Introductory Training for Bruker Alpha II FTIR

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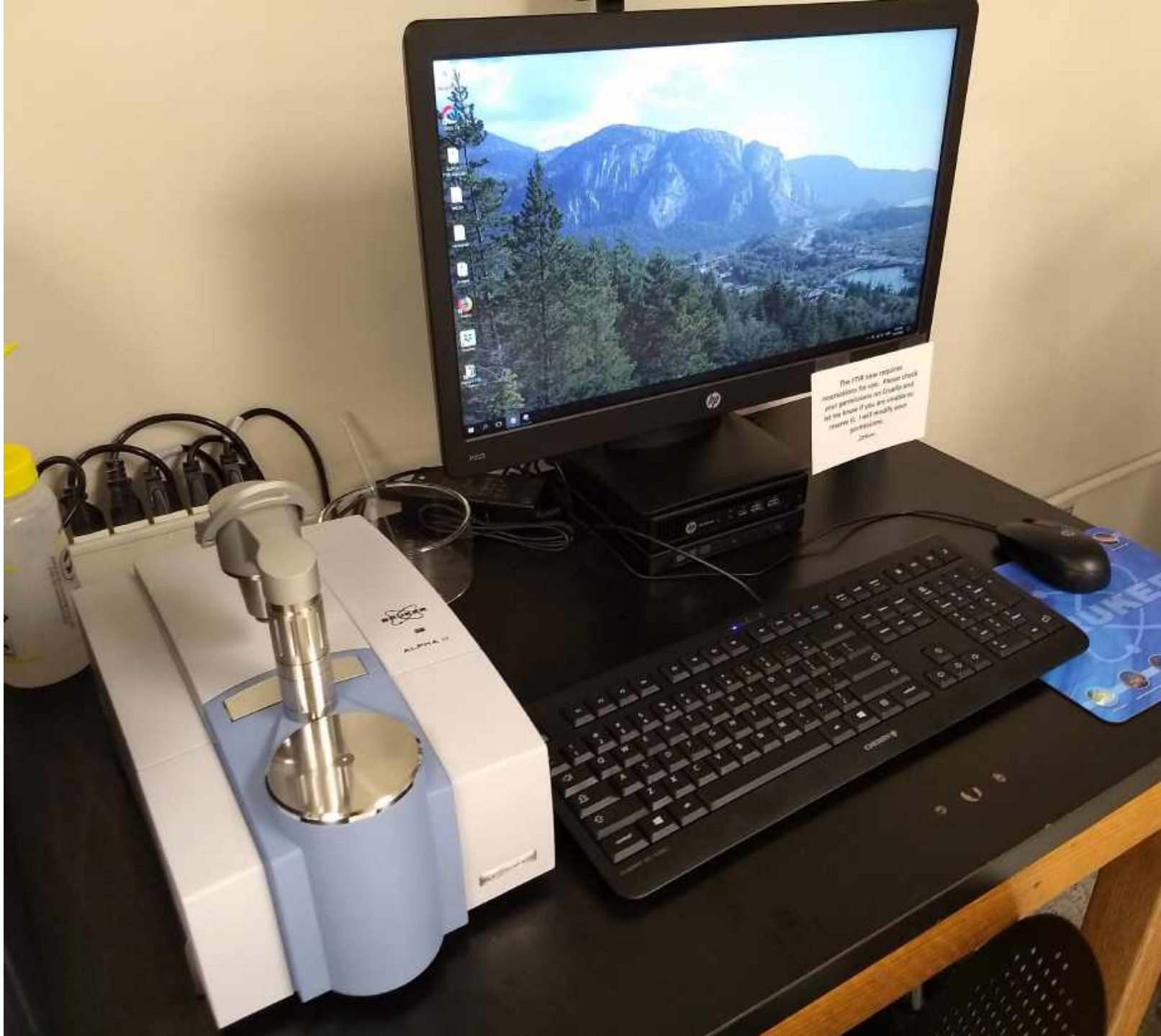
The FTIR is a Bruker Alpha II FTIR spectrometer with a Diamond Crystal ATR (**A**ttenuated **T**otal internal **R**eflectance) accessory. Measurements of liquid and solid samples can be made directly without the need for salt plate or specialized sample handling. The spectrometer runs Opus 7.8 software.



Instrument Parts

Alpha II FTIR with Platinum ATR Module

Adjustable pressure arm (swings in place over crystal)

Diamond Crystal

Anvil Tip

Samples

Sample Placment

- Samples are placed directly on the diamond crystal plate.

Crystal Preparation

- Using a Kimwipe, apply ^{i}PrOH, MeOH, or CH_2Cl_2 ***<u>to the Kimwipe</u>*** Then blot the crystal with this and wipe clean. Allow to dry before measuring your background spectrum.
- If performing a solids measurement clean the anvil tip in the same fashion

Sample Addition

- For a liquid, either use a glass pipet or a pipettor. You only need 5-10 μl of a solution that is a few mg/ml in concentration. When using a glass pipet just your fingertip to blot one drop on top of the diamond, never user the bulb. Fan the solvent to evaporate it, if it is volatile. Apply another drop if this did not cover the diamond crystal.
- For a solid apply enough powder to coat the crystal with approximately 1 mm thickness of material. Position the pressure arm over the sample and apply pressure to the sample.

