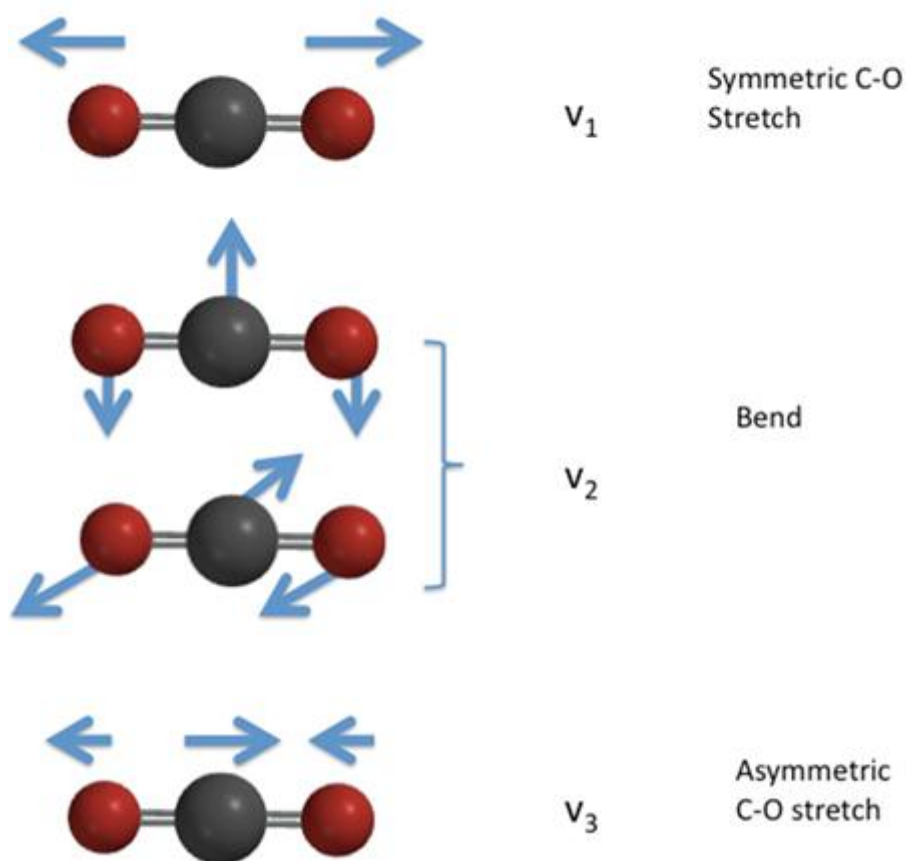


ATR-FTIR

PRESTIGE 21



7/16/2012

Basic Operation and SOP

Created by Jeffery E. Raymond (7/16/2012)

ATR-FTIR Prestige 21

BASIC OPERATION AND SOP

WARNINGS AND NOTES

NEVER...

Turn off the laser power

Use bases or acids ($\text{pH} > 9$ or $\text{pH} < 5$)

Apply pressure to the crucible without monitoring the spectra

Use corrosive liquids or strong chelates

Use volatile solvents without proper ventilation or use of the kinetics cap

Prepare samples at the instrument

Baseline correct your raw data

Use acetone or other spray bottles anywhere near the instrument

Use anything except cotton balls, loose cotton or lens paper to clean the crucible

Take the IR spectra book away from the IR station for more than a couple hours.

BASIC BACKGROUND FOR APPLYING IR SPECTROSCOPY

It is important to know what the inputs and outputs of any analytical instrument are. In this instance, a broad band infrared laser is used to excite vibrational energy bands of symmetric stretches and bends of molecules. The laser is bounced off a diamond crucible which allows for excitation of only the closest ($< 200 \text{ nm}$) parts of the sample in contact with the crucible. The detector includes a monochromator and a power meter at the back end. Therefore the monochromator selects the wavelength and the resolution (via moving slits) and the power meter measures energy of the signal from the laser. Comparing the energy that gets through to the background and initialization files, an absorption/transmission % value can be returned. Presented as a function of the energy of the photons monitored, these values provide a spectra for the vibrational absorbance of your sample.

