

IFS66V FTIR Operating Instructions

Revised 01/07/2015 S.V.

Location: 1240 Hach Hall
Contact: Steve Veysey, 1234 Hach Hall

Safety

All researchers working in 1240 Hach Hall must complete the EH&S course: *"Fire Safety and Extinguisher Training"*. When preparing samples in this room, please wear all appropriate personal protective equipment. Aprons, safety glasses, and rubber gloves are available in 1238A Hach Hall. If solvents are involved, consider preparing your sample in room 1238A.

Properly dispose of glass pipettes in the container provided. Waste solvents can be disposed of in the waste containers provided in 1238A. All of the computers in this lab have direct links from the desktop to MSDS sheets, the EH&S Laboratory Safety Manual and to the CIF Safety Manual.

Introduction

The Bruker IFS66V FT-IR is a versatile research grade instrument capable of measuring in the visible, near-IR, mid-IR and far-IR regions of the spectrum. Measurement in each spectral region requires a different configuration of source, beamsplitter, and detector elements. This document describes use of the instrument for routine measurements in the mid-IR spectral region, from 4000 cm^{-1} to 400 cm^{-1} .



IFS 66v FT-IR SPECTROMETER

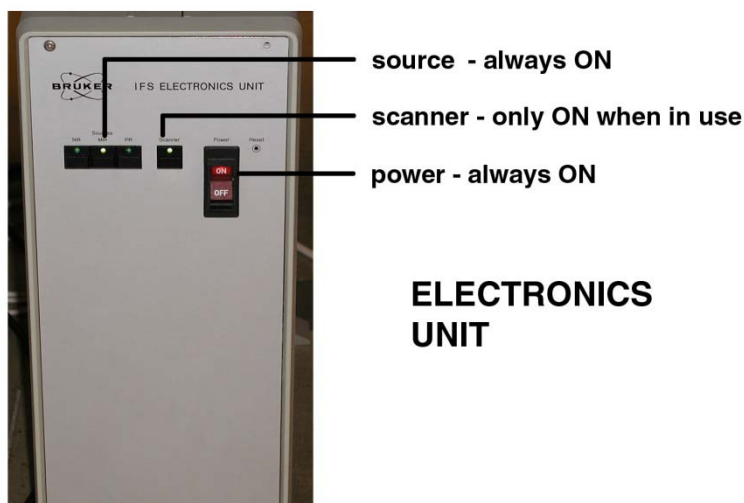
Overview

The IFS66V FTIR is controlled by a Windows XP PC communicating with several microprocessors located on the instrument. One additional microprocessor, the Vacuum Control Microprocessor (VCM), is not interfaced to the PC. You should not attempt to manipulate the VCM without first receiving permission.



DO NOT press any buttons on the vacuum control unit without permission!

You will however be required to turn on the “*scanner*” at the beginning of your session, and to turn it off when you are finished. This button is on the front of the IFS Electronics Unit located on the floor beneath the spectrometer table.



The “*power switch*” and “*glowbar source*” are normally left on at all times.

Sample and reference cells are placed into the purge box as required. It takes only a few seconds for the room air moisture to be purged from this small compartment. You are required to provide your own sample and reference cells. Be sure to insert your holder completely, and to immediately replace the plexiglass lid. The nitrogen purge flow can be increased for air-sensitive samples.



Samples are easily inserted into the "purge box"

If the purge box is not in place, the instrument may be configured for a special experiment. Consult with me before proceeding further.

