

# Instruction Manual FTIR

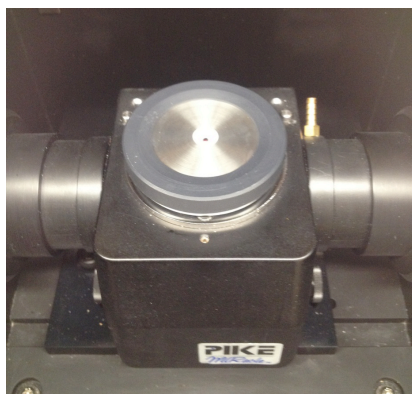
## 1. Starting the Spectrophotometer

- 1.1 Turn on the FTIR
- 1.2 Click on IRSolution on the Desktop
- 1.3 IRsolution starts
- 1.4 Click on [Measure] button
- 1.5 Select [Measurement] menu on the IRsolution
- 1.6 Select [Initialize] on the menu
- 1.7 The PC initiates communication with the FTIR
- 1.8 To accept the Autoadjust process, click OK.
- 1.9 When the Status Monitor on the right window shows a Green color the system is OK (Humidity - Lamp - Laser)

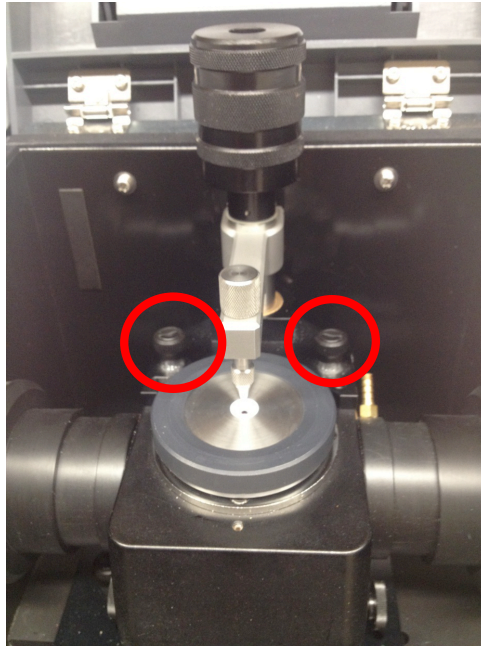


## 2. Data Acquisition and Processing

- 2.1 Scan Parameters on the right of the window
  - 2.1.1 Measurement Mode: % Transmittance
  - 2.1.2 Apodization: in normal measurement, select “Happ-Genzel”, in measurement of high resolution, such as gas, “box-car” of higher resolution can be selected, and in ultra-micro analysis in which better S/N ration is required, “SqrTriangle” can be selected.
  - 2.1.3 No. of Scans: 4 to 4000 maxi
  - 2.1.4 Resolution: best 2.0 to 4.0
  - 2.1.5 Range: 600 Min and 4000 Max  $\text{cm}^{-1}$
- 2.2 Mounting Procedures for pressure clamp
  - 2.2.1 The IR will be assembled with the ATR crystal plate, mounting ring, and liquids retainer (picture 1).

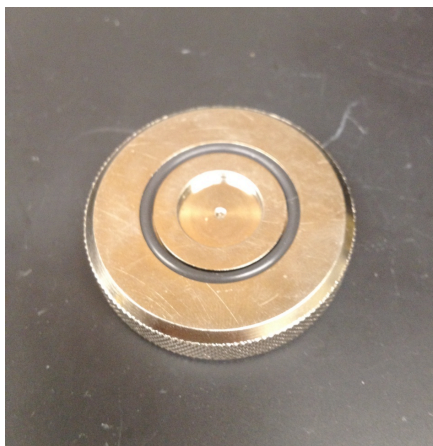


**Picture 1:** ATR crystal plate assembly.



**Picture 2:** High pressure clamp attachment.

- 2.2.2 Attach the High pressure clamp using the two captive thumbscrews (see picture 2) and tightening them completely.
- 2.2.3 In the case of measuring powders or liquids (neat), the setup will look like picture 2. If measuring crystals, the mounting ring and liquids retainer are not necessary. If measuring solutions with volatile solvents, a disk (picture 3) can be placed on top of the crystals plate to prevent solvent loss during the measurement. Also, different tips (picture 4) can be used: the flat tip is used for most samples, such as powders, the concave tip is used with beads or pellets, and the swivel tip can be used for polymers or irregularly-shaped materials as well as powders if aligned properly. Please replace with flat tip if you change for your measurement.

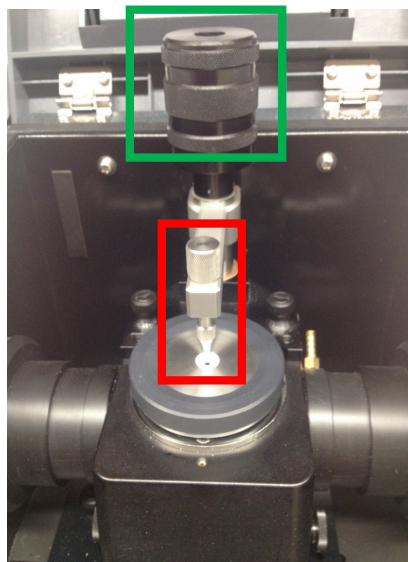


**Picture 3:** Disk to prevent solvent loss.



**Picture 4:** Different sampling tips.

- 2.2.4 **WARNING:** The crystal plate can be damaged by improper use of the pressure clamp. At all times the silver screw tip (red box, picture 5) above the sample area should be fully screwed in away from the crystal surface. Then, the black pressure lever (green box, picture 5) should be slowly turned to lower the clamp onto the crystal surface and apply a light pressure on your sample. Once a slight resistance is felt stop turning. High pressures will crack the crystal surface.



