Operating Procedures for Bruker Vertex 70 with ATR and DRIFTS

Updated on January 6, 2012

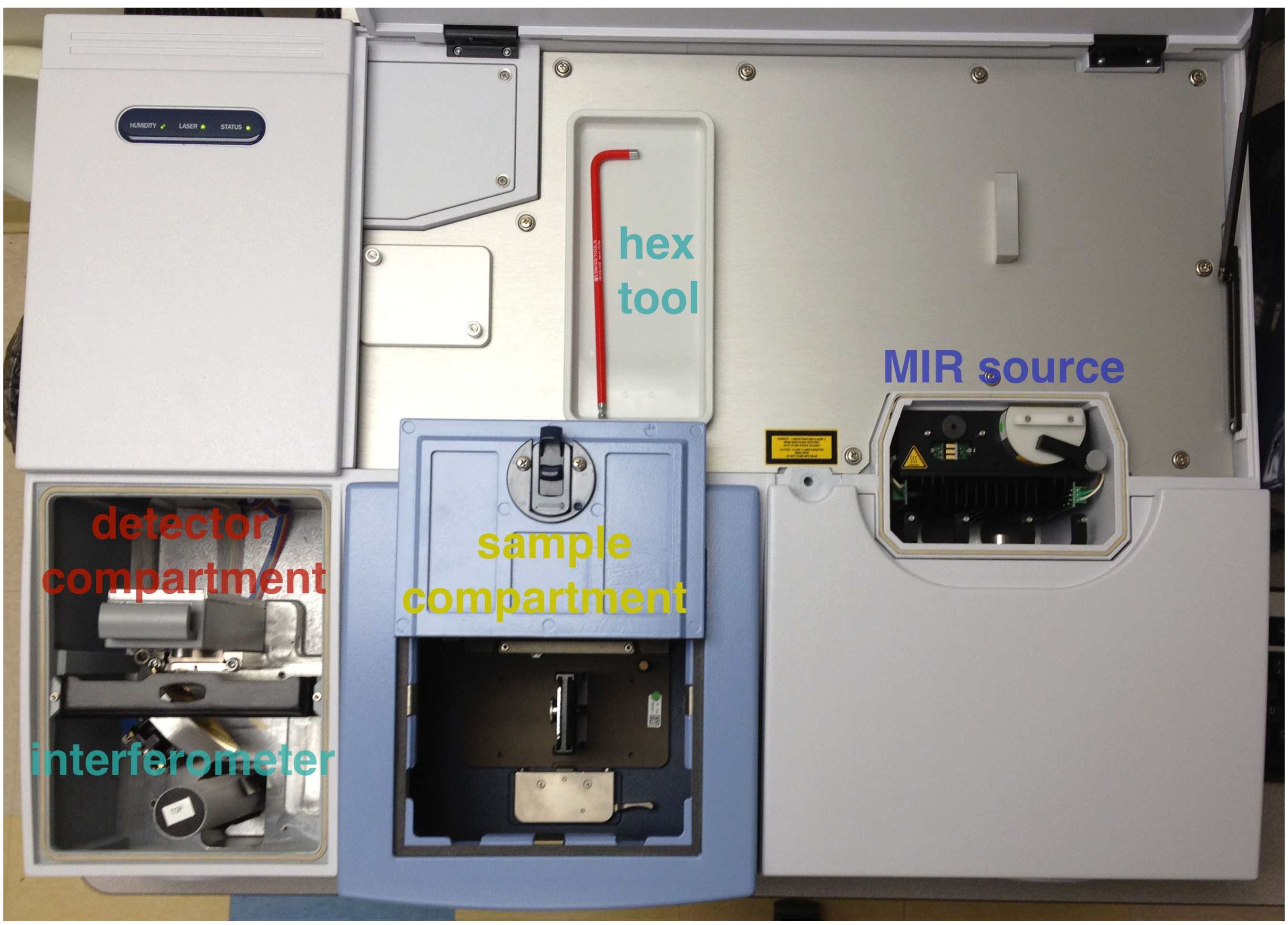
Safety Rules and General Considerations

1. All users are required to complete FTIR training given by the FTIR czar (Joe Elias).

2. The spectrometer has a HeNe laser; absolutely under no circumstances should the user have his or her face in the beampath. This is especially true for ATR, as the beampath is directed upward from the top-most slit of the VeeMAX accessory.

3. Great care must be taken when changing the detector and sampling accessory **not to touch any part of the mirrors and windows with anything**. These components are very fragile and sensitive to oil from people's fingers.

