

Lab 3, Fourier Transform Infrared (FTIR) Spectroscopy  
September 22/23, 2008

**Objective:** The goal of this lab is to use Fourier Transform Infrared (FTIR) Spectroscopy to measure the infrared spectrum of several polymer systems. For well-characterized polymers, the spectra will be compared to contrast the vibrations of similar functional groups in different polymers. For less well-defined polymers, the spectra will be used to identify the functional groups that are present.

**Safety:** The main risk in this experiment comes from the acetone and methanol solvents used to clean up after the polymer samples. Acetone vapors are flammable between concentrations of 2.5 and 12.8 vol%; for methanol the limits are 6.0 and 36 vol%. Ingestion of acetone at 5.8 g/(kg of body weight) has been identified as the LD50 for rats (half of them die at that level), and vapors of both are harmful if inhaled or swallowed. Methanol is a poison, and a fatal dose is usually about 100-125 mL. (We will use much less than that!) See MSDS sheets for complete information. A second risk is exposure to the degradation products in the unknown sample. It is unlikely that the instrument will need to be opened. If so, then it is important not to run the instrument during that time. Doing so would lead to exposure to the intense source used to create the infrared light.

*Precautions:*

- Wear “rubber” gloves and safety glasses when preparing and cleaning the sample area.
- Dispose of the used samples in the small waste bottle, which will be indicated.

**Instrument Preservation:** This FTIR instrument relies on infrared absorption into the surface of a solid sample that is pressed against a ZnSe (zinc selenide) crystal. This method is called Attenuated Total Reflectance (ATR). The beam is reflected at the ZnSe/sample boundary, but the amount reflected is decreased (i.e. *attenuated*) by the absorption in the sample near the interface. It is important for the crystal to remain clean and undamaged. Acetone and/or methanol will be used to clean after each sample, as specified below.

### Procedure to perform FTIR Spectroscopy

The FTIR spectrometer is located in the INBRE (IDeA Network of Biomedical Research Excellence) Lab, 222 Fogarty Hall. Dr. Aftab Ahmed, the lab manager, has prepared a visual guide for conducting an FTIR experiment. This Standard Operating Procedure (or SOP) should be followed. The instructions below complement the information in that SOP.

*Prepare the instrument*

1. Clean the sample holder and pressure counter-surface with acetone. Use as little as possible.
2. While the acetone evaporates, study the different experimental parameters that are available in the software.
3. Collect a background spectrum at the beginning of the session. It may be necessary to take a background spectrum before each sample. (This will be determined during the experiment.) Check the C=O frequency range in the background spectrum to confirm the absence of residual acetone or polymer on the surface.

#### *Prepare the sample*

4. The instrument measures the spectrum of a solid that is pressed against the ZnSe crystal by a pressure lever. Place the polymer sample on the surface and rotate the pressure lever to reach the appropriate level (“12” is indicated in the INBRE standard operating procedure). If necessary, grind the sample to create an appropriate size.
5. Follow the INBRE SOP instructions.

#### *Choose the instrument settings and Measure the spectrum*

6. The default instrument parameters can be changed for each spectrum. Some will be modified as described below.
7. See the INBRE SOP for specifics of how to change the settings.
8. See the INBRE SOP for specifics of how to measure the spectrum.

#### *Save the spectrum*

9. Make sure that you save the spectrum after each measurement. They are “auto-saved”, but those are hard to find on the computer.
10. Use the instrument software to help with interpreting the spectrum.

#### *Changing the sample*

11. Rotate the pressure rod to raise it.
12. Remove the polymer and place the sample in the waste bottle.
13. Clean the surface with solvent as directed in the procedure.
14. Prepare the new sample and put it in the instrument, as directed above.
15. Conduct the next experiment as directed above.

#### *Finishing the experiments*

16. Perform a final cleaning of the surfaces.
17. Exit the software, as directed in the INBRE SOP.

### **Experiment 1: Results for a well-characterized polyvinyl acetate sample**

The goal of this experiment is to obtain experimental spectra for a poly(vinyl acetate) sample.

Using the same sample, you will take the spectrum several times by varying parameters such as the number of scans (at a single resolution). The resolution (4, 2, or  $1\text{ cm}^{-1}$ ) would also be interesting to vary, but doing so would take away time that is better spent measuring different polymer samples. BEFORE adding the sample, take a background at the desired resolution. Then prepare the sample as directed above, add it to the instrument, and make the measurements. How do the results compare for the various run-time conditions of number of scans? Clean using methanol.

For one of the resolution/scan combinations, use the software with the instrument to analyze the data. What main peaks are present, and what do they indicate? It may be necessary to perform this step in detail outside of the usual lab period, depending on time constraints.

### **Experiment 2: Results for a well-characterized polymethylmethacrylate sample**

The goal of this experiment is to learn about how the spectrum changes for a polymer with similar functional groups that are arranged in a different way.

Use your top-choice resolution and number of scans from experiment 1. Take a new background (if necessary) and add the PMMA sample. Measure the spectrum. How does it compare to the spectrum for poly(vinyl acetate)? Focus on peaks related to functional groups that are in both polymers, such as  $\text{-O-C=O}$ ,  $\text{CH}_2$ , and  $\text{CH}_3$ . Clean using acetone.

### **Experiment 3: Functioning polyurethane from Mackal Gym**

The goal of this experiment is to learn via the FTIR spectrum about the functional groups present in a polyurethane that “works” as a gym floor.

**Background:** Parts of the Mackal Gym floor are undergoing a degradation process that workers in the field term “reversion” (in the sense that the solid polyurethane is “reverting” to its roots as liquid monomers). In some of the experiments in this course we will evaluate samples taken from functioning and nonfunctioning parts of the floor. The best-case scenario is that we are able to help the athletic department understand more about what is happening to their floor and why.

Measure the IR spectrum for a piece of the non-degraded floor (sample provided). Note if the sample corresponds to the “2003 floor” or “2005 floor”; part of the floor was replaced due to an earlier reversion problem, and both are again showing signs of reversion. Identify the main peaks that are present. Focus on peaks that characterize the functional groups found in urethane polymers. Clean using acetone.

### **Experiment 4: Nonfunctioning polyurethane from Mackal Gym**

Use a spatula to apply a sample of the degraded floor to the spectrometer. Measure the spectrum and repeat the analysis of experiment 3. Which peaks are similar? Which peaks are changed? Which new compounds may be present in the degraded sample? What kinds of chemical degradation pathways could lead to these functional groups being present? Clean using acetone.