**Picture 5:** Pressure plate adjustment.

- 2.2.5 There are two different types of crystal plates available: Diamond with Zn/Se backing or just a Zn/Se crystal plate. The diamond plate will be attached to the instrument normally and should work for most measurements. It is chemically inert and can withstand the high pressure but still be careful. The Zn/Se plate is more reactive and cannot be used with strong acids or bases or any complexing reagents, such as EDTA.

**\*\*If you need to change the plate (ie your sample contains graphite or something with a refractive index like diamond), you MUST contact one of the people in charge of the IR as the IR will need to be purged after the change.\*\***

## 2.3 Measuring the background

- 2.2.1 In the Measurement File Area complete - Comment and Data File, for example: Bkg-1
- 2.3.2 Click the [BKG] button to start the background, verify the system is clean, and no sample is the set in the sample hole. Use the same pressure setup as you will use for your sample. Keep all the same parameters (ie number of scans) for the background and the sample measurements.

## 2.4 Setting the sample

- 2.4.1 Added the sample through the Sampling Area just enough to cover the red light (5-10 mg). Lower the pressure module with the black control lever (green box, picture 5) until slight pressure is applied.

- 2.4.2 In the Measurement File Area completed - Comment and Data File, for example: SG041
- 2.4.3 Check the Auto Increment stays Blank and your spectrum will be saved automatically in your folder.
- 2.4.4 Click the [Sample] button to start the sample measurement. There is no waiting period after clicking [Sample], so make sure your sample is prepared and the pressure clamp has been lowered.
- 2.5 Display View
  - 2.5.1 Click on the [View] Tab to display the view window, the upper window is called the Overview window and the lower one the Zooming window.
  - 2.5.2 For Zooming in a spectrum using the mouse to create a rectangle by dragging and dropping on the zooming window. Click the position to be zoomed. The zoomed position is displayed on the Overview Window (up).
  - 2.5.3 Click the right side of the mouse to display the menu Autoscale – Range List and Full view.
- 2.6 Manipulation
  - 2.6.1 For Baseline Correction, select [Baseline] on the pull-down menu of [Manipulation1] to display three types of baseline corrections, [Zero], [3 point] and [Multipoint]
  - 2.6.2 For Smoothing, select [Smoothing] on the pull-down menu of [Manipulation1], adjust the noise by increasing the Number of Points.
  - 2.6.3 For labeling the peaks, select [Peaktable] on the pull-down menu of [Manipulation1]. The [Peak Detection] parameters will appear on the right side of the window. First, click calculate to give peaks below your threshold (ie 80 %) and then add the peaks manually or automatically. If necessary, adjust the Noise and the Minimum Area; however, the Threshold has to be adjusted every time. Click OK to save the peak values and display them on the spectrum.
  - 2.6.4 For general changes to the display, right click on Graph preferences to adjust the color, the size of the scale and peak values, and the thickness of the spectrum line. The color will not store for the printing described below, so if black is needed simply take a screen shot and save your spectrum as an image file for presentation purposes.
- 2.7 Print
  - 2.7.1 Click on [Print form] button.
  - 2.7.2 Add title in text box displayed.
  - 2.7.3 Keep the Template open and select [File] menu.
  - 2.7.4 Click on Print Preview and Print your spectrum.
  - 2.7.5 Your spectrum will be printed to the printer of your choice.
  - 2.7.6 Also, you can take a screen shot of your spectrum and print the image.



- 2.8 Save
  - 2.8.1 The spectrum should be saved automatically after measurement in personal folder.
  - 2.8.2 Select [File] button and go to Save As, the personal file will be saved as a .smf format.
  - 2.8.3 To export the personal data, go to [File] menu, select Export (.txt, .asc, .irs, .dx).

### **3. Cleaning the FTIR**

- 3.1 Isopropyl alcohol is used to clean the FTIR.
- 3.2 Unscrew all parts on the ATR (high pressure clamp, mounting ring, and the liquids retainer) and apply the solvent with Kimwipes on all elements.
- 3.3 Gently remove all of the sample. Do NOT try to recollect your sample by scrapping it off the crystal surface.
- 3.4 Repeat this procedure until all traces of the sample have been removed.
- 3.5 Replaced the high pressure clamp in the black box.
- 3.6 Many paper products are abrasive and could cause scratching of the soft crystal surfaces, such as Zn/Se, so only use cotton balls for cleaning this crystal.

### **4. Quitting the System**

- 4.1 Exit the IRsolution software
- 4.2 Turn off the FTIR and close the ATR cover.
- 4.3 Complete the logbook.

### **5. Important Notes**

- 5.1 Please use the high pressure clamp according to the rules above. Excessive force will crack the crystal surface, and they are expensive to replace (\$1500-4000).
- 5.2 If they are errors/problems with the instrument or measurement, please record them in the logbook and contact the person in charge.
- 5.3 One common problem will be the appearance of two strong bands around  $2300\text{-}2400\text{ cm}^{-1}$  (usually out-of-phase). These are CO stretches caused by the ionization of air in the machine. This can be eliminated by purging the instrument with nitrogen gas for 2-3 hours, but the people in charge must know about the problem to fix it. Also, sometimes the noise regions can get very bad on the spectrum, so please report any of these issues.