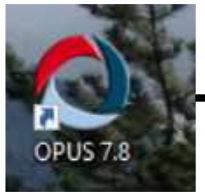
Instrument Care

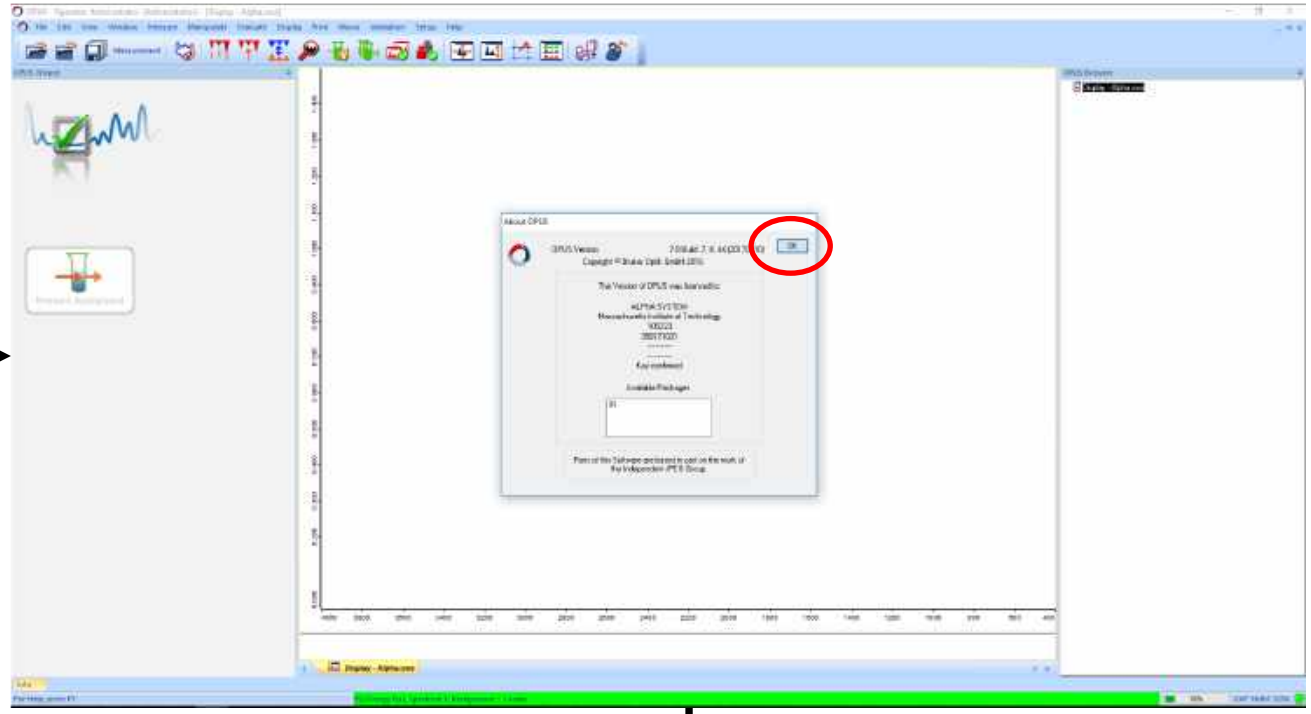
- ***Never use a squirt bottle to directly wet the crystal!!*** *Spilled solvent will damage the coating of the instrument.*
- *When using the pressure arm for solids, be absolutely certain to <u>fully release</u> the pressure before swinging the arm out of the way. While you can't hurt the diamond, you can easily gouge the stainless steel staging area by raking the unreleased pressure arm across the staging area.*

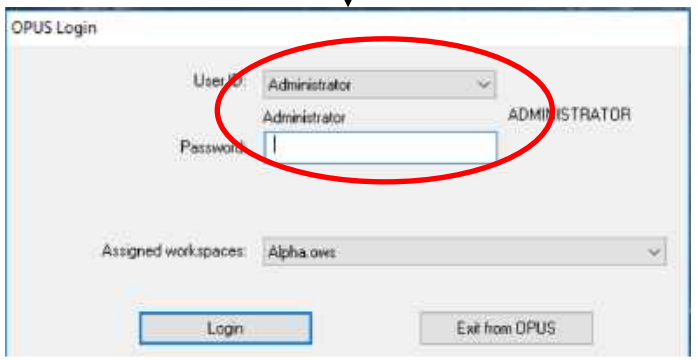
Leave no Trace!! This is a shared resource. The instrument should always be left cleaner than how you found it.

Starting FTIR Software

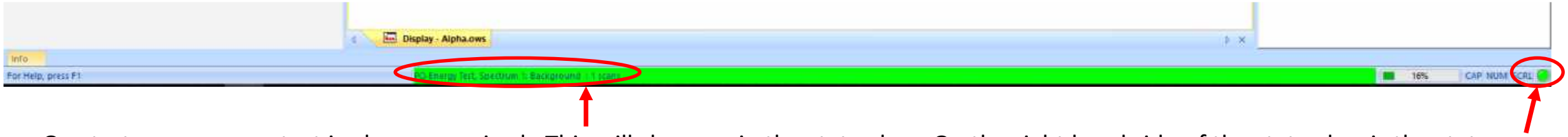
On the FTIR user switching is handled through the software, thus *you should never logout of the computer*. If someone has, find a DCIF staff member who can log back in to the operating system. The program for the FTIR is named “Opus.” Click the icon on the desktop to start Opus. This will open up the program and show the larger window on the right. When you select “ok” it will bring up the login window. Select your DCIF user ID from the dropdown and enter your password.



