

Jasco 4700 FTIR

Operation instructions

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Turn the system on

- 1. Turn on the computer in case it has been switched off. Normally computer stays on.
- 2. The instrument should be always in standby mode and RESUME button should be always ON. Do not touch it, please.
- 3. Turn on FTIR (ON/OFF switch on top of the instrument). Wait for 3 characteristics beeps from the instrument.



4. Double-click "Spectra Manager" icon on a desktop. Double click on Spectra Measurement.



Cleaning the ATR module

Sometimes it is worth to clean the ATR crystal before your measurments since the previous user may fail to do the proper job. You can skip this step if you are not concerned about previous user or your background spectrum remain only the features of H₂O and CO₂.

- 1. Do not use any spatulas or sticks!!!
- 2. With the remaining solid at the edges, apply some methanol (please, do not use acetone), dab the top with a Kim-wipe paper. Repeat this 4-5 times.
- 3. With methanol and Kim-wipe scrub the general area in and around the crystal surface. The solvent should not be applied in a way that the setup is soaked since it will leak into the optics as well. Soak the paper only and then apply the paper to the cleaning area.
- 4. Do not forget to clean the pressing pin.

Sample rules

- 1. For liquid sample the pH factor has to be from 5 to 9.
- 2. For solid samples like wafer and single crystals, please, check with Laboratory Director prior to the perform experiments.

Acquiring the background spectrum

A background measurement must be made prior to each sample measurement.

1. Confirm that the sample chamber is empty, then choose **Monitor Background** from the **Measure** menu of the Spectra Measurement program or using the shortcut icon.



2. The **Monitor Background** window will be promptly displayed. The spectrum is displayed in the **Monitor Background** window in real time, so measurement parameters may be changed while observing the spectrum change. Note that the spectrum preview is always displayed using an instrument resolution of 16 cm⁻¹, and that changing the wavenumber range may not immediately alter the preview.



- 3. On the **Standard** tab
 - Set the Number of Scan. Click on the Auto check box to deselect Auto if necessary.
 - Set the **Resolution** by changing the value in the drop-down list.
 - Set the **Range** of the measurement.
 - Check the **Fit "view" range to "data" range** check box if necessary.
 - Set **Background** to Single. This can be set using the same procedure that was used to change **Resolution**.

🔤 Parameters Advanced 🛛 🚺 🔀			
Standard Information Data Optics FFT/Timer			
Number of scan : 40			
Estimated scan time 00:00:41			
Resolution : 4.0cm-1			
Range : 7800 - 500 cm-1			
<u>V</u> iew range 7800 - 500 cm-1			
Eit "view" range to "data" range			
Vertical axis			
Sample: %T 🖌			
Background : Single			
Auto baseline correction Settings			
Auto background scan Settings			
Evaluation Settings			
Accessroy : Not equipped			
Default Basic			
Open Save OK Cancel Help			

- 4. Select the **Information** tab.
 - Enter the **Sample Name**. Change other settings as desired.
 - The **Operator** and **Division** fields are input automatically for the current user.
 - To input a **Comment**, click on the comment field.
 - If the **Information** fields are to be changed before every measurement, check the **Display "information" dialog before measurement** check box.
- 5. Select the **Data** tab.
 - Select %T, Abs, %R, Int from the drop down menu for the Data Type.
 - Ensure that Auto save and Print out data are unchecked (i.e., OFF), but that Send data to "spectra analysis" is checked.
- 6. Select the **Optics** tab.
 - **Source** is "Standard".
 - **Detector** is "TGS"
 - Sample chamber is "Standard".
 - In addition, all of the IF (interferogram) parameters (Gain, Aperture, Scan speed and Filter) are set to "Auto".
- 7. Select the **FFT/Timer** tab.
 - Apodization function is "Cosine".
 - Phase correction is "Mertz".
 - **Phase correction filter** is "Auto".
 - Check the **Zero filling** check box and uncheck the **Use timer** check box.

Standard Information Sample : Diperator : Division Comment :	Data	Optics	FFT/Timer
Sample : Operator : Division Comment :			
Display "informatio	n'' dialog	g before	
easurement measurement			
Default			Basic

Standard Info	mation Data Op	tics FFT/Timer
Data type :	%T, Abs, %R,	Int 📓
Auto save	o save	
Folder :		
		Browse
Eormat :	Date-No.	~
String :		
Send data		
Send data		
Send da	ata to "spectra analysi	s''
Print out	ata to "spectra analys	s"
Print out	ata to ''spectra analysi t data	s"
Print out Print out Print out Iemplate :	ata to "spectra analysi : data	s"
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Print out Print out Print out Iemplate :	ata to ''spectra analysi : data	BIOWSE
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Print out Print out Print out Iemplate : Default	ta to ''spectra analysi : data	BIOWSE