4. For time-resolved experiment and use of *in situ* cells, refer to the specific manuals.

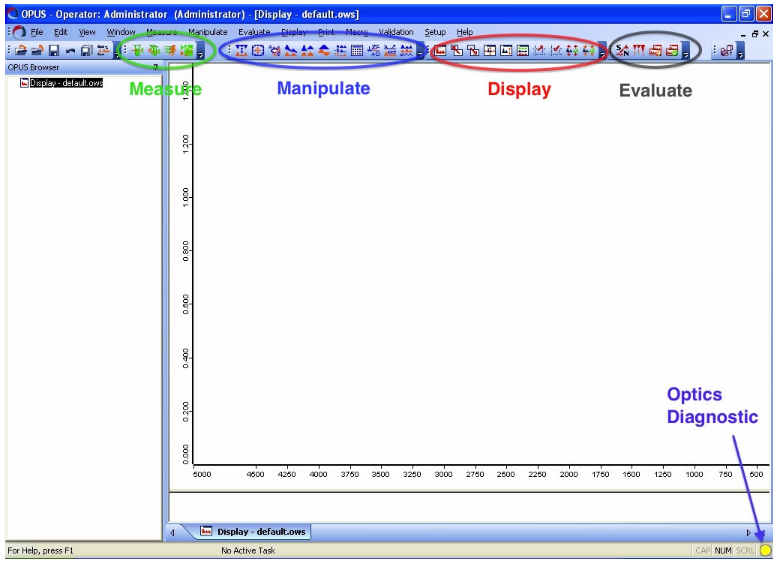


Starting Up and the Graphical User Interface

1. First, ensure that the dry air-compressor is on (green button on left of compressor unit).

2. Open *OPUS 7.0*. Log in as 'Operator,' leave the password field blank and use the default workspace.

3. After clicking 'OK,' your workspace will load:



4. At the top, there are four tool-bars of note that correspond to selections from the 'Measure,' 'Manipulate,' 'Display,' and 'Evaluate' dropdowns customized for your convenience. **NOTE:** There are *many* more options in the dropdown menus.

Measure: Loads the experimental setup editor, including the four most popular experiments:

 **Advanced data collection** Single, static experiments

 **Repeated data collection** A series of experiments using the same setup

 **Rapid Scan TRS** For rapid scan experiments (112 spectra/s at 16 cm^{-1} spectral resolution). Requires MCT detector, see manual for more details.

 **Step Scan TRS** For step scan experiments (up to 12.5 μs spectral resolution). Requires MCT detector, see manual and the *Introduction to Step-Scan* book.

Manipulate Spectral manipulation options are found here, including baseline correction, post zero-filling and spectral conversions

Display Here you can find options for displaying your data, including scaling axes and stacking spectra

Evaluate Data evaluation options are found here, including peak picking and a signal to noise calculator

5. The **OPUS Browser** on the left side of the workspace includes *all* specta you load and collect. Below the filename are displayed blocks that represent your raw and manipulated spectra as well as data logs. Data from these blocks can be manipulated and displayed by right-clicking.

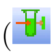
6. Before starting data collection, click the **Optics Diagnostic** button on the bottom right. This will bring up an instrument status window. **If you are the first user of the day, it is most likely that you will have to perform a PQ test, and an icon on the left will displayed a light-blue 'Expired' message.** To perform a PQ test (5 minutes), simply double click the PQ icon and click the 'Run Tests' button.

Quickstart: Transmittance

The spectrophotometer should always be equipped with the transmittance sample holder by default.

1. Ensure the air compressor is on (green button on the left)

2. Start up OPUS and perform any PQ tests as needed before loading sample (**Optics Diagnostic** button).

3. Click the 'Advanced data collection' button () on the tool-bar at the top. This will bring up a window with 10 tabs: **Basic**, **Advanced**, **Optic**, **Acquisition**, **FT**, **Display**, **Background**, **Check Signal**, **Beam Path**, and **Spectral Range Selection**. If at any time the experimental parameters you enter here are incompatible, a red 'Caution' icon will be displayed and the problematic field will be highlighted in red.

4. **Basic:** Load the MIR_TR.XPM experiment and name your sample. This tab will also be used, once your experiment is set up, to actually run your experiment.

5. **Advanced:** Specify the name and path of the saved file, select your spectral resolution, scan time, and the spectral range you will save. Below, select Transmittance and select the data blocks to be saved.

6. **Optic:** Controls optical parameters, most notably, the aperture size can be set here as well as the scanner velocity and any signal gain needed.

7. **Acquisition:** Options don't generally need to be changed here. You will find filter and acquisition mode options here.

8. **FT:** You will find parameters associated with the Fourier Transform here. For general purpose spectroscopy, use *the Mertz correction* mode with a *phase resolution of 16* and a *Blackman-Harris 3-Term apodization function*. Zerofilling can also be set here, but note that you can always zero fill your spectrum after you acquire it (under the 'Manipulate' dropdown or toolbar).