Typical IR spectra are presented in units of Transmission % v. Wavenumber (cm^{-1}).

A few things to note about the technique are the limitations and strengths it has. The major limitations of the instrument are the laser spectra (very weak below 650 cm^{-1}) and the fact that almost everything has some sort of IR signature (pollution of background from incidentals and solvents). The second is a particular pain when working with water or other centri-symmetric small molecules. In terms of strengths, the technique is fast, easy to use, is sensitive to even small amounts of a functional group, and can be used to generate powerful libraries of intermediates and raw materials.

An analog spectroscopic technique is Raman spectroscopy, which measures asymmetric stretches and bends and does not have a problem with water. However, Raman cannot be applied to emissive samples.

BASIC OPERATION

1. Initial cleaning
 - Disassemble the sample head fully and clean with acetone and cotton.
 - This includes breaking down the Teflon and making sure that the area is wiped down of particulates.
2. Open IRsolution Software and Initialize
 - Measure Tab → Measurement Drop Down → Initialize → Accept deletion of old background
3. Make quality decision (Measure tab→Measure tab)
 - Number of scans dictates background and time/scan
 - Resolution dictates slit size and hence wavenumber resolution and background
 - Range should never go below 650 cm^{-1} as the readings are unreliable below 650 cm^{-1}
4. Confirm that the instrument is in ATR-FTIR collection (Measure tab→Instrument tab)
 - Beam → Internal; Detector → Standard; Mirror Speed → 2.8
5. Take back ground (Measure tab→BKG button)
 - If liquid: Remove retaining ring, remove spacer. Use the same exact solvent for your background. Aqueous samples will always be terrible to the point (often) of telling nothing.
 - If solid surface or powder: Use air as the background.
6. Set up data file - typically to your own folder. (In Measure tab)
7. Take sample (Measure tab→Sample button)
 - if liquid: Remove background material, clean surface, place small amount of sample over diamond, if volatile solvent is used, use the vent arm and/or the cap.
 - if solid: press Monitor button under Measure Tab, place solid sample onto holder and slowly apply pressure with the press. Never go past a pressure of 'snug' - and 'tight' is way out. You should look at the monitor to observe at least a signal of 90% for your highest peak.
8. Clean between samples as per step 1
9. When done, close IRsolution and clean the instrument one last time.

USING LIBRARIES

The library interface can be found under the Search Tab from the main screen. While there are some libraries already in place, with many pure polymer samples for comparison, users can make their own libraries by selecting the NEW button. It suggests that you put your new library in the library folder. If you do this, then the library will show up in the future without searching for it. Make sure to label your library with your initials and a word or two about the project. This will make it clear what you were trying to do with that library to you, others in your subgroup and folks that follow when you leave. The search area then allows you to search through your library by name or by spectral matching for samples that are similar to whatever you just took spectra for. An example might be to put in (polystyrene) and compare the spectra from the library to your block co-polymer. Once you have some spectra in your library, you can rapidly compare a current batch to a previous one – or all previous for the best match. The software does an auto correlation for your sample by comparison of normalized spectra and regional integration, returning a value from 0 to 999, with 999 being a perfect unambiguous match.

DATA ANALYSIS

While in-depth analysis skills are something that an individual must acquire painstakingly over time, there are some resources to get you started. First, you should be starting with a re-read of the IR theory and practice chapters of an analytical chemistry book. Second, you should have done your lit searches and have an idea of how your materials, or one's like them, have been analyzed in the past. Third, having a good book of spectra helps a lot when initially investigating a sample by IR.

In order to do data analysis correctly, you cannot have modified data sets. Things like background correction and multipoint correction are very bad news and should not be used for quantitative studies of any sort. Also, the IRsolution software package, with the exception of the libraries, is a very weak system for analyzing data. When it matters, use Origin or Excel.

For quantitative work, one must be prepared to perform peak de-convolution and very long scans. Peak intensity as a quantitative measure of extent of reaction, percent yield or compositional ratiometrics is never good enough.

VERSION NOTES

This SOP was created by Jeff Raymond on 7/16/2012.

This SOP was revised by --- on ---.