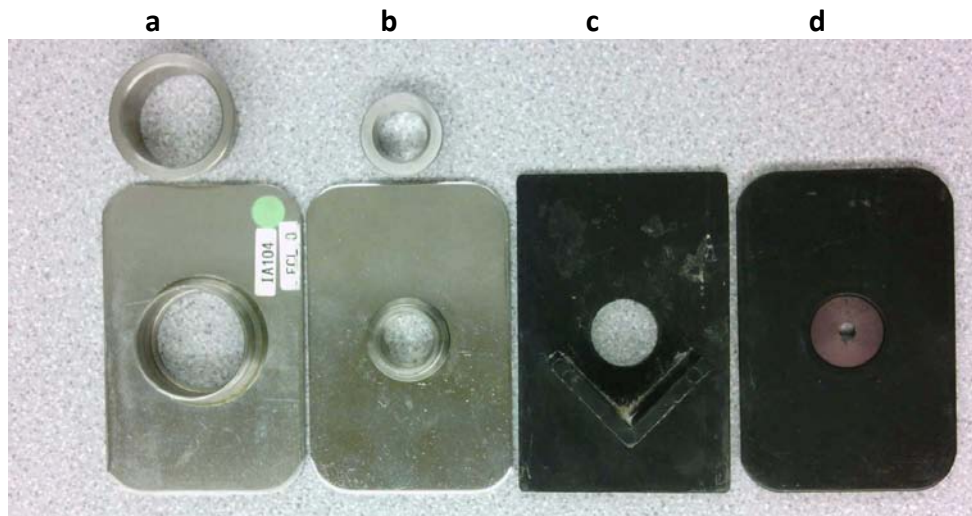
FTIR uses "single-beam" measurements in either transmission or reflectance mode to generate transmittance or absorbance spectra. The simplest measurement requires only two single-beam curves, one for the reference, and one for the sample. In this case, the final transmittance spectrum would just be the ratio (division) of the sample file to the reference file. Since absorbance is the negative log of transmittance, spectra viewed initially as Tr can be converted to the Ab view (and vice versa) without having to reacquire single-beam sample and single-beam reference files.

For critical work, consider acquiring three "single-beam" measurements. The first file would be your reference ratioed to the instrument background (empty channel); the second file would be your sample ratioed to the same instrument background file. The spectral subtraction features of the software would then be used to generate the most correct final spectrum of your sample.

Sample Preparation

The quality of an infrared spectrum is heavily dependent upon sample preparation. There are many possible ways to prepare your sample. This is a choice you must make based upon the nature of your sample and the research question you are trying to answer by acquiring infrared spectra. We offer a number of sample holders you may use once your sample is prepared.

Various Sample Holders



- a. Used to hold 25mm single salt plates or two sandwiched salt plates.
- b. Used to hold 12.5mm KBr pellets
- c. Used to hold salt plates or the student KBr pellet assembly (7.5mm pellets)
- d. Various aperture holders allow smaller samples to be mounted using tape.

NaCl Salt Plates and IR Cards

One of the easiest sample preparation methods involves dissolving a bit of your solid sample in a solvent (other than water or DMSO!) and spotting a drop or two onto a NaCl salt plate. When the solvent evaporates a thin film of your sample will normally be centered on the salt plate. If your sample is already a viscous liquid or paste, just smear a bit on the salt plate and form a thin layer by pressing with a straight-edge or another salt plate. Unpolished NaCl salt plates can be purchased from Chem Stores for \$15-20. We provide a polishing kit in the lab for your use, and also a grinding kit should your salt plates eventually become pitted or wavy.