9. **Display:** Allows you to set up how your spectra will be displayed. You also have the option of displaying scans before and during the experiment.

10. **Background:** If you would like to use a preexisting background instead of acquiring one prior to acquisition, you can load it here (*this is not recommended*).

11. **Check Signal:** Check the HeNe interferogram and press 'Save Peak Position.' The amplitude should always be less than 30,000 (absolute value).

12. **Beam Path:** Displays a schematic of the beam-path and cannot be edited.

13. **Spectral Range Selection:** The spectral-range can be edited here, but note that you will only save the data specified under the **Advanced** tab. Note that the selected spectral range (white region) must be within

the operational ranges of the optical components displayed below the spectral selection graphic.

14. **Collect a background spectrum:** Once all parameters are set, return to the **Basic** tab, and click 'Background Single Channel' to collect a background spectrum.

15. **Collect a sample spectrum:** Once the message bar on the bottom of the screen is grey, and displays 'No Active Tasks,' open the sample compartment, load your sample in the sample holder, close the compartment, and click 'Sample Single Channel' under **Basic**

16. **When you are finished:** Remove your sample from the sample holder and close OPUS. Close the the sample compartment and leave the air-compressor on.

Quickstart: ATR

1. Ensure the air compressor is on (green button on the left)

2. Start up OPUS and perform any PQ tests as needed before loading sample (**Optics Diagnostic** button).

3. **Check the Throughput of the ATR accessory:**

a) Click the 'Advanced data collection' button and load MIR_ATR_Throughput.XPM under **Basic**.

b) Take a background with the empty transmittance sample holder in the compartment: **Basic** > 'Background Single Channel.'

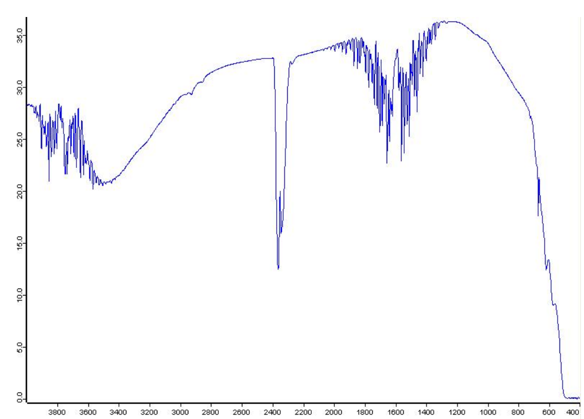
c) Open the sample compartment, push the base-plate trigger on the bottom of the compartment and remove the transmittance sample holder.

d) Install the VeeMAX II accessory by lining up it's pin-hole connector with the base-plate of the spectrophotometer. Once the instrument begins to beep, push it down into place while pushing the base-plate trigger (it should click).

e) Place the ZnSe ATR crystal, flat side up, on the open end of the VeeMAX II and set the incident angle to 45 °. Install the pressure-tower on top of the accessory; you will have to push down on the tower's screws before screwing them down.

f) Take a sample spectrum, with nothing on the ATR crystal: **Basic** > 'Sample Single Channel.'

The spectrum should look similar to the one below, and the % transmittance at 1000 cm^{-1} should be greater than 20 %T (typically 35 %T). If the throughput is significantly below 20%T, either realign the VeeMAX II accessory (see the manual) or contact the FTIR czar (Joe Elias: 610-304-2647).



4. Under 'Advanced data collection,' load the experiment 'MIR_ATR.XPM and adjust desired parameters (see steps 4 - 12 under 'Quickstart: Transmittance' above), making sure to select 'Absorbance' as the resulting spectrum under the **Advanced** tab.

5.**Collect a background spectrum:** With the clean ZnSe crystal mounted, take a background: **Basic** > 'Background single channel'

6. **Collect a sample spectrum:**

a) Cover the ZnSe crystal surface with your sample, place a glass slide on the top, and screw down the pressure tower until it clicks.

a) Collect a sample spectrum: **Basic** > 'Sample Single Channel.'

8. **When you are finished:** disassemble the pressure-tower, pour off your sample (*never* scrape it off of the crystal with *anything* dry), clean the crystal with a *kimwipe wetted in acetone*, and replace the transmittance cell to the sample compartment. Close OPUS. Leave the air-compressor on with the sample compartment closed.

Quickstart: DRIFTS

1. Ensure the air compressor is on (green button on the left)

2. Start up OPUS and perform any PQ tests as needed before loading sample (**Optics Diagnostic** button).

3. **Check the throughput of the DRIFTS accessory:**

a) Click the 'Advanced data collection' button and load the MIR_Throughput.XPM experiment.

b) Take a background with the empty transmittance holder in the sample compartment: **Basic** > 'Background Single Channel.'

