Optical Lab Room 3217 Nexus 670 Instructions

BEFORE YOU START

Please write your name, PI, and time on the instrument in the Logbook.

The Nexus 670 should always be on. Check LEDS on the top left of the instrument, all should be on, with scan light blinking.

<u>START</u>

Click on the icon for "Omnic ESP". At this point you can also use the Help Menu for further information in addition to these instructions. See "Getting Started" in the help menu.

After Omnic opens, look for a green check mark and the words Bench Status in the top right corner. Sometimes there is a few minutes delay while the program is initializing. Note that you still have to check the interferogram as follows.

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Go to **Collec**t, **Experiment Setup**, **Bench**, to observe the interferogram. In the middle of the display is a *characteristic signal maximum*. Check to see if the interferogram signal is within limits, (min 2, max 9.8)

If it is a flat line go to Collect, Diagnostics and click on Reset Bench.

With a gain of 1 (set by going to the **Bench Tab** in Experiment Setup) check the p-p voltage-it should be greater than 8 volts. This is with no accessories and a clear path in the sample compartment. Also be sure the aperture is set to 100, normal for resolution 4. A smaller resolution will automatically set the aperture smaller. This means the interferogram peak-to-peak voltage will be smaller.

It is very important to check the experiment setup bench and collect tabs to see if the peak to peak level and parameters are correct. Check the aperture to see if it is 100%, unless you are using a resolution smaller than 4. Also, it is important not to overload the detector with too much signal, which will happen if using the MCT detector option. There is a drop-down window where another detector can be selected. The room temperature detector is DTGS KBr. This is the default detector and normally used.

Go to **Collect**, **Experimental setup** to select: Number of]Scans, (default 32) Resolution Default 4, Absorbance or Transmission, Correction for CO2 And other options. It is usually set up for the transmission experiment.

BACKGROUND

Collect background first with no sample. Collect Menu, or click on the Collect Background button on the toolbar.

When the collection is finished, you can save the background to a window if you want it available later for a background handling option, or simply close the Collect Background window. The spectrum is not saved then, but remains the current background. This step eliminates the background characteristics of the spectrometer so the peaks in the final spectrum are due solely to the sample.

COLLECTING SAMPLE

Use Collect Sample in the Collect menu to collect the spectrum of a sample. There is a progress indicator bar on the lower left hand side of the screen. The spectrum of the sample will be ratioed against the single-beam spectrum of the background.



EDIT MENU

This allows you to change the size of the annotation numbers when printing,

COLLECT MENU

This can bring up "experiment setup", with ability to change number of scans and resolution. Final format can be absorbance or transmission. There is a box to check "save interferogram" to have the ability to process the spectrum later. Corrections that can be made later include one for ATR wavelength dependence of attenuated total reflection. Background handling determines when and how often background is taken. Also you can use a saved background file.

ANNOTATING THE PEAKS

Analyze Menu: Click on the top toolbar, then **Find Peaks**. Click on the line where you want peaks to be annotated. On the left side there is a slider marked "Threshold" to select minimum threshold for peak selection. Click on **Replace** button on top right before printing.

The palette tool labeled T will select individual peaks.

Palette Tools



Selection Tool: Display specific regions, zoom in and out, grab and move spectra, select single or multiple spectra, define (x,y) coordinates.

Region Tool: Selects X-axis start/stop frequency, use for manipulations such as library searches, blank/straight lines, noise calculations, uncorrected area calculations.

Spectral Cursor Tool: Reports value of data points (x.y); used for spectral comparisons; if you have a log file running, the data points will be recorded in the log.

Peak Height Tool: Calculates peak intensities, allows user-defined baseline, calculates height-to-zero as well as relative measurements, stores measurements in log file.

Peak Area Tool: Integrates the area under a peak, allows user-defined baselines, calculates area-to-zero as well as relative measurements, stores measurements in log file.

Annotation Tool: Manual labeling of spectrum, labels are stored with the spectrum

(highlight to delete), style and font is adjustable (Display setup and Options, see below). SHIFT KEY + Click of mouse will pinpoint the maximum for Spectral Cursor, Peak Height, and Annotation tools. Automatically finds the point of greatest intensity near the cursor.

RIGHT Mouse Click will bring up an information box for whatever the cursor is resting on. The box will always have a "Discussion" button and sometimes a "How To" button to help you with the features of the object.

FILE MENU

This allows you to save in different data formats. Accepted Formats Extensions OMNIC Spectra *.SPA OMNIC Group *.SPG JCAMP-DX *.JDX PCIR *.IRD Nicolet SX/DX *NIC or *.SPC Grams 386 *.GLD Peak Solve *.0?? Mattson *.IGM, *.ABS, *.DRT, *.SBM, *.RAS Windows Metafile *.WMF TIF *TIF Comma Separated Value *.CSV Also, if you go to file, print, cute pdf will convert to pd "Save Group" allows you to save several spectra within the same file.

THE ATR MODULE

The ATR , or Attenuated Total Reflectance module has a very small window which the sample can contact. Infrared light is reflected by a mirror to a conical zinc selenide lens with a diamond coating on it. Use great caution when touching this surface, <u>DO NOT HIT IT WITH THE STEEL ANVIL</u>.

Turn the knob counterclockwise to retract the anvil before you move it into place. Don't touch the surface directly with the steel anvil. The steel anvil is only intended to press solid or powder samples against the window.

Take background measurements with the cell window clean. Don't lower the anvil down because air is the reference we use. Also, there is a risk of damaging the window surface. Use auto gain for the gain setting.

For a liquid sample, only a small amount of liquid is required to cover the cell. Don't allow liquid to run down the plate.

When taking powder samples, use a small amount to cover the cell.

Turn the knob counterclockwise to retract the anvil. Pull back on the smaller knob to release the arm and lock it down. Then turn the knob clockwise until the anvil presses down on the sample material. The knob will click when there is enough torque.

After you collect the sample. Carefully wipe powder up into the kimwipe with ethyl alcohol, do not drop powder over the edge of the plate or anywhere else. The ATR method saves time, but the hardware is very expensive and requires neatness and care.

TR Module with plastic sample:



Anvil with plastic film sample pressed onto sample cell window.