IR Card and NaCl Salt Plate

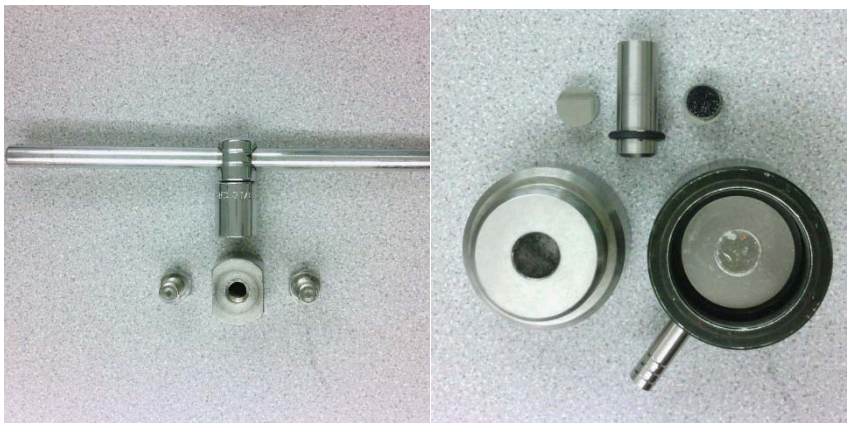


IR cards consist of a thin stretched plastic mounted in a cardboard or metal frame. The plastic is generally either Teflon (C_2F_4 polymer) or polyethylene (C_2H_4 polymer). A drop of solution (or liquid) is placed directly on the plastic; as long as the plastic appears to “wet”, you should get a reasonable spectrum. There will be strong absorption bands from the CH_2 -stretch or the CF_2 -stretch creating a spectral dead-zone in those regions. These cards can be purchased from various on-line sources.

KBr Pellets

A solid-solid mixture of 1% sample and 99% spectral grade KBr can be pressed into a glass-like pellet. The powder mixture is then sandwiched between two steel dies with highly polished surfaces. Pressure is applied to form the pellet. We have a simple die-set available for student use. The pellet formed is small (7.5mm) and cannot be extracted from the die assembly; it is run in-place. Also shown is a more expensive version used to form removable 12.5mm pellets. This is not available for student use.

Routine 7.5mm Student Die set and Research 12.5mm Die Set



A hydraulic jack suitable for use with the more expensive styles of research quality KBr die sets is located on the table in 1240 Hach Hall. You are required to provide your own die set.

Hydraulic Jack and Crystal Polishing Kit Located in the Lab



Liquid Cells

These are not provided. Liquid cells tend to be specific to the project requirements. The proper crystal material and pathlength must be chosen based upon your needs. Generally, one set of liquid cell support hardware can be used with a variety of sealed (fixed pathlength) cells, or demountable cells that allow pathlengths to be adjusted by using different spacers. For aqueous use, demountable “minicells” with AgCl windows are often used.

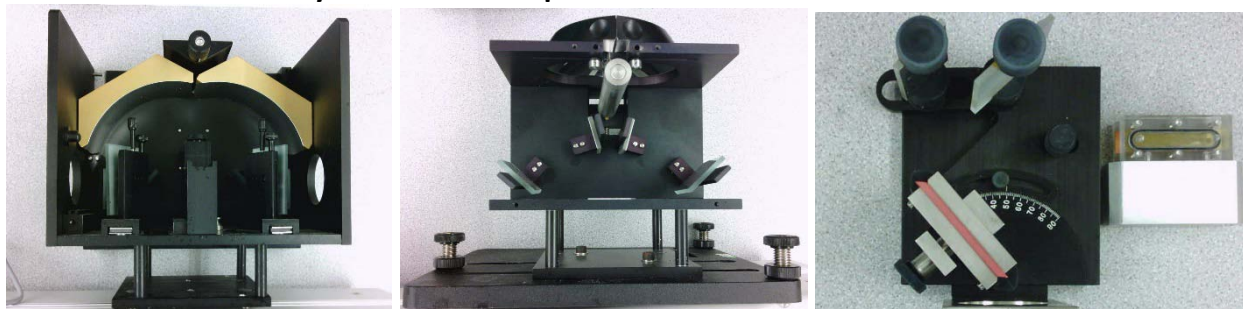
Example of a Fixed Pathlength Liquid Cell



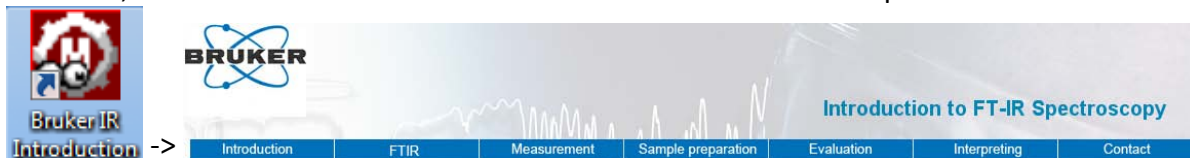
Reflectance Measurements

We have a variety of older diffuse reflectance, specular reflectance, and attenuated total reflectance (ATR) devices previously used with other FTIR's we have had. These can be made to work in the IFS-66V through the addition of the proper baseplate or mounting hardware. This will require advance notice and you will be billed at \$25.60 per hour for the time it takes staff to mount and align the attachment. For the ATR assembly, you must purchase the appropriate crystal. These expensive crystals are easily damaged and are not usually shared.