Source : Standard Detector : TGS Sample chamber Standard Accessory : Not equipped Gain : Auto Aperture : Auto Scan speed : Auto Filter : Auto Optical Filter : Ge/KBr	tandard Informatio	n Data Op	tics FFT/Timer
Detector : TGS Sample chamber Standard Accessory : Not equipped Gain : Auto Aperture : Auto Scan speed : Auto Filter : Auto Optical Filter : Ge/KBr	Source:	Standard	-
Sample chamber Standard Accessory: Not equipped Gain: Auto Aperture: Auto Scan speed: Auto Filter: Auto Optical Filter: Ge/KBr	Detector :	TGS	~
Accessory: Not equipped Gain:: Auto Aperture: Auto Scan speed: Auto Filter: Auto Optical Filter: Ge/KBr	Sample chamber	Standard	~
Gain : Auto Aperture : Auto Scan speed : Auto Filter : Auto Optical Filter : ✓ Beam splitter : Ge/KBr	Accessory :	Not equipped	
Aperture : Auto Scan speed : Auto Filter : Auto Optical Filter : ✓ Beam splitter : Ge/KBr	<u>G</u> ain :	Auto	~
Scan speed : Auto	Aperture :	Auto	~
Filter : Auto	Scan speed :	Auto	
Optical Filter : v	<u>F</u> ilter :	Auto	~
Beam splitter : Ge/KBr	Optical Filter :		~
	Beam splitter :	Ge/KBr	
	Optical Filter : Beam splitter :	Ge/KBr	×
	1.0		

Parame	eters Ad	vanced		
Standard	Information	Data	Optics	FFT/Timer
Apodiza	ation <u>f</u> unction	n:		
Cosine	2	~		
✓ Zero Phase	o filling			
Mertz		~		
<u>F</u> ilter wi	dth :			
Auto		V		
<u>Interva</u>	timer I	min (0	sec
Defa	ult	OK	Cance	Basic

8. Click the **OK** button to start actual measurement. The following screen is displayed. A progress bar is displayed in the toolbar while data measurement is in progress. When the background measurement is finished, sample measurement may begin.



9. If you would like to re-measure the background spectrum with the same paramters, just click the following icon:



Sample setup on the ATR module

- 1. Apply one drop or a small micro-spatula portion (15-20 mg) to fill the dwell 60-80% full **Make sure not to scratch the crystal with the spatula**. If possible use the soft material spatulas (wood, rubber). If you use metal spatulas, do not touch the crystal surface.
- 2. Apply the press to the sample. Use only two fingers to screw the press. Normally once you feel the resistance while screwing by two fingers is when the applied pressure is enough for the measurement.

Sample measurement

The instrument provides a %T, %R or Abs spectrum by measuring a Single spectrum with the sample in the sample compartment, and obtains the ratio to the background already measured (stored in instrument memory). Solvents can also be measured as the background and used as a reference spectrum.

1. Select **Monitor Sample** from the **Measure** menu of the Spectra Measurement program or shortcut icon.





2. The Monitor Sample window opens.

- 3. Select the **Information** tab in the **Monitor Sample** dialog, and enter sample name. The comment can also be changed if desired.
- 4. Verify the preview of the spectrum profile and click the **OK** button to start measurement. A progress bar appears during measurement as for background measurement.
- 5. When measurement finishes, the Spectra Analysis program is started automatically and a window displaying the last spectrum measured opens. This window is called a View window. View (Memory-1) is displayed on the title bar because the measured spectrum is not yet saved to disk. If the measured spectrum did not automatically appear in the Spectra Analysis window, the Send Data check box in the Data parameters tab may not have been checked. Select File Send Data from the main menu to transfer the spectrum to the Spectra Analysis program.



- 6. The spectrum can be saved to file from either the Spectra Analysis or Spectra Measurement programs as follows:
 - Select Save As... from the File menu. The Save As dialog appears.
 - We strongly recommend to save data both in Jasco format (*.jws) and ASCII format (*.csv, *.dat, *.txt).

Data processing

In Spectra Analysis you can use the following options/tricks to clean up your spectrum.

1. Click the ATR icon.



2. Click **OK**, and a 2nd "View" window pops up. Note that the peaks on the left side increased in size.



- 3. CO₂ reduction.
 - Click the CO₂ shortcut icon.



• Click **OK**, and a "View" window pops up. Note that the peaks in the CO2 window disappeared.



- 4. H2O reduction.
 - Click the H_2O shortcut icon





• Click **OK**, and a "View" window pops up. This step might have to be performed twice in order to remove the water peaks from both ranges.

- 5. Automatic baseline correction.
 - Click Automatic Baseline Correction shortcut icon.





• Click **OK**, and a "View" window pops up.

- 6. Peak find.
 - Click the "Peak Find" icon.



• Click **OK**, and a "View" window pops up. The noise level can be adjusted by either choosing the upper and lower limit numerically or by dragging the appropriate horizontal lines in place. Click "Apply." Additional peaks can be added by dragging the vertical line to the peak and then clicking the "Add" button.



Cleaning the ATR module

You have to clean ATR unit upon the end of your experiments.

Scratching the crystal surface must be avoided at all cost!

- 5. Do not use any spatulas or sticks!!!
- 6. With the remaining solid at the edges, apply some methanol (please, do not use acetone), dab the top with a Kim-wipe paper. Repeat this 4-5 times.
- 1. With methanol and Kim-wipe paper scrub the general area in and around the crystal surface. The solvent should not be applied in a way that the setup is soaked since it will leak into the optics as well. Soak the paper only and then apply the paper to the cleaning area.
- 2. Do not forget to clean the pressing pin.

Switching the instrument off

- 1. Switch off Spectra Measurement program.
- 2. Switch off the instrument **Power** button to OFF position.
- 3. Make sure you do not touch **Resume** button and it is ON position.