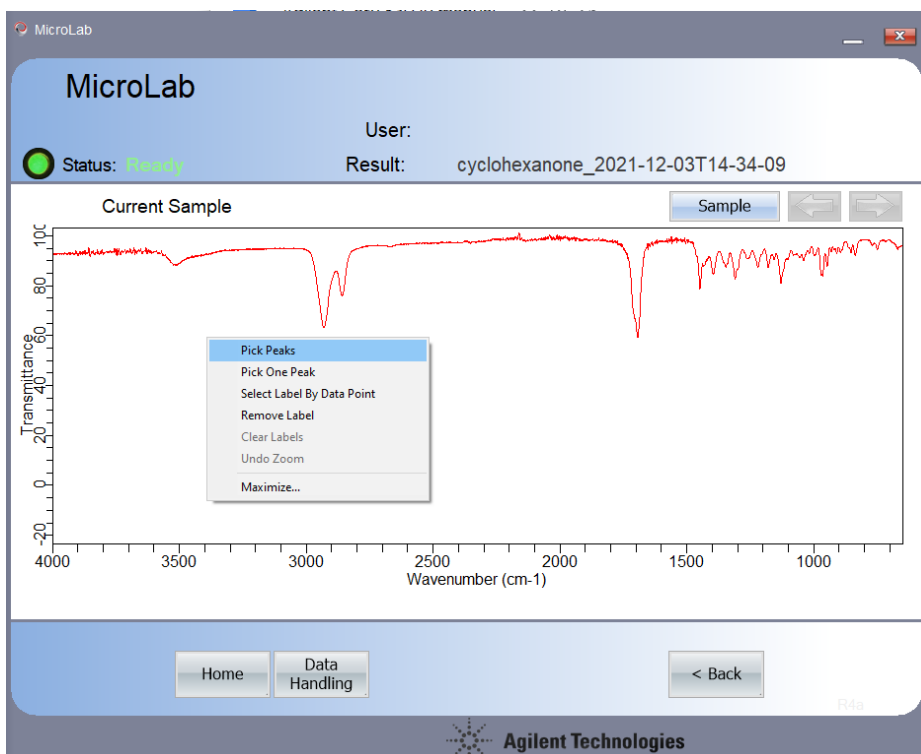
Then, draw a threshold by clicking and dragging across the spectrum

Once you let go, the peaks will be picked.

Afterwards, go to data handling

Here, you can select to generate a report - this will open a PDF document with details about the method, as well as the spectrum and a table of picked peaks.

You can also choose to export the file to a excel spread sheet or other file type.

Note, by default, McNeil ATR and McNeil Dial Path methods automatically save the results. To view previous results, click "Previous results" on the home page.

11. To finish the analysis, click done. If it asks to overwrite the data (which it always will if you pick peaks) then select "yes".

12. Clean the instrument and close the microlab software when finished. **Do not turn off the instrument** - according to agilent, keeping it **on** when not in use helps to keep the components dry and therefore maximizes the lifetime of the instrument.

Troubleshooting

Issue: the instrument says "ready" but the status light in MicroLab is yellow.
Potential Solution: the gain on the instrument is not ideal for the module. To fix this, go to Home > Advanced Features > Diagnostics > Gain Adjust. The ideal "Signal" is 26,000 - if the signal is far from this value, then set the gain higher or lower until the gain is near 26,000 then select "set Method gain" or "set instrument gain". If this does not resolve the issue, then go to the method, and manually input the appropriate gain value.

Issue: The instrument is taking a long time to take a background
Potential Solution: Check to make sure that there are no vapors (or humans) near the instrument that could be affecting the background scans - if there is too much variance between scans, the background is trashed.

If that is not an issue, then you may make a new method and, in that new method, reduce the number of background scans, reduce the resolution (by increasing the value of the resolution from its default of 2cm-1), decrease the spectral range, or change the threshold for the background (from its default of 0.002).

Issue: The instrument is taking a long time to take a sample scan
Potential Solution: this may happen if you have too much sample. Reduce the amount of sample present.

Notes:

Instrument obtained in December 2021.
Manual written by Matthew D. Hannigan, December 2021.