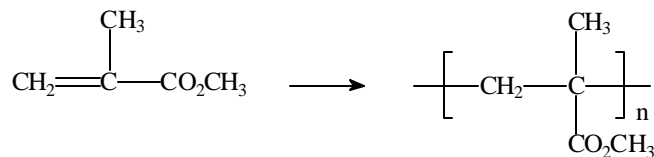
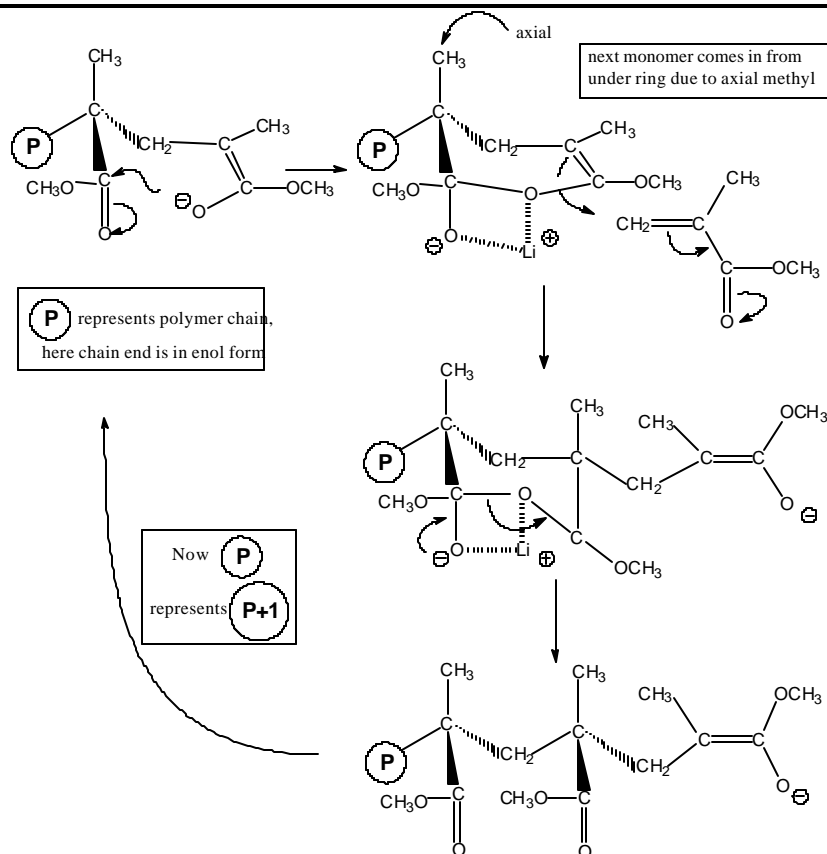


THE PREPARATION OF ISOTACTIC POLY(METHYL METHACRYLATE)



Introduction: Isotactic poly(methyl methacrylate) can be prepared by chain growth polymerization with an anionic initiator. The tacticity results from the polymer chain growing at the end via a six-membered transition state. The chain end is in an enol form and coordination of the counterion (Li^+) of the initiator with oxygen atoms in and attached to the ring facilitates formation of this transition state. Steric effects dictate that the incoming monomer attacks from *under* the ring which gives rise to the observed stereoregularity.

THE MECHANISM FOR FORMATION OF ISOTACTIC POLY(METHYL METHACRYLATE) IN AN ANIONIC POLYMERIZATION

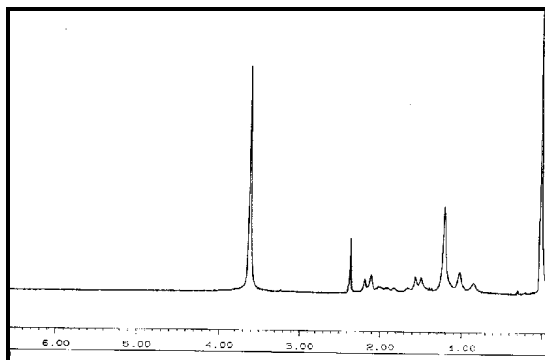


Procedure:

***** ALL OPERATIONS MUST BE CONDUCTED IN A HOOD *****

1. Fit a clean, dry eight inch test tube with a rubber septum (the septum should be attached while the test tube is still hot and a positive nitrogen atmosphere should be maintained in the test tube while the test tube is cooling and throughout the rest of the experiment). While the test tube is cooling, prepare a dry ice-acetone low temperature bath (-78°C).
2. Using a syringe, transfer 30 mL of dry, distilled (from sodium) toluene and 3 mL of distilled methyl methacrylate to the test tube.
3. Cool the test tube in the low temperature bath (NOTE: Be sure that there is a sufficient nitrogen pressure to prevent the bubbler oil from backing up during cooling.) After about five minutes in the low temperature bath, purge the test tube with a nitrogen flow for about 3 minutes (use a small needle to allow the nitrogen to escape from the test tube and pinch off the bubbler to begin the purge). Agitate the test tube periodically during the purging.
4. After the purging, return to a positive nitrogen atmosphere by opening the bubbler line and removing the small needle from the septum (keep the nitrogen needle in the septum). As directed by your instructor, inject 0.1, 0.2, or 0.3 mL of a 1.6 M n-BuLi solution into the test tube.
5. Allow the polymerization to continue for one hour. During this time, remove the test tube at 6 minute intervals. Allow to warm for three minutes then swirl for one minute and return to the bath.
6. After the polymerization is complete, add 5 mL of methanol to the test tube via syringe. Remove the test tube from the low temperature bath and allow it to warm to room temperature.
7. Place 400 mL of diethyl ether in a 600 or 800 mL beaker and equip with a magnetic stirrer. Pour the contents of the test tube into the diethyl ether while it is stirred vigorously.
8. Discontinue stirring, allow the mixture to settle, and decant as much of the diethyl ether as possible. Collect the resultant polymer by suction filtration using a sintered funnel. Allow the polymer to air-dry overnight then continue the drying in a vacuum oven (no heat) for 24 hours.

9. Weigh the dry polymer and store covered on a watch glass or in a beaker for NMR analysis next week.



A 200 MHz ^1H -NMR of isotactic poly(methyl methacrylate). Absorption at 2.4 ppm is residual solvent. (UWSP)