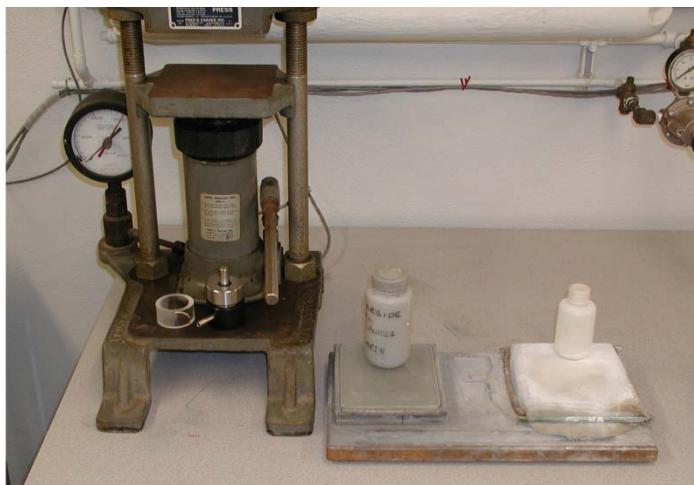
A Variety of Diffuse and Specular Reflectance and ATR Assemblies



For more information about IR spectroscopy, measurement choices, and sample preparation methods, click on the Bruker IR Introduction shortcut on the desktop.



A hydraulic jack suitable for use with KBr pellet presses is located on the table near the FTIR. You are required to provide your own die set.



- A hydraulic jack is available for pressing pellets
- You must provide your own die set
- NaCl salt plates can be easily polished in just a few minutes

Another common sample preparation method used for oils, viscous liquids, pastes, or Nujol mulls, involves smearing a thin layer on or between NaCl salt plates. These salt plates can be obtained from Chemistry Stores but must be polished before use. The polishing procedure is fast and simple, requiring only two or three minutes to complete.

Getting Started

Each registered user has been given a login account on the FTIR instrument control computer. The operating system is Windows XP.

Always log in using your own account, not someone else's, since this is how we track usage for billing purposes. The FTIR software is called "OPUS".



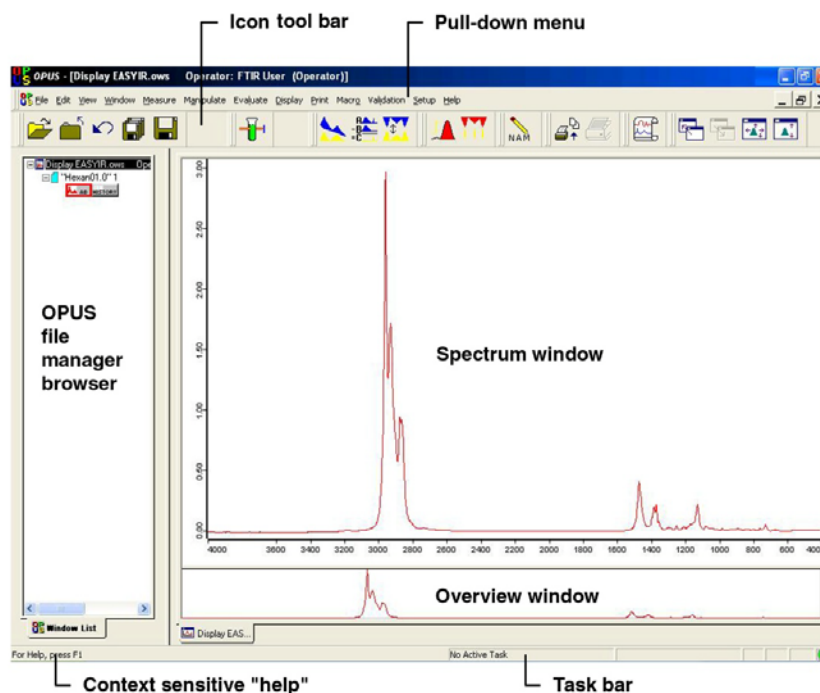
Double click the OPUS icon. located on your desktop. This will bring up the OPUS Login view. You are not assigned an individual account within OPUS. You are a member of the FTIR Users group. The password is "ftir123".

