Fourier Transform Infrared Spectroscopy (Perkin Elmer - Spectrum One)

This operating procedure intends to provide guidance for transmission/absorbance measurements with the FTIR. For additional modes of operation, i.e. Attenuated Total Reflectance and Diffuse Reflectance measurements, please contact NCC staff members.

Before you start: You must have received formal training from the laboratory manager or, trained graduate student (designated by laboratory manager) related to machine safety and operation.

Safety Precautions: Please use gloves for handling samples.

System Preparation: Log-on computer, the IR stays on at all times, do not turn the IR off.

General Information

FTIR spectrometers record the interaction of IR radiation with a sample, measuring the frequencies at which the sample absorbs the radiation and the intensities of the absorption. Determining these frequencies allows identification of the sample's chemical make-up, since chemical functional groups are known to absorb radiation at specific frequencies. The intensity of the absorption is related to the concentration of the component. Intensity and frequency of sample absorption are depicted in a two- dimensional plot called a spectrum. Intensity is generally reported in terms of percent transmittance, the amount of light that passes through it.

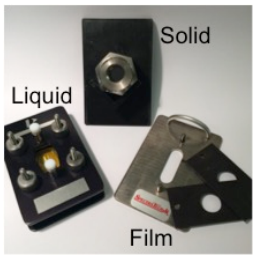
In the interferometer the light passes through a beam splitter, which sends the light in two directions at right angles. One beam goes to a stationary mirror then back to the beam splitter. The other goes to a moving mirror. The motion of the mirror makes the total path length variable versus that taken by the stationary-mirror beam. When the two meet up again at the beam splitter, they recombine, but the difference in path lengths creates constructive and destructive interference pattern called an interferogram.

The recombined beam passes through the sample. The sample absorbs all the different wavelengths characteristic of its spectrum, and this subtracts specific wavelengths from the interferogram. The detector now reports variation in energy versus time for all wavelengths simultaneously. A laser beam is superimposed to provide a reference for the instrument operation.

The Perkin Elmer Spectrum One FTIR Spectrometer is capable of data collection over a wavenumber range of 370-7800 cm^{-1}. The best resolution is 0.5 cm^{-1}. A mid-range Deuterated triglycine sulfate (DTGS) infrared detector processes signals with a 68340 integrated chip.

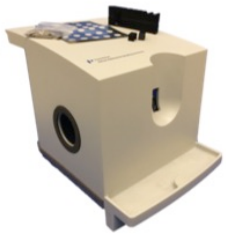
The Perkin Elmer – Spectrum One has the capability of working with liquid, solid and film samples and has the following accessories:

- HATR – A wide range of optional top-plate materials and angles of incidence is available
- Diffuse Reflectance Accessory - Simplifies analysis of powders and difficult solid materials



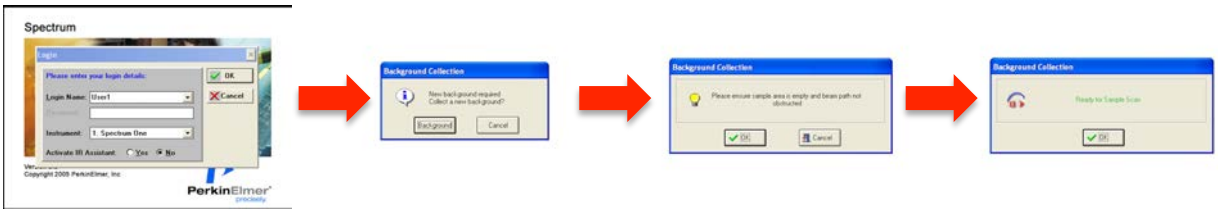
Transmittance/Absorbance Sample Holders


Horizontal Attenuated Total Reflectance (ATR)


Diffuse Reflectance

Starting the System

1. Logon to the computer and start SpectrumOne software
2. Logon to the software as User 1 and make sure the device is selected as SpectrumOne.
3. The software asks to collect the background immediately. Collect the background scan. The system is now ready to run samples.
4. Follow the sample preparation guidelines according to your sample.

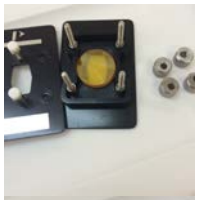


Sample Preparation

Liquid Samples:

Disassemble the liquid sample holder, this holder contains two ZnSe discs (they should be handled very carefully)

Using a Pasteur pipette, place a drop of the sample on one of the ZnSe discs.



Cover the sample with other ZnSe disc, ensure no air bubbles left between the discs (if so, gently rotate the top disc to spread the sample, the air bubbles will move to the sides)

Secure the liquid sample holder with the screws.

Solid Samples (KBr Pellets)

Gather the components for sample preparation:

3 mm die set, handheld KBr pellet press, sample holder, agate mortar and pestle, spatula, KBr.

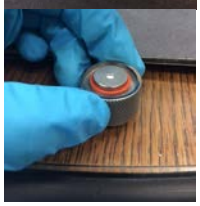
Pulverize the KBr with mortar and pestle (approximately 1 min) until it becomes paste-like.

Mix your sample with the KBr (5-10 % by weight), pulverize for another minute.

