

[=>Back To Characterization Lab](#)

## Spectroscopic Analysis of Polymers

**Objective:** The objective of this lab is to become familiar with spectroscopic analytic techniques used in [polymer analysis](#). The most common techniques are IR spectroscopy and proton NMR from polymer solutions. Since the department does not own an NMR spectrometer we will need to make special arrangements for the entire class to observe a sample being run in the chemistry department (**either Doug Bowling or Sathish Sukumaran will do this**). Data from this measurement will be analyzed by the class.

### Materials:

Polymethylmethacrylate (PMMA) of 3 tacticities: "atactic", predominantly isotactic, predominantly syndiotactic. Tetramethylsiloxane (TMS). Benzene.

### Procedure:

- 1) 5% solutions of the PMMA samples with a trace amount of TMS (0.1%) will be used to obtain proton NMR spectra.
- 2) The same samples will be examined using transmission IR.
- 3) Thin films of the solutions will be cast on surfaces for attenuated total reflection IR measurements.
- 4) Thin solid sheets of the PMMA samples will be made by hot pressing small amounts of the samples in a Carver Press. These will be examined using transmission IR.
- 5) Make KBr pellets from ground PMMA samples and examine using Transmission IR.

### Analysis:

- 1) Identify all NMR bands, especially those associated with tacticity in the PMMA samples.
- 2) Identify as many bands as possible in the IR spectra using group contribution methods.
- 3) From the NMR spectra determine the triad tacticity of the PMMA samples.
- 4) From the IR spectra determine the triad tacticity of the PMMA samples.
- 5) Compare the IR spectra obtained by ATRIR, Transmission IR from films, Transmission IR from the solutions, transmission IR from KBr pellets.

### Questions:

- 1) Explain the advantage of NMR over IR in the determination of tacticity and chemical composition.
- 2) Compare the cost and complexity of the two measurements and the flexibility of the sample preparation.
- 3) Why might you want to know the triad tacticity of a polymer?
- 4) What is the group contribution method and why is it advantageous for polymeric systems?
- 5) Spectroscopic techniques generally rely on the presence of a quantized transition which leads to a narrow band of absorption of electromagnetic radiation. What quantized transition exists in IR and NMR. What wavelength of electromagnetic radiation is involved in the two techniques? How do these transitions differ in energy?
- 6) Why is IR data plotted against wavenumber while NMR is plotted against a parameter called the chemical shift? What is the difference between these two parameters and why aren't the spectra plotted against wavelength or frequency?
- 7) Why was PMMA chosen for this experiment?