Select :

UserID: <FTIR Users>
Password: <ftir123>
Assigned Workspace: <C:\OPUS\MIDIR.ows>

Press <Login>

A software license page will be displayed. You must click OK to complete the login process. The program will now open to display a number of views and tools.



Routine MIDIR data acquisition and processing can be accomplished using just the icons on the tool bar. Each icon's function can be displayed by placing the mouse over the button.

Acquiring Data

On the IFS Electronics Unit, verify that the "glowbar source" is ON. Turn the "scanner" ON.



Use the <Measure> button to acquire new data. This will open a multi-tab Measurement view. It is only necessary to work with the *Basic* tab. You may also modify some of the elements on the *Advanced* tab. Do not modify any elements on any of the other six tabs! Do not <save> any changes made on the *Advanced* tab!

The screenshot shows the 'Measurement' dialog box with the 'Basic' tab selected. The 'Experiment' dropdown is set to 'MIDIR1.XPM'. The 'Operator Name' is 'FTIR User'. The 'Sample Name' is 'hexane / xylene'. The 'Sample Form' is '0.5mm liquid cell, transmission'. The 'Path' is 'C:\OPUS\MEAS' and the 'Filename' is 'WORK'. There are buttons for 'Background Single Channel' and 'Sample Single Channel'. At the bottom are 'Exit', 'Cancel', and 'Help' buttons.

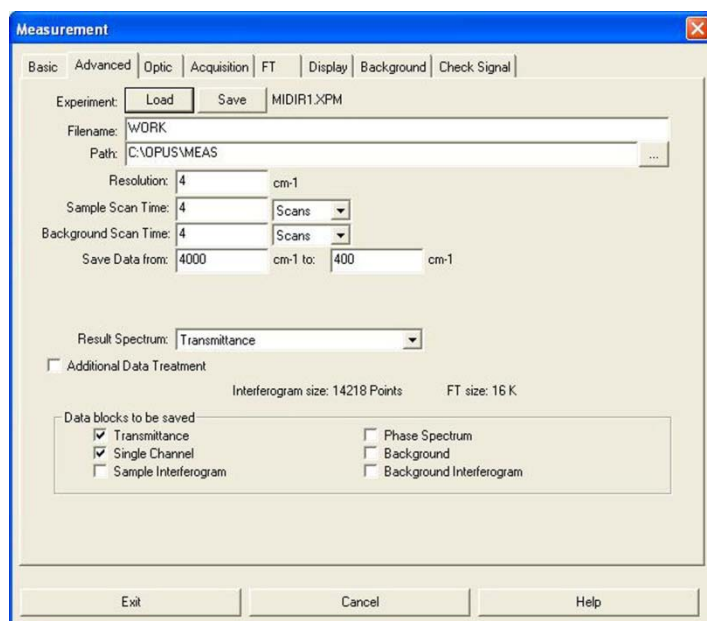
Measurement - Basic Tab

Verify –

Experiment: MIDIR1.XPM
Operator: FTIR User

Modify –

Sample: <type in a descriptive name>
Sample Form: <type in sampling device>



Measurement - Advanced Tab

Modify -

Resolution: 4 cm^{-1} is standard; 0.5 cm^{-1} possible.

Sample Scan Time: 16 scans standard; choose any number.

Background Scan Time: 16 scans standard; choose any number.

Save Data From: 4000 to 400 cm^{-1} (KBr); 4000 to 600 cm^{-1} (NaCl).