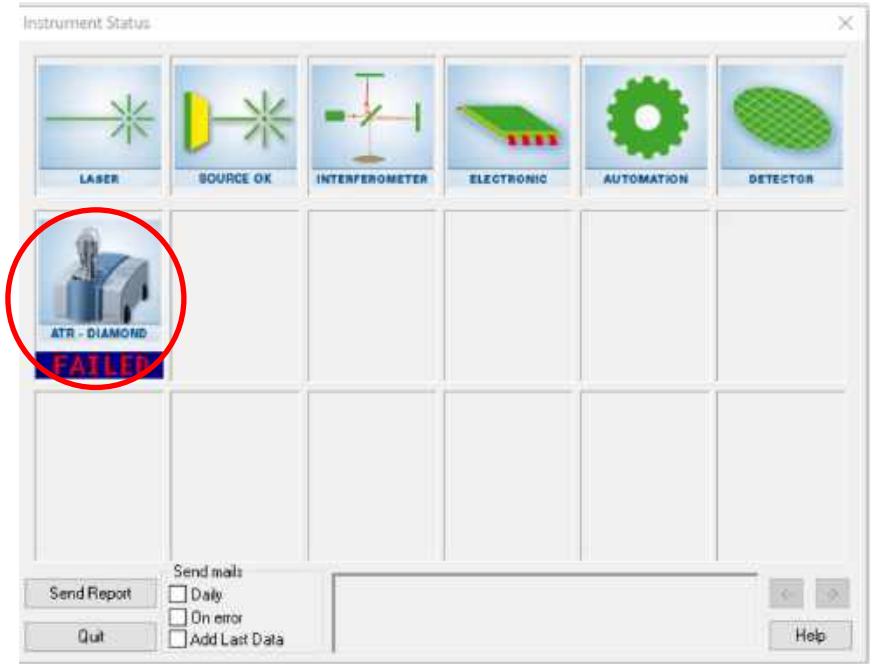


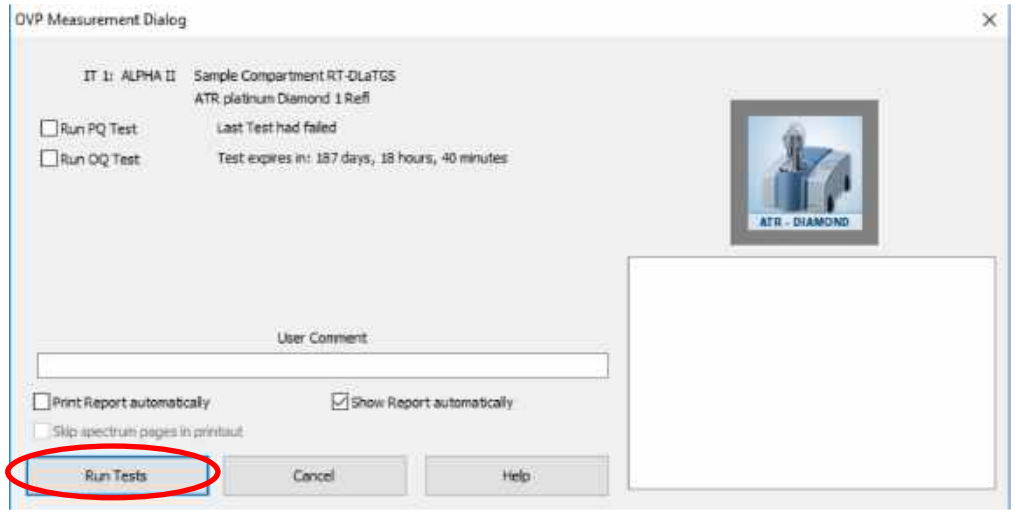


FTIR Startup



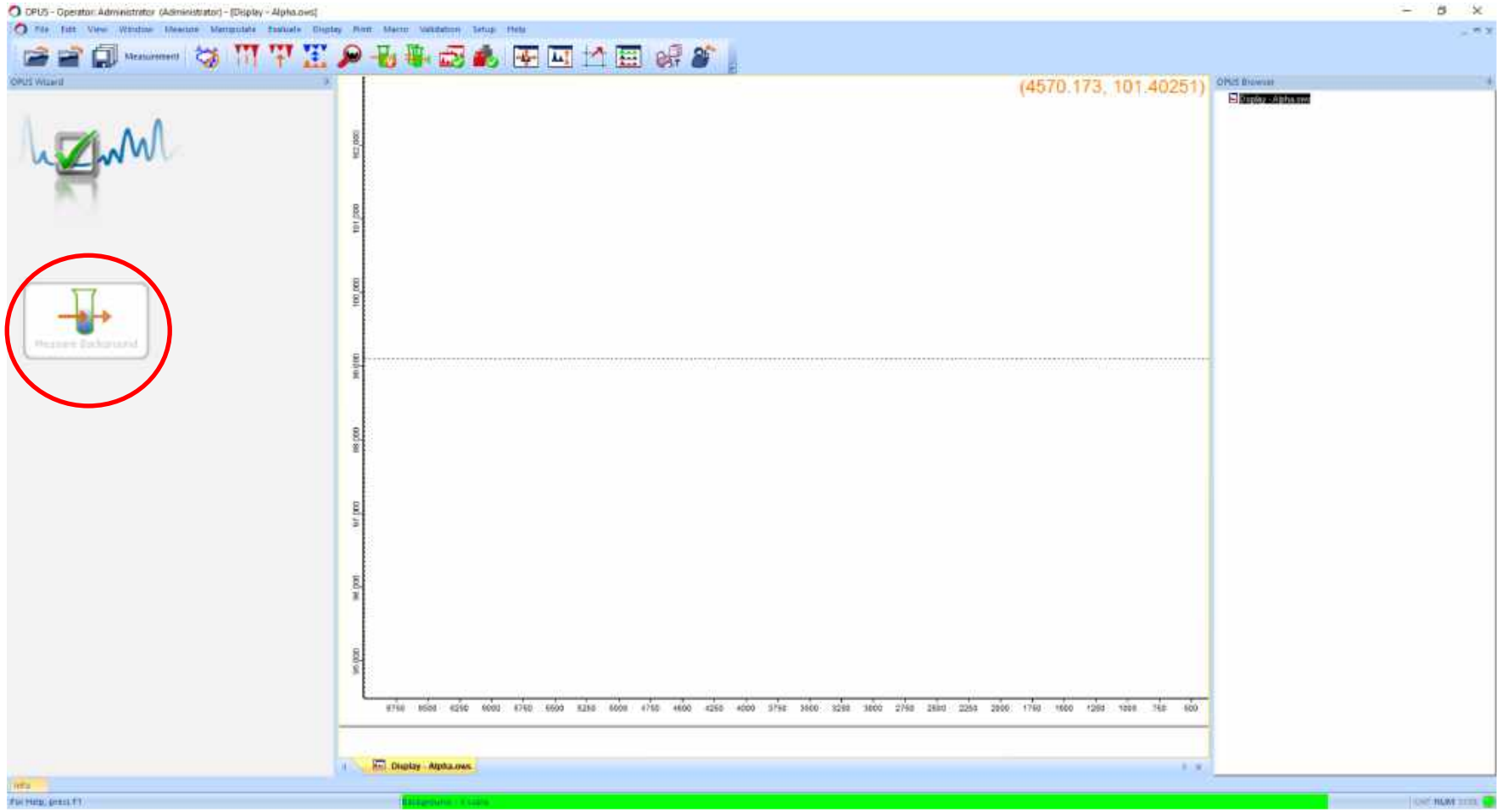
On startup an energy test is always acquired. This will show up in the status bar. On the right hand side of the status bar is the status light that is either green, yellow, or red. If it is red, then the system needs a PQ (Performance Qualification) test. This is set to be required once every 2 months. Double clicking on the status light will bring up the bottom left window. Next double click the ”FAILED” option and select “Run Tests.” The system will run a few test and then open up a pdf report of those tests. Just close the report and proceed with your measurements, at this point the status light should be green





