SOP for Bruker Vertex FT-IR Spectrometer

Note: This document is frequently updated; if you feel that information should be added, please indicate that to the facility manager (Currently Philip Carubia; pmc228@cornell.edu; Office, Bard B56; Phone, 255-6757).

- **Reservations and enabling on coral**
    - The Bruker Vertex FT-IR Spectrometer is interlocked with the CCMR Coral equipment reservation and enabling system. You need to have a CCMR user account in order to reserve and enable the equipment on Coral. More Information on getting an account and using coral can be found at: http://www.ccmr.cornell.edu/facilities/becoming-a-ccmr-facility-user/

About the Vertex FT_IR

- The Vertex FTIR is a high resolution vacuum spectrometer that is designed to perform in a research environment. The instrument boasts a spectral resolution that is better than 0.2cm-1 and a high signal to noise ratio particularly when the sample is placed in vacuum.
- There are two detectors associated with the Vertex FT-IR:
    - Mercury Cadmium Telluride (MCT): The MCT Detector uses liquid nitrogen to cool the detector to cryogenic temperatures which greatly reduces the noise and thus the sensitivity of the detector. The tradeoff is that the detector can saturate and the user needs to take precautions to adjust the incident light intensity to avoid saturation.
    - Deuterated triglycene sulfate (DTGS): The DTGS detector is good all-purpose detector that is suitable for moderately absorbing samples. The DTGS detector requires no cooling.
    - Both detectors can be used with any sample acquisition mode (ATR, transmission, Etc.). Instructions for using each detector can be found below.

Getting Started

- **Equipment to be left on at all times**
    - The power to the spectrometer should remain on at all times.
    - Due to the water sensitive KBr beam splitter located in the optical bench of the spectrometer, the optical bench should remain under vacuum at all times to prevent water damage to the beam splitter.

  - The MCT detector requires cooling using liquid nitrogen. To fill the detector you must first fill the 1 L Dewar from the 50 L Dewar. When fully charged with LN2 the detector should stay cool for 4 to 6 hours.
    - To fill the 1 L Dewar: Adorn required safety gear: safety glasses, cryogenic gloves, and closed toe shoes. Place the 1 L Dewar on the floor next to the 50 L Dewar. Gently place the filling tube in the small Dewar and slowly open the liquid valve on the Dewar. It will take some time for the N2 gas to evacuate the hose before LN2 will begin flowing. Be cautious to not open the liquid valve to much as the bottom of the small Dewar is curved and it will shoot LN2 back at you. Fill the Dewar ~¼ full.
    - To fill the detector. Remove the plug on the top of the microscope exposing the detector fill tube. Insert the liquid nitrogen funnel into the tube. Pour from the 1 L Dewar into the funnel. A room temperature detector will take approximately 4 funnels worth of LN2. Try not to overfill the detector; if it is overfilled, LN2 will spill across the top of the microscope, Step back and allow excess to run off without spilling on you.
- **Log on to computer**
  - Log on using the FTIR User Account. The Password is FTIR
- **Start OPUS**
  - Double click the OPUS 6.0 Icon
  - Log in screen will appear enter the following fields
    - Find your user ID (it will be your Cornell ID)
    - The default password is OPUS
    - Press login button; press OK on the about OPUS Pop up window.
    - You can change your password from the default once in OPUS.
  - You have been assigned a workspace file. This file has the extension .ows. This file is specific to your user account and can be modified by you. It is how you will interact with opus. You can add or remove tool bars, customize the view and save in your user folder which is named with your Cornell user I.D.
  - Note that the status light in the bottom right hand corner of OPUS is green. If the light is red there is a problem with the microscope or spectrometer that needs to be corrected before taking measurements. Please contact the facility manager.
  - Opus has an excellent help menu. Almost every OPUS Dialogue contains a help button which leads you directly to the appropriate online description. This feature can be extremely helpful.