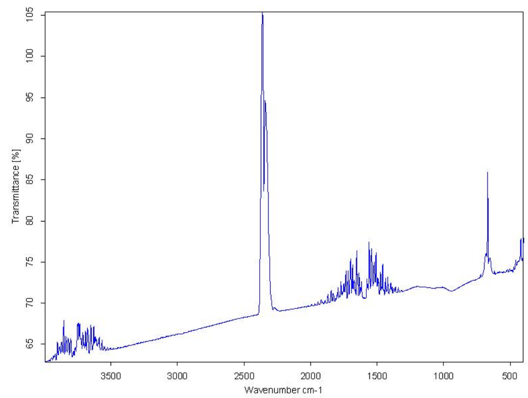
c) Open the sample compartment, push the base-plate trigger on the bottom of the compartment and removed the transmittance holder.

d) Install the DRIFTS accessory by lining up it's pin-hole connector with the base-plate of the spectrometer. Once the instrument begins to beep, push it down into place while pushing the base-plate trigger (it should click). Extend the telescoping purge-seal tubes so they fit snugly over the FTIR's windows, and tighten their screws.

e) Open the DRIFTS sample compartment and insert the alignment fixture with the horizontal mirror first. Maximize the signal intensity by adjusting the sample stage height (knob on right) while monitoring the HeNe interferogram's amplitude in the **Check Signal** tab.

f) Collect a sample spectrum: **Basic** > 'Sample Single Channel' .

The resulting spectrum should look like the one below. The throughput of the accessory at 2500 cm^{-1} should be at least 40 % T (typically 65%). Refer to the Praying Mantis manual if the throughput is less than 40 % or contact the FTIR czar (Joe Elias: 610-304-2647)



4. **Collect a KBr background:**

a) Open OPUS and load the MIR_QuickDRIFTS.XPM experiment. Vary any parameters, as described in steps 4 - 12 in the 'Quickstart: Transmittance' section, making sure the resulting spectrum is in Kubelka-Monk units.

b) Load a DRIFTS sample cup with KBr (located in a glass desiccator in lab 31), level off the surface with a flat blade.

c) Insert the KBr sample cup into the sample compartment of the DRIFTS accessory and close the lid.

d) Maximize the signal intensity (**Check Signal** tab) by adjusting the sample height knob. *IMPORTANT:* The height knob does not have a stop; the sample cup should never come very close to the parabolic mirrors.

e) Take a background: **Basic** > 'Background Single Channel.'

5. **Collect a sample spectrum**

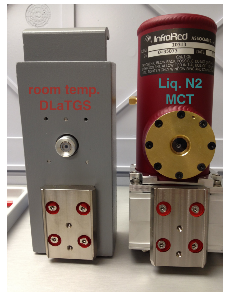
a) Unload the sample cup, and replace it with your sample (diluted in KBr if it is a strong absorber). Level off the surface and insert it back into the DRIFTS accessory.

b) Collect a sample spectrum: **Basic** > 'Sample Single Channel.'

6. **When you are finished:** Remove sample from DRIFTS accessory, empty the cup and wash it out with acetone. Remove the DRIFTS accessory and replace it with the transmittance cell in the sample compartment and close OPUS. Leave the air-compressor on with the sample compartment closed.

Changing the Detector

Time-resolved spectroscopy (rapid scan and step-scan techniques) require a detector with a faster response than the standard, room-temperature DLaTGS. We use a photoconductive, liquid nitrogen-cooled mercury cadmium telluride (MCT) detector with a spectral range of 12,000 - 850 cm^{-1} (the DLaTGS is 8,000 - 350 cm^{-1}). The MCT detector is cooled with liquid nitrogen using the supplied funnel once the MCT is installed.



1. **Remove the DLaTGS detector:**

a) Open the compartment on the top right of the spectrometer to recover the red hex tool. Unscrew and removed the detector panel (left panel).

b) Gently unscrew the hex screw at the base of the DLaTGS detector (green arrow) and remove the detector by pulling up with its handle. The DLaTGS should be stored with desiccant (silica gel or drierite) if unused for extended periods of time.

2. **Insert the MCT detector:**

a) Remove the plastic cap from the MCT detector and *carefully* insert it into the detector mount until it is flush and the instrument beeps.

b) Screw in the MCT detector into the detector mount with the red hex tool.

3. **Install the dewar connector:**

a) Screw the spring-loaded dewar connector to the top of the MCT detector as shown above.

b) Remove the plastic cap from the left port of the detector panel and replace the panel, screwing it in while ensuring that the dewar connector and O-ring are lined up with the port on the panel.

c) Insert the blue plastic adaptor into the dewar connector on the left port: Scuba-Steve in Cyborg mode.

4. **When you are finished:** Reinstall the DLaTGS detector by going through steps 1-3 above in reverse. Place the empty MCT detector back in it's box.