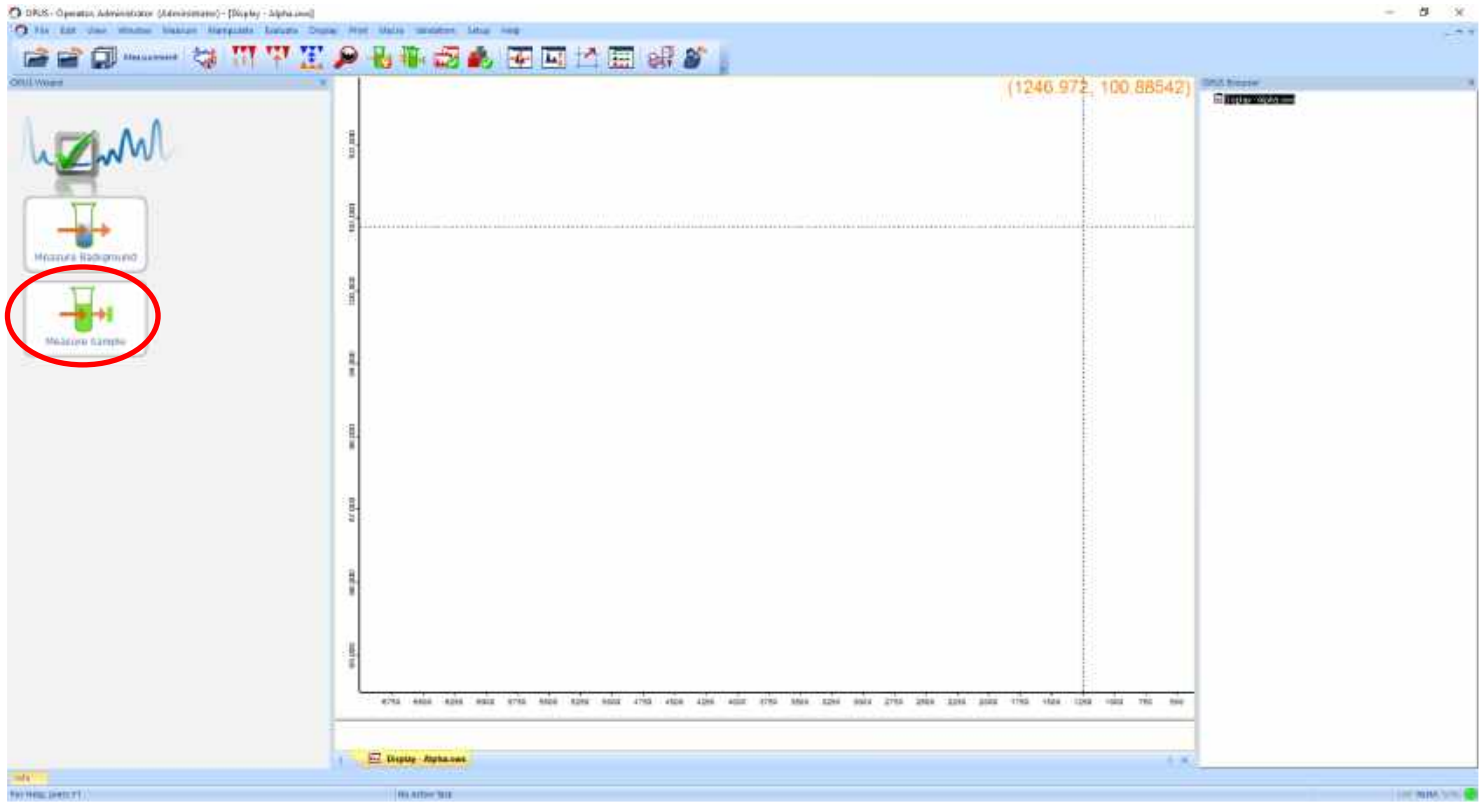
FTIR Background Measurement

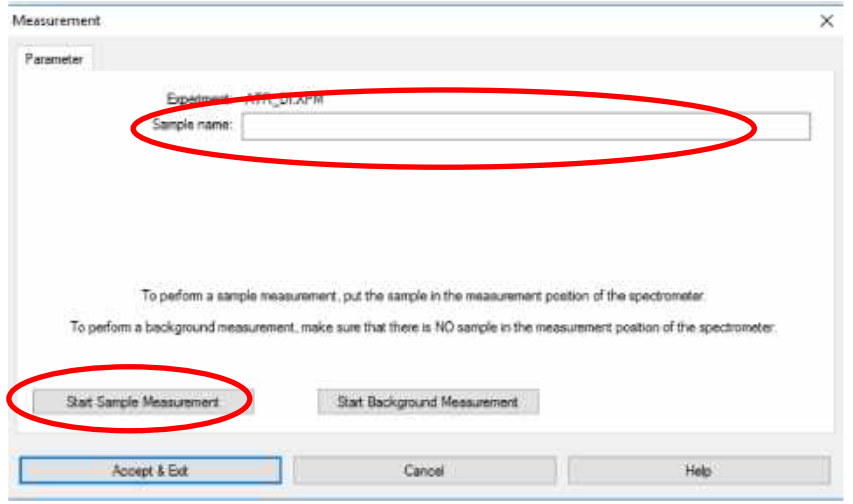
Remember to always clean the diamond crystal before use. When taking a measurement, the background is always measured first. If your sample is in a volatile solvent, the background measurement is just of the atmosphere. In the case of a non-volatile solvent, apply the solvent to the diamond crystal and then select the “Measure Background” button. The status bar will show you that the instrument is acquiring background scans, but there is no spectrum that appears at the finish. When it is done a new button will show up below the “Measure Background” button.



FTIR Sample Measurement

Click on the ”Measure Sample” button and the window to the right will appear. Enter your sample name in the “Sample name” field and then click “Start Sample Measurement” to acquire an IR spectrum. If you mistakenly click “Accept & Exit” the machine will store the name but not start a spectral acquisition.

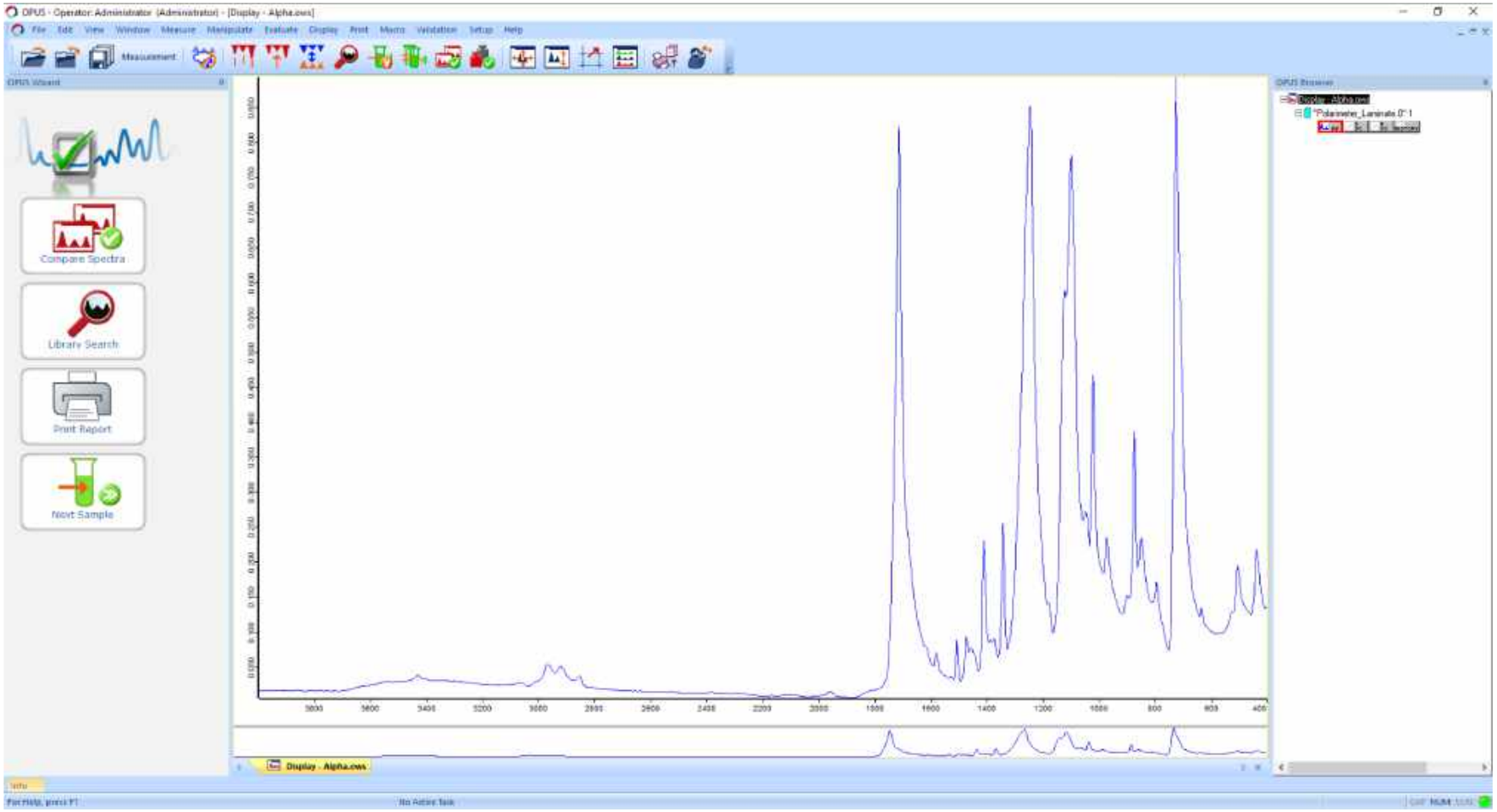




I have a spectrum!!