During your training you will also be shown how to use the “*Measurement - Check Signal*” tab to verify instrument performance. It is not necessary to do this.

To acquire data, you must be viewing the “*Measurement – Basic*” tab. After placing your reference sample into the purge box, press <Background Single Channel> to acquire the reference file. Acquisition progress is displayed on the Task Bar. If you did not view the “*Check Signal*” tab, there will be a delay of approximately 30 seconds before the first measurement occurs while the instrument re-indexes the position of the optics components. This is normal.

After acquiring the reference, place your sample into the purge box and press <Sample Single Channel> to acquire the sample file. After the file has been acquired, the resultant transmittance spectrum is displayed in both the Spectrum Window and Overview Window of the display.

Processing Data

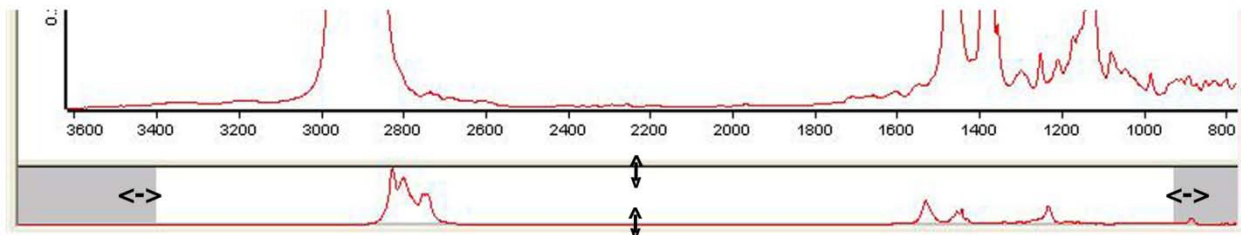
The current spectrum should now be displayed in the Spectrum and Overview windows. You may also retrieve previously acquired data stored on the hard disk or on a floppy by using the <Load>



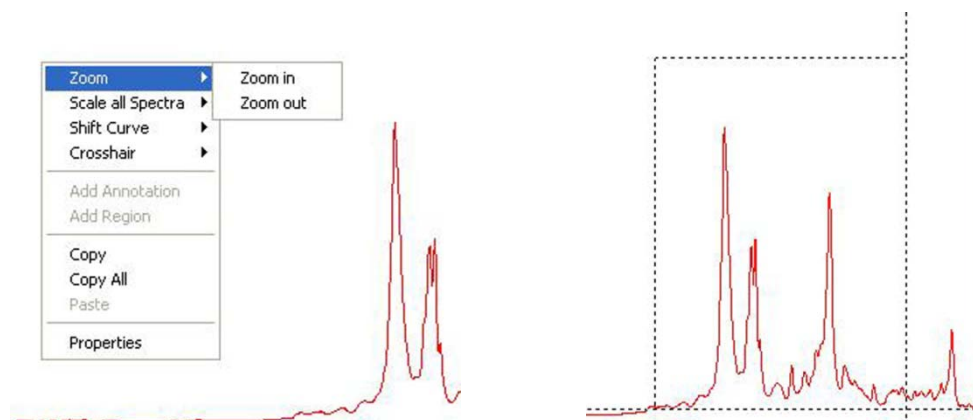
button. Select <view browser> from the View pull-down menu to display the OPUS File Manager Browser frame. You should become familiar with the use of this browser frame if you plan to process previously acquired data, or view more than one data file at a time.

Adjusting the Display

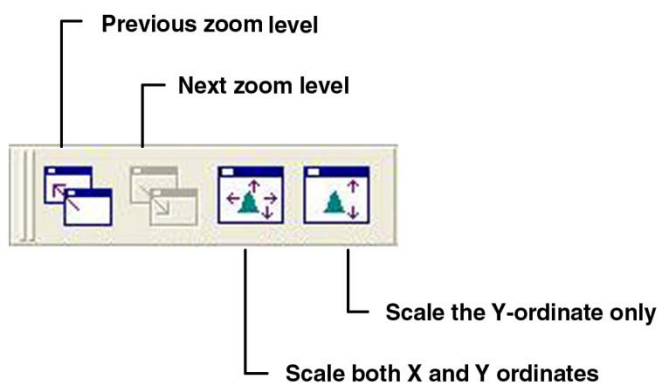
The spectral display-limits may be adjusted from either the Overview window or the Spectrum window. In the Overview window, use the left mouse button to zoom in the X or Y direction by grabbing the edges of the display.



