

## Use and Maintenance of Bruker Vertex80 FTIR

Manufacturer: Bruker  
Model: Vertex80

The instrument is equipped with a Bruker Platinum ATR cell in default. The sample cell can be replaced with a transmission cell or other compatible IR cells.

Workstation: username: (check the login notebook)

Password: (check the login notebook)

Data path: D:\...\BrukerFTIRData

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### 1 Turn on nitrogen purge at the nitrogen tank.

Check the instrument status LED lights (six LEDs) on the left back corner of the spectrometer. When all of the six LEDs are green, or five of them are green, except the "STATUS" LED, the instrument is good to operate. If the "PRESSURE" LED is red, which means the nitrogen supply runs out, the spectrometer is not ready to work. Please check the gas supply (the liquid nitrogen tank or nitrogen cylinder), make sure the outlet valve on the tank/cylinder is open, and the pressure is in proper range (30-40psi). If the liquid nitrogen tank or nitrogen cylinder is empty, please replace the LN tank or N2 cylinder.

**Set the rotameter to 20 sccm.**

2 Sample preparation. The default sample cell is the Bruker Platinum ATR cell, which is equipped with a diamond ATR plate on which one total reflection will take place. The ATR cell works for both liquid samples and solid samples.

#### 2.1 Liquid samples

Liquid samples can be directly added on top of the ATR plate. One drop will be good enough for the measurement.

#### 2.2 Solid samples

Solid samples can be powders or films. Add the proper amount of sample to cover the ATR plate. Turn the metal tip down, and turn the knob to adjust the height until the sample is firmly pressed onto the ATR plate. Do not press too hard.

#### 2.3 Cleaning of the ATR cell.

Use proper solvent and KIM wipe to clean the surface of the ATR cell. The ATR plate is made of diamond, and the sealing is platinum. For the cell itself, there is no limitation for the cleaning solvent. But considering the solvent residual, low boiling solvent is recommended.

### 3 Software

Data acquisition and analysis are both using OPUS. There are two software icons on the desktop, one called "OPUS", and the other one called "UWiz". "OPUS" is recommended to access fully functional capability.

Double click on the "OPUS" icon on the desktop. In the login dialog box, input following information:

UserID: default  
Password: OPUS  
Workspace: default

### 4 Set up method.

Use the little test-tube icon or "Measurement" -> "Advanced Measurement" to open the method setup page for continuous scan.

4.1 "Basic" page: Input sample descriptions, including the sample name, sample format, operator name, etc. These information will be added to the data file as comments.

#### 4.2 "Advanced" page:

4.2.1 File name: this will be the name of the data file.

4.2.2 Data Path: Create user folder under the "BrukerFTIRData" folder. The "BrukerFTIRData" folder is uploaded to the Catalysis Facility data server.

4.2.3 Resolution: this is the spectrum resolution. 4cm<sup>-1</sup> is the most commonly used setup. Users can adjust this resolution number according to requirement. This number can be set as small as 0.2cm<sup>-1</sup>.

3.4.2.4 Sample scan time and background scan time: these two numbers mean how many scans will be taken for average to get the final result. Users can adjust this number according to their applications.

4.2.5 Data saving range: the spectrometer is equipped with a mid-IR source, which covers the wavenumber range of 4000-400cm<sup>-1</sup>. Users may adjust this range according to requirements, but the broadest range will be 4000-400cm<sup>-1</sup>.

4.2.6 IR spectrum can be collected in the format of transmittance or absorbance. In the data blocks, users can decide which traces to save in the data set. The "Transmittance" or "Absorbance" trace will be the most important information to collect. The "sample single channel" trace is the raw signal the detector sees, and the "background single channel" will be the raw signal for the background spectrum. These two traces are recommended to be kept in the data set. Check the box for "subtract water vapor peaks", then water vapor peaks will be subtracted from the transmittance or absorbance result. 3.4.3 "Optic" page:

External synchronization: Off

IR Source: Mid-IR

Beam Splitter: KBr

Aperture: 6mm for the ATR cell

Background channel: sample compartment

Measurement channel: sample compartment

Detector: There are two detectors available, select Detector 1 for the Room temperature detector, Detector 2 for the MCT detector, which works under liquid nitrogen temperature. The MCT detector provides higher sensitivity, and is capable of time-resolved measurements. If the MCT detector is selected, the user should bring and fill liquid nitrogen into the detector. A liquid nitrogen funnel is available in the drawer under the IR spectrometer.

Velocity: For RT detector, set at 10kHz to check signal, a signal ~5700 is expected. Upon the expected signal intensity, the velocity should be set at 40kHz for measurements. 40 kHz is the fastest scan speed for the RT detector. For the MCT detector, the velocity can go up to 320 kHz.

4.4 "Acquisition" page: make sure the acquisition range is broader than the data saving range (the number set on the Advanced page), there are no other adjustments need to be made on this page.

4.5 "Interferogram" page: The "phase resolution" should be adjusted to 8 times of the "resolution" on the "advanced" page. There are no other changes that need to be made on this page.

4.6 "Background" page: nothing to input on this page.

4.7: "Check signal": This page was mentioned before in the "velocity" set up.

4.8: "Beam path" page: this page displays a map of the beam path, which is quite helpful to understand the parts of the IR spectrometer.

4.9: "Spectrum range" page: this page displays the spectrum range for this instrument (4000-400 cm<sup>-1</sup>).

## 5 Measurement:

### 5.1: Collect background spectrum:

Clean the surface of the ATR cell. On the "Basic" page, click the "Background Single Channel" button to collect the background spectrum. The background spectrum will be updated and applied to the following samples until a new background is collected. It doesn't really matter whether the tip is on or off for the background measurement

### 5.2: Collect the sample spectrum:

Add sample on the ATR plate. Use the metal tip to press solid samples firmly onto the plate. Click the "Sample Single Channel" button to collect the sample spectrum.

5.3: When the measurement is completed, the collected data will be displayed in the OPUS Browser. The transmittance/absorbance spectrum will be displayed on the screen.

## 6 Data analysis

6.1: Transmittance-Absorbance convertor.

6.2: Baseline correction

6.3: Spectra subtraction

6.4: spectrum calculator

6.5: Peak picking tool

6.6: Integration

6.7: Curve fitting tool

6.8: Print -> Print spectra -> save as PDF

6.9: File -> Save as -> format tag -> data point table -> save as .dpt file, which could be opened in notepad, and imported into Excel or other data analysis software to create a figure.

6.10: right click menu: "shift curve" options; "single peak picking"

## 7 Troubleshooting

The most common issue for this instrument is that the nitrogen supply runs out. This IR spectrometer needs nitrogen to provide a back pressure for regular performance. Without sufficient nitrogen flow the instrument cannot work.

Check the status LEDs, when the Pressure light is red, it means that the nitrogen flow is not sufficient. Check the nitrogen tank and replace it when it runs empty.

Sometimes when users try to open OPUS and see an error, open the task manager and force quit the opus.exe in the process, the issue will be cleared.

#### 8 Maintenance

8.1 Replace the liquid nitrogen tank when the one hooked to the instrument runs out

8.2 Replace the IR source when the signal intensity on "check signal" page reads abnormally (expected to be ~5700 for RT detector with the ATR cell, aperture 6mm, velocity 10kHz)

8.3 The MCT detector should be evacuated overnight annually.