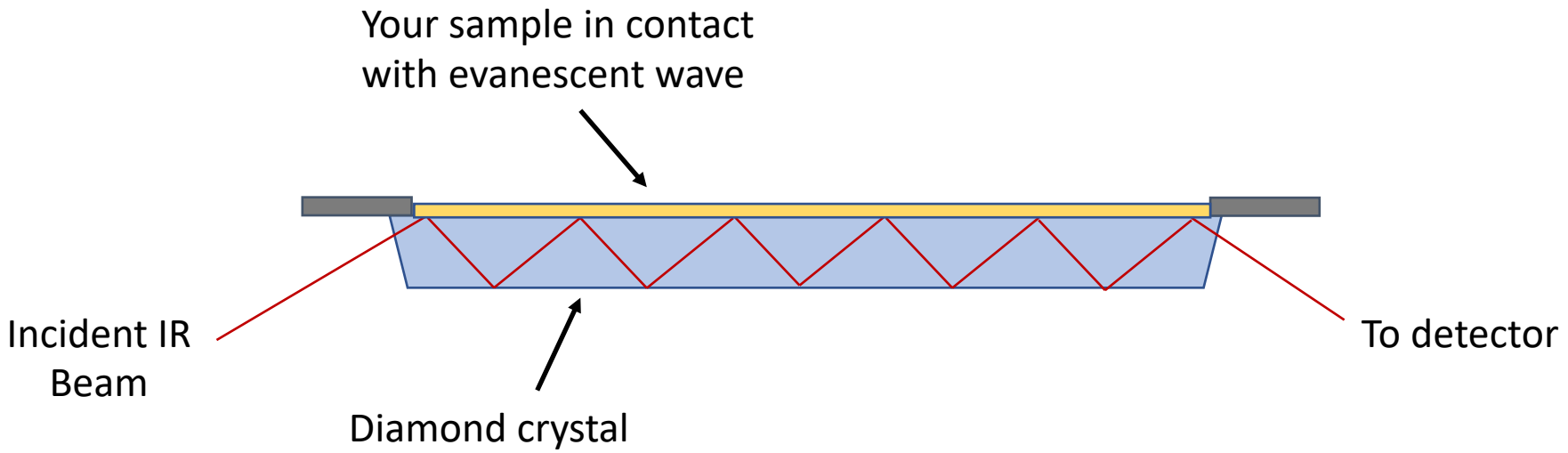
After acquisition is finished the machine will take the results of the background, your sample and combine them to generate a spectrum.

The spectrometer is actually measuring an interferogram. This along with the reference (background) interferogram are combined and Fourier transformed to generate the resulting spectrum.



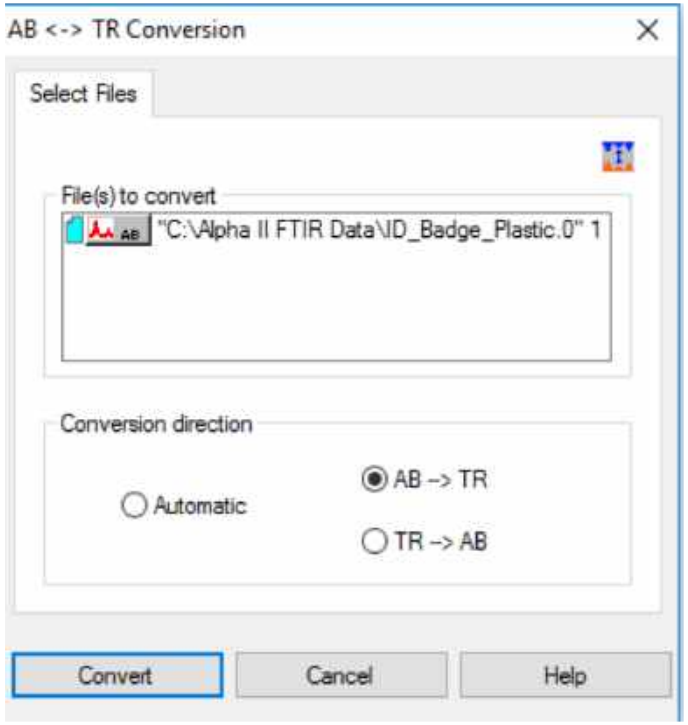
Why's my Spectrum upside down??