To adjust the display from within the Spectrum window, use the right mouse button to activate the zoom-in or zoom-out crosshairs. While holding the left mouse button, drag to form the desired expansion box. Click the left mouse button twice to create the expanded display.




Four buttons on the Icon tool bar can be used to move between various zoom levels:

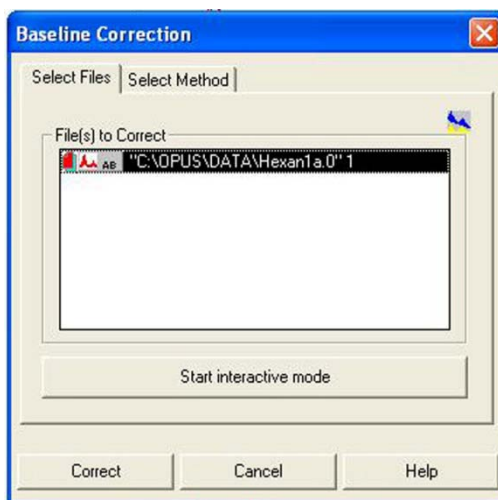


Baseline Correction


Sloped baselines are usually caused by poor sample preparation. Baselines should be corrected

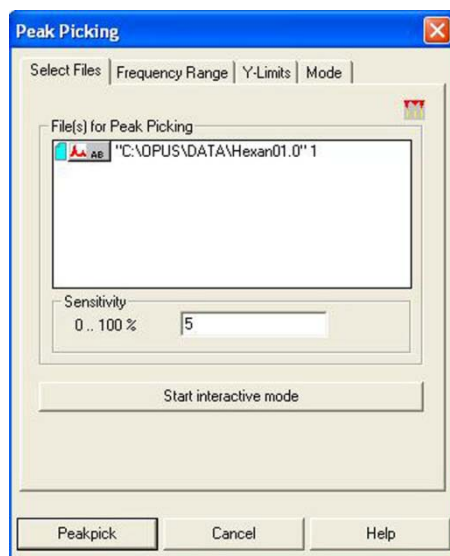
before attempting to “Peak Pick”. Click on  to enter the Baseline Correction view. On the *Select Files* tab, choose the appropriate file. If the wrong file is displayed, use the keyboard “delete” key to remove it from the view; then use your mouse to drag the correct file data block from the browser pane into the view.

On the *Select Method* tab, verify that “the “scattering” method using 64 baseline points has been selected. Click on <Correct> to perform the correction. Do NOT use the “Interactive Mode”.



Peak Picking

Double-click on  to automatically pick and label significant picks in the displayed spectrum. To modify the parameters used, single-click on the icon to enter the Peak Picking view. Do NOT use the Interactive Mode.