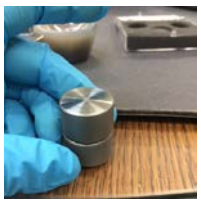
Place 3 mm anvil flat on the table. Stack the rubber gasket over the anvil shaft and 3 mm die on the rubber gasket.

Position the 3 mm die so that the anvil enters the die hole in the center.

Fill the KBr/sample mixture in the opening until half volume has reached. Remove excess material with straight edge or a wipe.



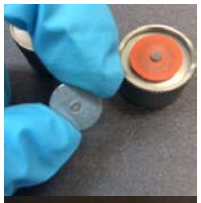
Gently tap the anvil / die set until powder sample spreads on the surface.



Place the top anvil to cover the set.

Carefully transfer the die set to the barrel of the handheld press. Adjust the top plate position on the handheld press until it reaches to the top of the die set.

Squeeze the handle gently and hold for 20 s.



Remove the die set from the press.

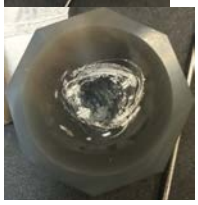
Disassemble the die. The pellet will be formed in the center of the die.

Carefully place the die in the sample holder.

Solid Samples (Nujol paste)

To prepare Nujol paste, use either ZnSe and NaCl discs. (NaCl discs are very fragile and must not come in contact with water)

Grind your sample with mortar and pestle for a minute. PS: The Nujol mineral oil is located in the drawer)



Add 3-4 drops of Nujol mineral oil and mix it with the sample.

Transfer the sample to the NaCl discs, and spread carefully.

Secure the discs in the liquid sample holder.

Sample Measurement

1. Once the background scan is completed. Place the sample holder in the transmission/absorption unit.

2. Go to Scan > Instrument to review measurement parameters.

Sample	Give a file name for the measurement, each measurement will be saved as an .sp file under c:\pel_data\spectra, measurements should be saved as .txt (ascii) file to export the data
Scan	Select range of the measurement, the type of data as transmittance (%T) or absorbance (A), once a measurement is completed it can be converted to other types. The complete spectrum will be presented as an average of number of scans (default is 8). Alternatively, the scan time can be specified.
Instrument	The default resolution of the measurements is 4 cm-1, changing the resolution to other value will require another background scan.
Accessory	The sampling accessory can be specified, such as liquid cell or KBr pellets

Once the scan details are reviewed, click on apply, then scan button to initiate a scan.

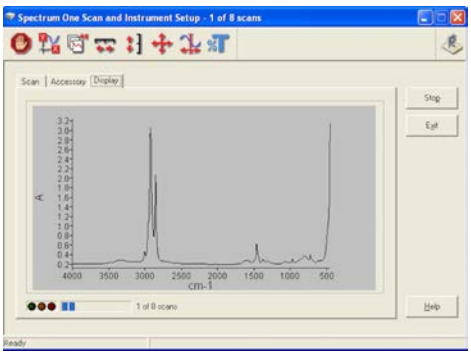


Figure 1. Progress of oleylamine in liquid sample holder scan.

3. When a spectrum is collected, the peaks can be labeled from View > Label Peaks option.

4. Alternatively, the spectrum can be processed for baseline correction (Process > Baseline correction) or the representation can be changed (Process > Transmittance).

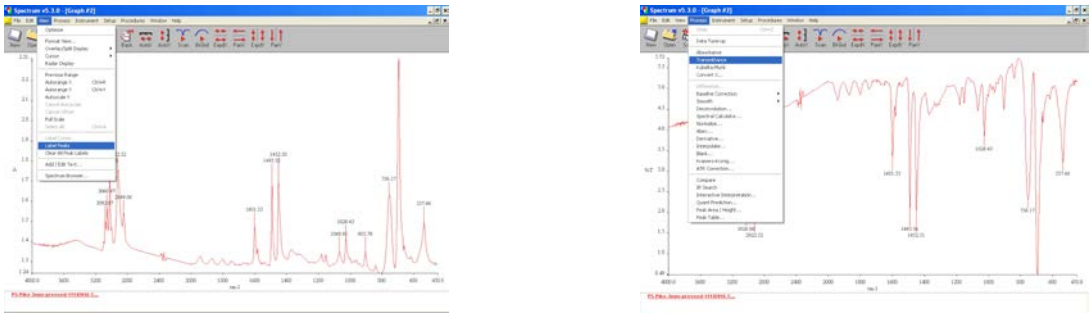


Figure 2. View and data processing options (a) labeling peaks (b) changing the data type to transmittance

5. All spectra collected in this software is saved as an *.sp file, in c:\pel_data\spectra folder. To convert the measurements files to text, please select the measurement and “save as” as an ASCII file to a folder designated with your name.

System Shut Down

1. Remove your samples from the sample holder, clean the sample holders with a suitable solvent and return to the sample holder box.

2. Exit from the software and transfer your data.

4. Log off from the system and record your usage.

Reference:

Butterfield, RC, 2009, *A Novel Laboratory Dispersive and Distributive MiniMixer and Applications*, PhD Thesis, University of Bradford