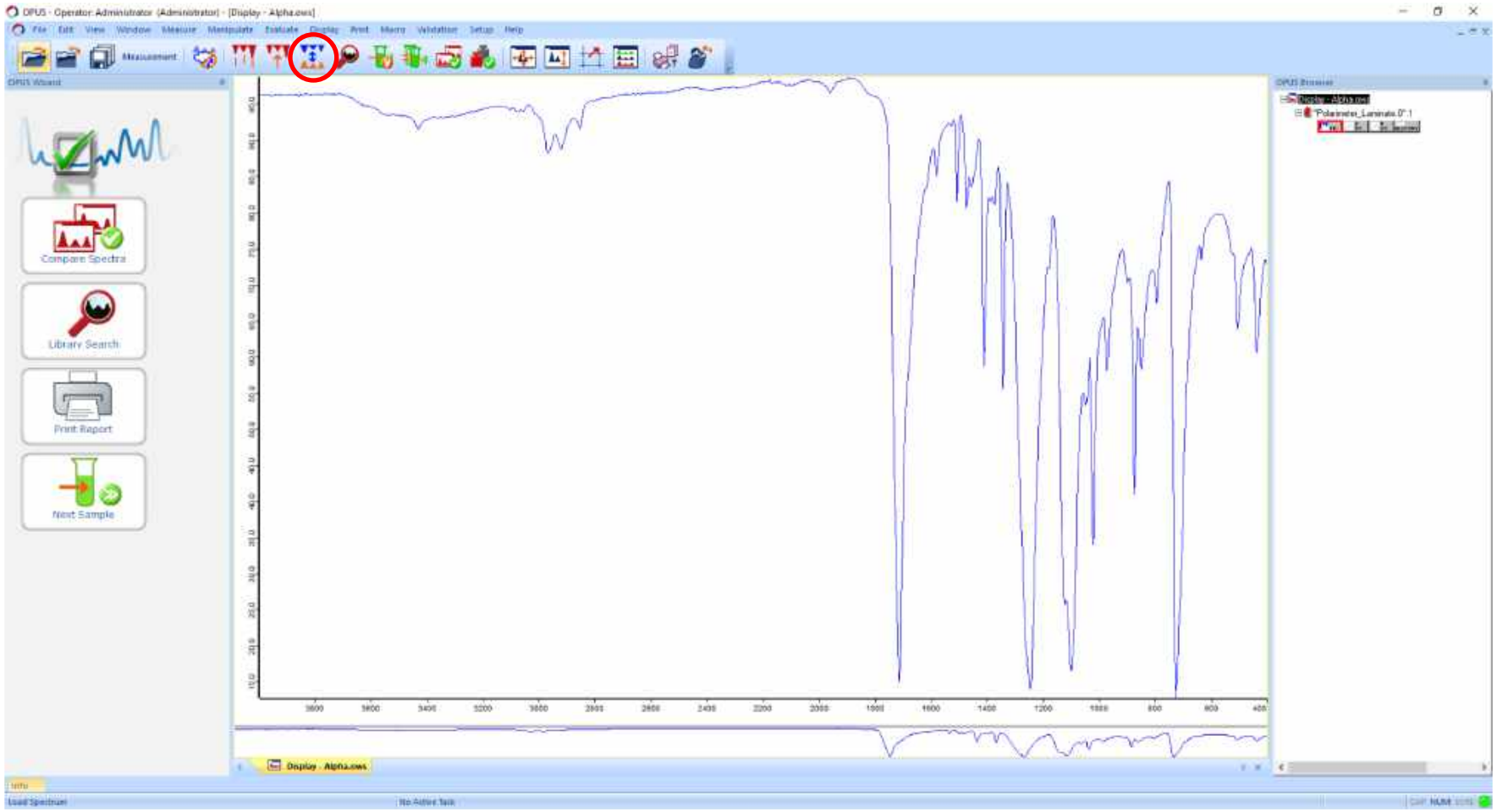
You are not measuring transmission of the IR radiation through your sample; you are measuring how much the IR radiation is attenuated by your sample. The sample is placed on a crystal with a high refractive index, in this case diamond. The intensity of the incident rays is known. When the light rays strike the interface between the diamond crystal and your sample, they undergo total internal reflectance. An evanescent wave travels beyond the surface of the crystal into your sample approximately 0.5 to 5 μm before being refracted back into the crystal. This is why your sample must be in tight contact with the crystal. the evanescent wave will be attenuated in regions where your sample absorbs IR radiation. The IR beam exits at the opposite end of the crystal and passes to the detector which measures the attenuation. Thus unlike a transmittance spectrum which is showing the % transmittance of IR light, this shows the amount of IR energy that is absorbed. But don't worry there is a button for converting between absorbance and transmittance.



Absorbance to Transmittance

Clicking on the indicated button will bring up a small box. Select the AB → TR option and click convert. This will convert the spectrum into the traditional transmission spectrum appearance.





Zooming

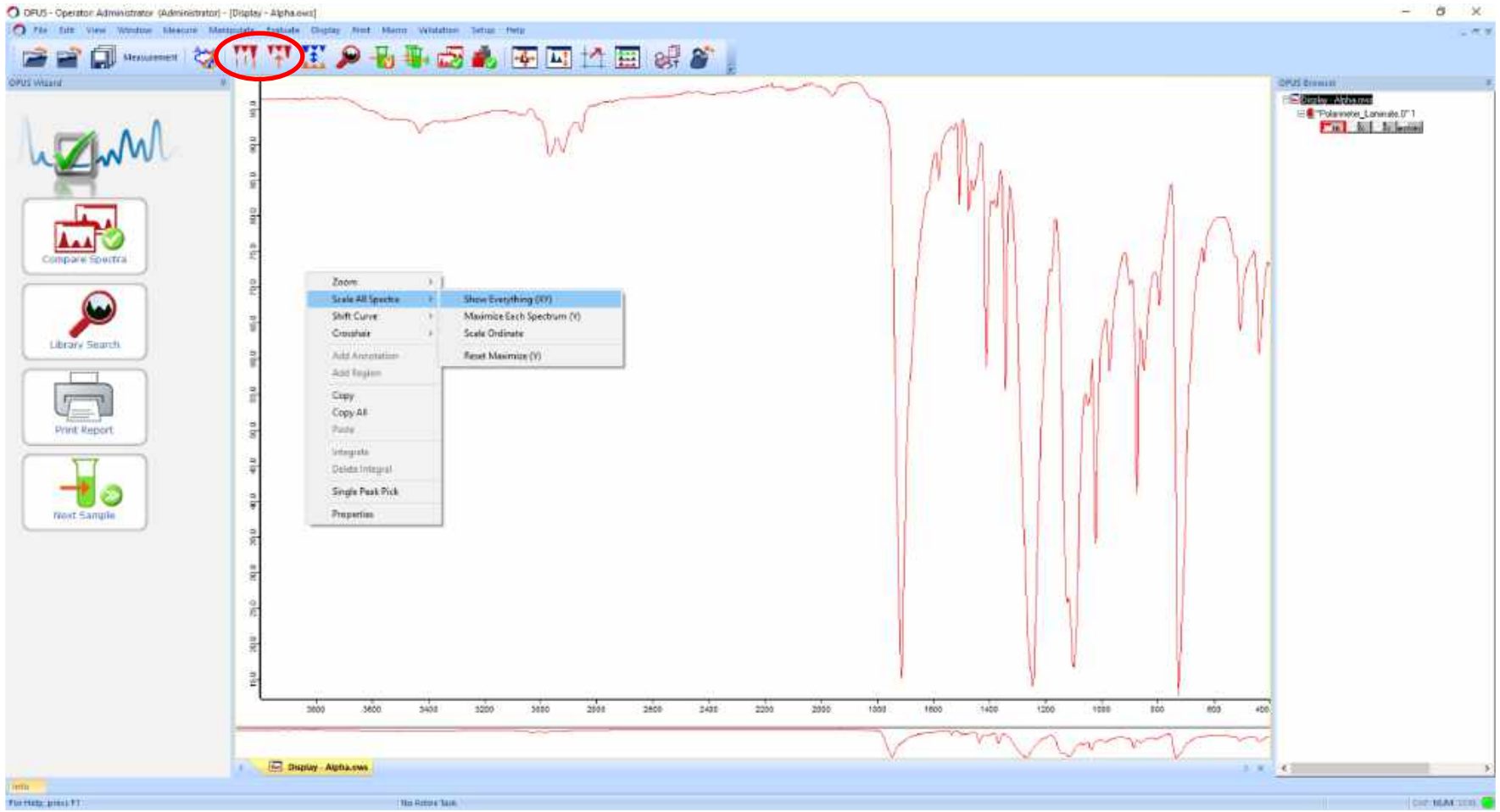
The default display mode for a spectrum comes with the zoom crosshairs showing. To zoom in, click once and then drag out a box. Click again to release the box and then set the box right over the area you wish to expand. Click once more and it will show the expansion. The three circled buttons are the “scale,” “scale ordinate,” and ”zoom.” These reset all dimensions, reset just the vertical scale dimension, or enter the zoom mode.