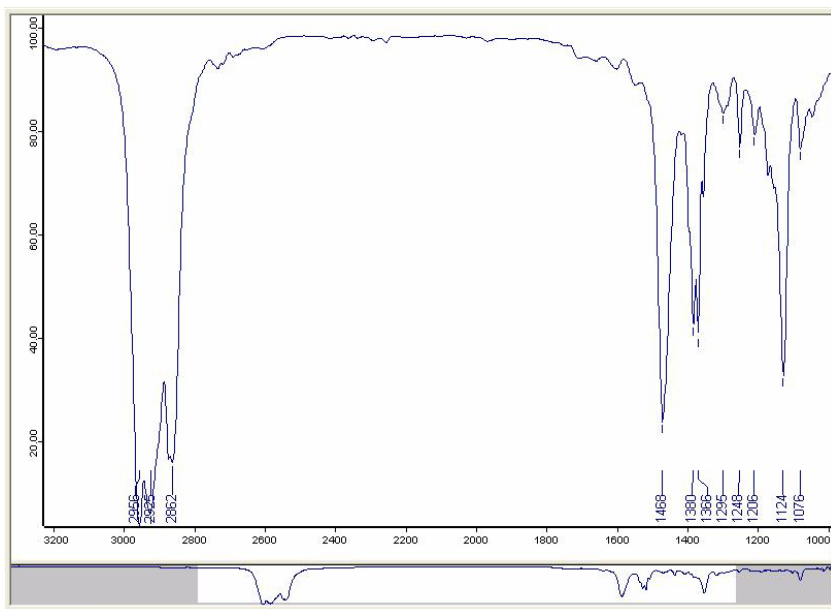


Start with *Sensitivity* set to a 5% threshold. To find more peaks, choose a lower number, to find fewer peaks, choose a higher number. Do NOT use the Interactive Mode feature.

The peak-picking spectral window can be selected in several ways on the “Frequency Range” tab. Do NOT use Interactive Mode.

The “Y-limits” tab can be used to filter which peaks are labeled. Although there may be values set for these filters, we normally we do not activate these filters.

The settings on the Mode tab will normally be “automatic peak detection”, “standard method”, and “overwrite the peak table”. “Append to peak table” can be used under some circumstances to pick one range of the spectrum using one sensitivity, and another range using a different sensitivity.



Spectral Subtraction

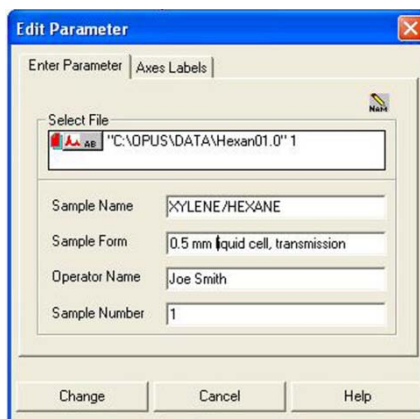


Spectral subtraction is a special topic not covered in this basic guide. Review the OPUS help topics, and request assistance as necessary.

Adding Comments



By clicking the Info icon, it is possible to edit some of the information that will be printed with the spectrum.



Printing the Spectrum



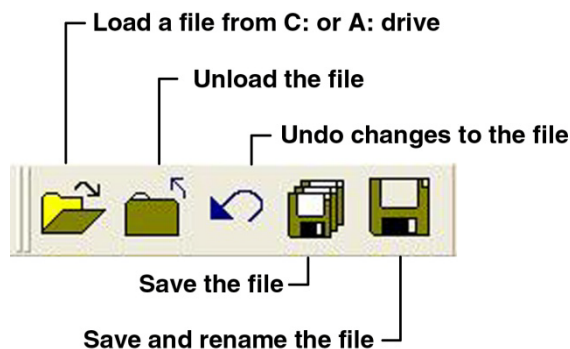
Use the Quick Print icon, to print the spectrum displayed in the Spectral Window. Do NOT print from the File pulldown menu.

Saving Data

All data is instantly saved in the *C: \Opus\Meas* directory with a filename *work.xxxx*, where *xxxx* is an automatically incrementing number. Record the *work.xxxx* number in your notebook. If you modify the file (baseline correction, peak picking, et cetera) the changes are *not* stored automatically. You will be prompted when you exit the program to save or discard the changes.



This can also be accomplished during your session by using the “Save” icon. Do NOT rename the file if it is being stored in the *C:\Opus\Meas* directory. This directory should only contain *work.xxxx* files. I do not usually save modified files; I prefer to keep the raw data.



You have the option of also having space in the *C: \Opus\Data\your_netID* directory. You should only request this if you wish to save files you have modified, or to save files in a format other than OPUS; this is necessary if you wish process data with other software you may have. For example, you may use the “Save As” icon to save the file in “Data Point Table Format” which creates a table of X-Y pairs suitable for graphing in Excel or other plotting software. As part of the process, be sure to select the correct path for saving the file.