Digilab Excalibur Series IR Spectrometer Operating Instructions

- Depress toggle switch on the back panel of the instrument
- Turn on computer and monitor
- Open Digilab Resolutions program on the desktop
- Align the spectrometer by clicking on the interferogram icon directly below the “Scan” menu
  - click the Align button near the bottom right of the pop-up screen
  - Alignment may take up to two minutes
  - Verify that the magnitude of the peak is less than 10 volts (If not, consult Merlin Manual)
  **Note that the spectrometer should be re-aligned daily before running samples
- Calibrate the spectrometer by selecting Calibrate under the Setup page
  - A “Calibration Complete” message will appear when the operation is complete
  **Note that the spectrometer should be re-calibrated once per day or after changing resolutions
- Press the ok button to save the alignment and calibration

Collecting Data
*There are two methods of data collection:
  - Scanning- used for relatively stable sample
  - Multi-scanning- used for samples that change over time

1. Scanning Method
- Select the “Scan” menu and then “Scan” again
  - click through the different options under Sample, Background, Options, Accessory, and Methods tabs to change the scanning method to the desired settings
  **Note that the Resolution for the sample and background must be the same
- Click on the Background icon (below the “Help” menu) to collect the background
  - If asked, save the background spectrum in an easy-to-remember place
  - It may be useful to name the background with some form of the sample for which it is the background
- In the chart at the bottom, name the background spectrum
- Insert sample into cell compartment inside the instrument
  - To scan the sample, click the icon to the right of the Background icon
    - Again, save and name the spectrum

2. Multi-Scanning Method
- Select the “Scan” menu and then “Multi-Scan”
  - Clicking through the six tabs of parameters, change the settings to collect data for the changing sample
  - Trial and error may need to be used in order to adjust the settings properly for the sample. The parameters depend on the sample itself, how fast it is changing, etc.
    “Scans Per Spectrum”- how many scans should be co-added to create a spectrum
    “Number of Spectra”- how many spectra are to be collected
    “Initial Delay”- wait this time after the Scan button is pressed before data collection is started
    “Delay Between Spectra”- after one spectrum has been collected, how long to wait until beginning data collection of the next spectrum

Performing a Subtraction
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-From the left file holdings, click and drag the files of interest into the spectrum chart

-The spectrum which is inactive (not highlighted) will be the substrahend

-If the wrong spectrum is selected, click the Swap button on the control panel sidebar or highlight the other spectrum

-Click the subtraction icon (up/down arrow with a minus sign) in the Interactive Operations bar

-The sample spectrum and substrahend are shown in the top panel

-The subtraction result is shown in the bottom panel

-The default subtraction factor is 1.00. A better factor can be obtained by clicking the Autofactor button on the right of the two windows

-This is a calculated minimum of the sum of the mean-square differences between the sample and substrahend

-To replace the old sample spectrum with the new subtracttioesult spectrum, select Replace

-To add the subtraction result as a separate file, select Add

Peak Picking

-With the spectrum of interest highlighted, select Low sensitivity from the Peak Picking Menu right below the background and sample scanning icons

-Click the Peak Picking icon to the right of the Peak Picking Menu

-Peaks are automatically selected and a Peak Table is displayed in the Results Window

-If some peak labels overlap, you may click and drag the labels to more convenient places

Changing the Size of the Spectrum

-Place the mouse arrow on the grey portion of the X- or Y-axis scale and hold it there until a thin, double-ended arrow appears

-Left click and hold while moving the mouse up or down

-Adjusting where you click and hold on the axis allows you to change the size of the spectrum in different ways (Expanding/Contracting)

-To Auto Scale both the x- and y-axes, click the icon with a quadrupole-ended arrow

Adding a Vertical Line

-Sometimes it is useful to add a vertical line in order to easily observe peak shifts

-Place cursor at desired x-value location (in the grey scaled region)

-Right click, select New, then Vertical Line

-To find the exact position of a drawn line, left click on the line in the grey region, then right click and select Properties

-Lines may be dragged to different positions by left clicking and holding on lines until positioned as desired

-To delete a line, place cursor on the line and right click, then select Delete

Labeling and Annotating Peaks

-Right click on the peak to be labeled, select New, then Position Label

-The point of attachment may be moved by placing the cursor on the point, left-clicking and holding which re-positioning the point

-The label may been moved to a convenient place by clicking, holding, and dragging

-To annotate a peak, right-click above the peak of interest, select New, then Annotation

-Enter description as desired

-Move the annotation box to desired location on the spectrum

-Font size and style can be changed in the box
- The size of the box can also be adjusted by clicking and dragging the black squares around the box.
- Boxes can be deleted by right clicking, and selecting **Delete**

**Printing Spectra**
- If multiple spectra are to be printed, be certain that all files are open (the opened spectra are all listed in the chart in the lower half of the screen).
- Click on the Setup Report icon (shows a printer with red, yellow, and blue blocks).
- In the files box on the left, click on the “+” of the Print Composition1 file.
  - The spectra which you had open for printing should be shown here.
  - Left click on the first spectrum to be printed and drag the spectrum into the composition area.
  - The spectrum should then appear on the template as it was last left (with peak labels, etc.).
  - You may then add other labels, such as the sample ID, time the spectrum was taken, etc.
  - Left click on the desired add-ons under the History tab in the file box on the left.
  - Drag the add-on into the template.
- Adjust the size of the spectrum and the add-ons (time, ID, etc.).
- There is a dotted blue box to show where one page ends.
- When the composition is as desired, under “File”, select “Print”

* Export Files as **.csv** (comma-delimited) in order to open and plot in Excel.
  Be certain to reverse the values on the X-axis (**Click on X-axis → Scale → check “Values in reverse order” box**).