Zooming

You may also adjust the region shown in the spectrum overview at the bottom. Just grab the corner of the box and size it according to what part of the spectrum you want to show.

Peak Picking

More display options can be found by right clicking once to get out of the zoom mode, then right clicking again to bring up a menu. Peak picking can be selected in several ways, but it does not work when you have expanded a region. Under the dropdown menu shown on the left is the ”Single Peak Pick” option. There is also a button for single and multiple peak picking. Multi peak picking brings up the dialog box below where the cut off level can be set. After this click “peak picking” and it is done. If you use the interactive mode, you may graphically set a threshold line below which peaks are labeled

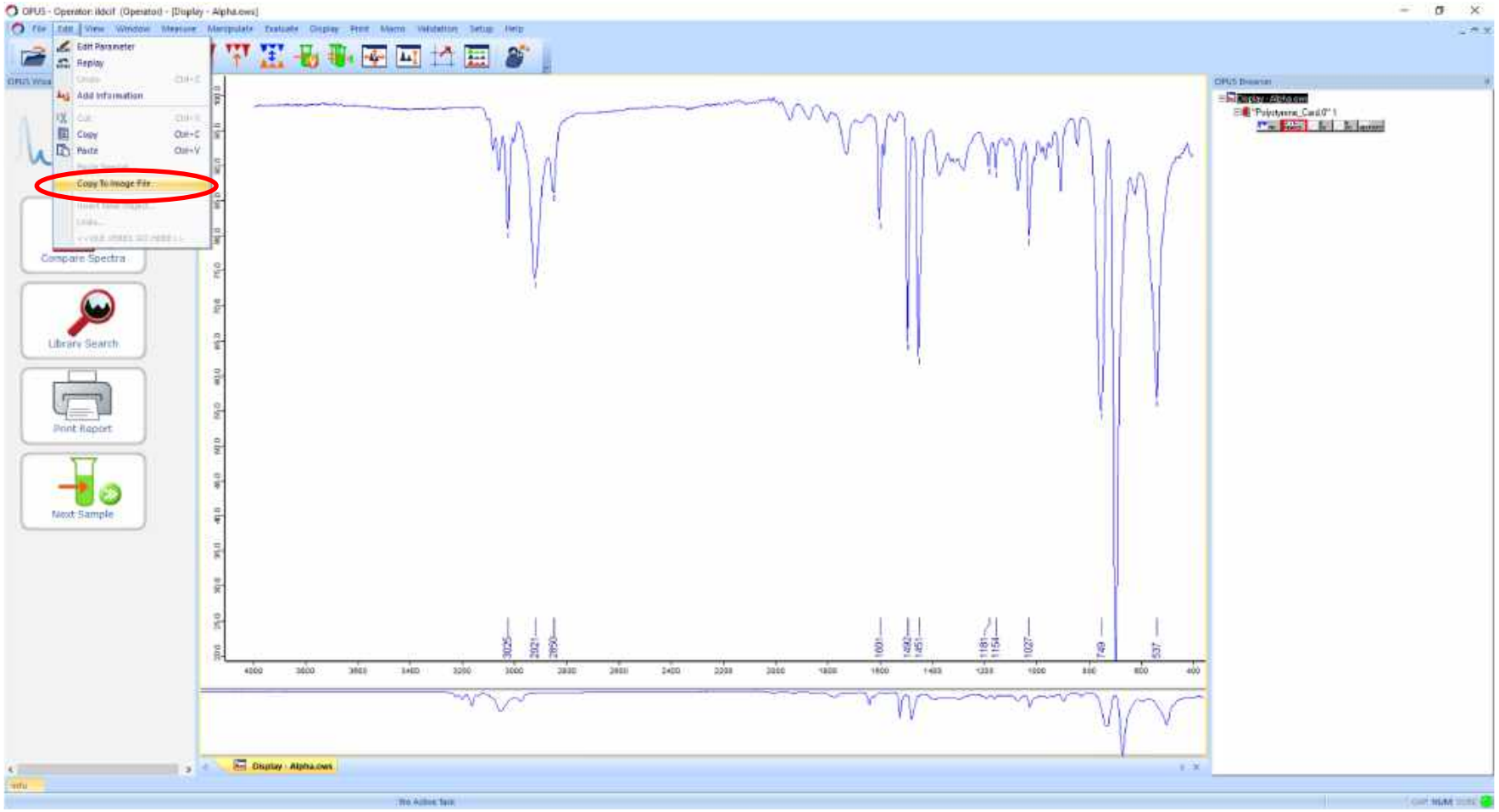


Opus Browser

The browser allows you to select different portions of the data. The boxes from left to right are TR (Fourier transform), peaks, ssc (sample interferogram), rsc (background interferogram), and history. You may select them by clicking on them and the right click to show options. When you are acquiring data from more than one sample, the system leaves previous spectra in the browser and display. The “remove from display” option can be useful as well as “Change Color.”