To set up for a measurement go to: Measurement > Advanced Measurement

- **Basic tab**
    - Be sure that you have entered in a sample description and sample form. They will be used to name your files when you save them. Files will be saved in C:\Documents and Settings\Administrator\Administrators documents\FTIR Users\"Cornell ID"
    - The file will be saved automatically with the following convention: Sample Description_Date. Sample Description and Sample Form can be modified on the advanced tab in advanced measurement.
    - Once the experiment has been set up you will come back to the basic tab to take the background single channel and sample single channel.
    - The vent sample compartment button allows the sample compartment to come up to atmospheric pressure for loading samples etc.
    - Please do not use the vent optical bench button as it will expose the water sensitive beam-splitter to atmospheric moisture. If you do accidentally vent this chamber just re-evacuate.
    - Opus saves files with a numerical file extension starting at .0
    - If files use the same name they will be appended with .01 .02 .03… .0x
    - File name and File path on advanced tab should not be changed as they will populate automatically using the guidelines stated above.
- **Advanced Tab**
    - Do not change the File name or path on the advanced tab!
    - Resolution: This should generally be set to 4 cm^{-1}. This value can be set as low as 0.2cm^-1 but it will drastically increase the time that it takes to take a spectrum.
    - Fill in the Sample scan time, Background scan time. This can be entered as a time or as a specific number of scans. Sample and background scan time do not need to be the same. If you have a weak signal you can increase the scan time which will allow for greater exposure to the detector. Also increasing the scan time will increase your signal to noise ratio.
    - Enter spectral range for saved data and the results spectrum type that you would like to view your data in. Generally for this spectrometer minimum value is 600cm-1 and maximum is ~5000cm-1.
    - Select the data-blocks that you would like to save. I typically recommend saving the sample single channel, background single channel and the absorbance or transmittance spectrum.
- **Optic tab**
    - External Synchronization
        - This should be set to off
    - Source setting

        - There is only one source, MIR.
    - Optical filter setting
        - Generally should be open
    - Aperture setting
        - Adjust this to increase or decrease the signal strength. This aperture is used to attenuate the signal to prevent the MCT detector from saturating. Also preamp gain can be adjusted to raise or lower the signal strength.
    - Measurement Channel
        - No adjustments necessary.
    - Background meas. Channel
        - This setting should be the same as the measurement channel
    - Detector setting: choose the detector that is appropriate for you sample.
        - The MCT detector is use for samples with weak signal. Caution must be used to prevent saturation during acquisition of the background and sample single channel.
        - The DTGS detector can be used for all samples without worry of saturation but has a lower sensitivity and lower signal to noise ratio.
    - Scanner velocity
        - This should be set to 20kHz for the MCT detector and ≤10kHz for the DTGS detector.
    - Sample signal gain
        - Leave at Automatic
    - Background signal gain
        - Leave at Automatic
    - Delay after device change
        - Change as needed
    - Delay before measurement
        - `Change as needed
    - Optical bench ready
        - Should be set to off
- **Check Signal tab**
    - The check signal tab allows the user to view the sample spectrum and the interferogram in real time. It is recommended that you check this tab prior to taking your spectrum. The show box located at the lower left of GUI allows you to toggle between the interferogram, spectrum, and the ADC count. The ADC count allows the user to monitor the Amplitude and zero-crossing position from a distance. The user also has the ability to scale the display.

- **Saving .xpm file**
    - Once the measurement is set up you can return to the advanced tab and save the measurement. The measurement can be saved in your user folder; it will be saved with the file extension .xpm. A .xpm file can be created for different types of measurements. To save go to the advanced tab and click the save button. Enter the name for the file and ensure that the file path points to the proper user folder and save.

Note: The following tabs are for advanced measurements contact the facilities manager if you would like help using these settings. Making changes will not hurt the spectrometer but it is recommended that you save a backup of your .xpm file.

- **Acquisition tab**
    - **High and low frequency**
        - These limits can be adjusted to a lower bandwidth but the high pass and low pass filters must also be set to filter all wavelengths outside of the high and low limits.
    - Acquisition mode
        - This is typically set to double sided forward-back
    - Correlation mode
        - Correlation can be used to perform a data integrity check
- **FT tab**
    - Phase resolution
        - This is typically set to provide at least 250 phase interferogram points.
    - Phase correction
        - Is typically set to Mertz
    - Apodization function
        - Is typically set to Norton and Beer med for most applications. If you are trying to attain a very high resolution you may want to consider other functions.
    - Zero filling factor
        - This will smooth your spectrum. The higher the number the greater the smoothing
- **Display tab**
    - Use to scale the display
- **Background tab**

- OPUS will use the most recent background spectrum taken when calculating your sample spectrum. If you would like to use a background spectrum that was taken previously you can load it on this tab and OPUS will use it.

- **Capabilities and notes**
  - Measurements in transmittance mode are suitable for very thin specimens (<50µm)
  - Refractive index of Ge ATR Crystal = 4. Sample must have a refractive index that is < ATR crystal.
  - Spectral range:
    - Beamsplitter: 5000 – 400 cm^{-1}
      - Note: it may be possible to attain measurements in the 6,000cm^{-1} range.
    - Mid band MCT detector: 12,000-600cm^{-1}
    - DTGS detector: 12,000 – 550cm^{-1}
  - Resolution: Better than 0.2cm^{-1}.
  - Wave number accuracy: better than 0.019cm^{-1} at 2,000 cm^{-1}