Viewing data away from the instrument

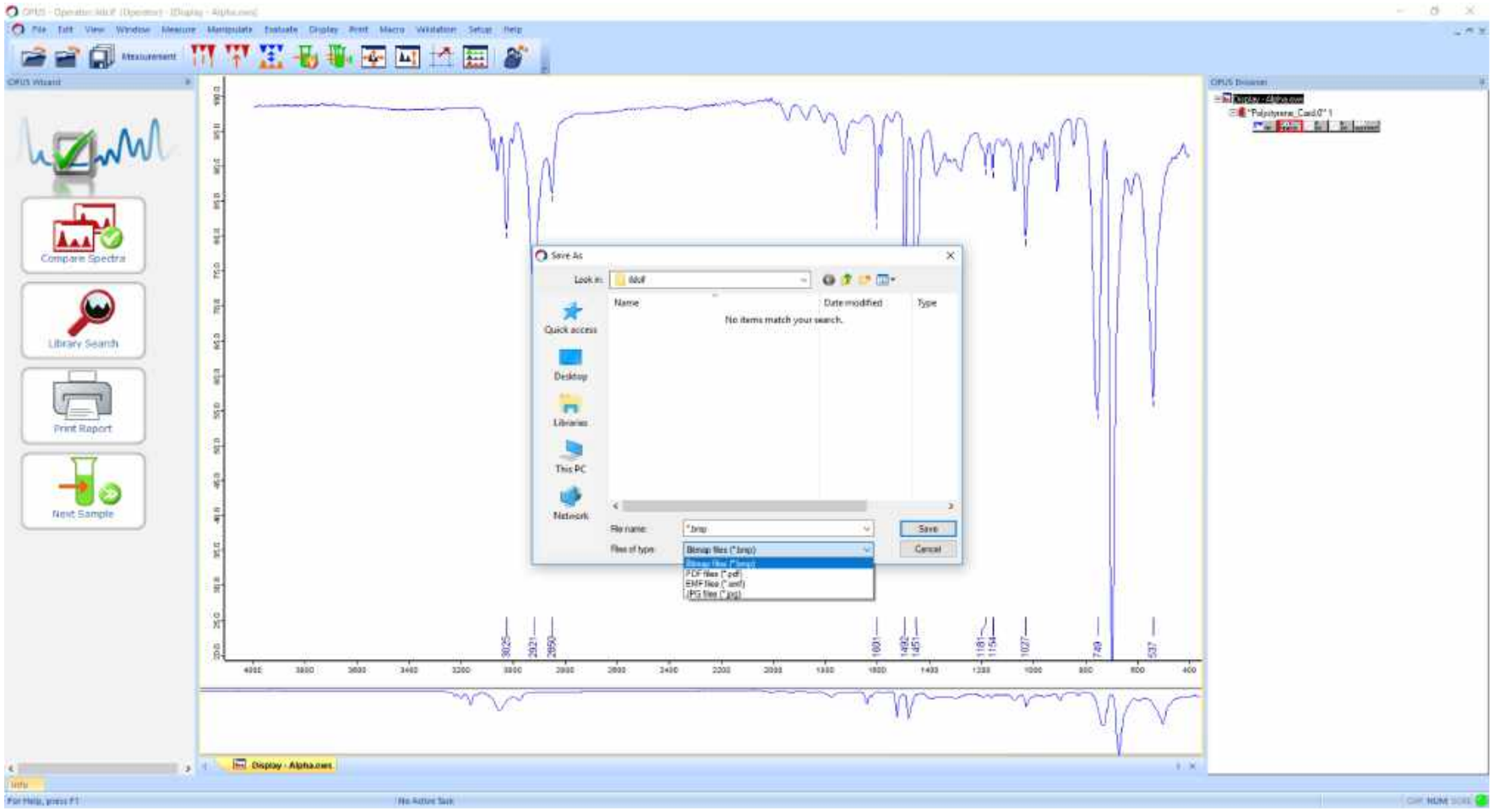
While the data is automatically saved in Bruker format, it is not readable using any other program. There are two options for saving you data to a different format so that you may view it on other computer systems. Under the file menu, and save as option, there is a tab for mode where you may select “data point table.” This is just x,y data which can be graphed in any number of programs. There is also the option to generate a pdf, jpg, png, or emf format file. Select edit, then “copy to image file.”

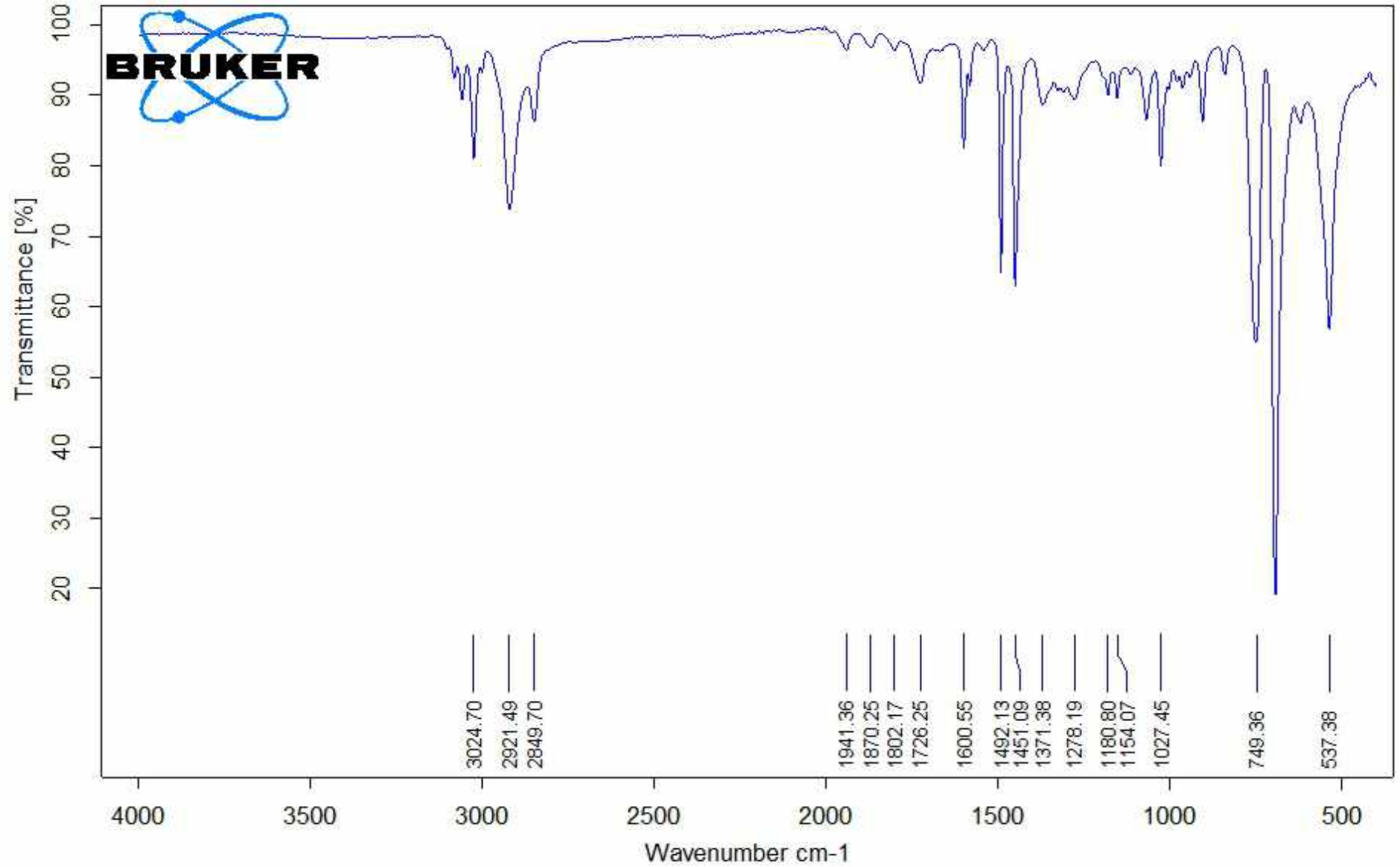


Data is saved in c:\Alpha II FTIR Data\<DCIF-user-ID> but currently is not backed up to the DCIF Server.

Copy to Image File

Here you can select the file type and browse to the location you want to save your file. An example is shown on the following slide.





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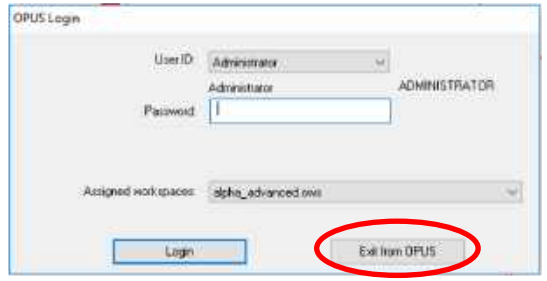
Logging out and shutting down

Select “setup” then “logoout” and then finally “exit from opus.”



Remember to never log out of windows!!