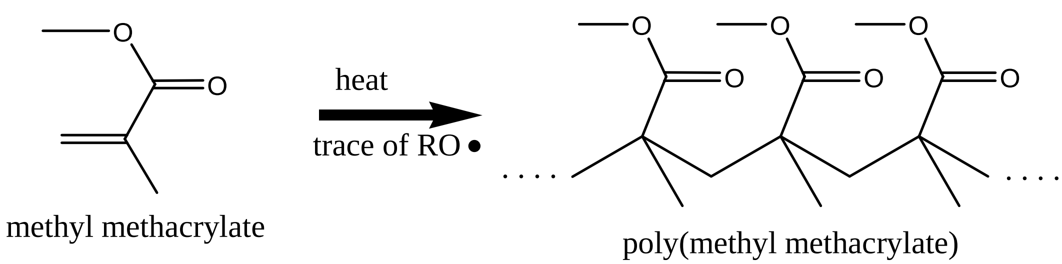
CHEM 321 Synthesis of Two Polymers: Plexiglass and Nylon-6,6

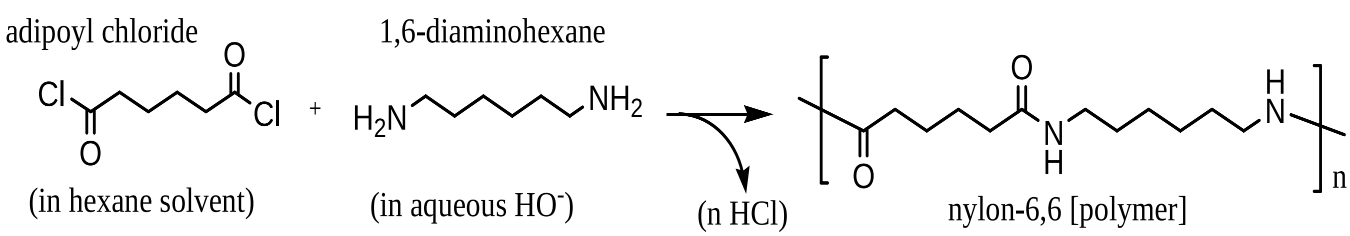
Polymers are large molecules made up of many repeating groups of atoms called subunits. They can be extremely useful materials because they can be made to have an enormous variety of physical and chemical properties. Modern life depends heavily on such materials. Examples of useful polymers include fibers like nylons and polyesters from which clothing, carpets and upholstery can be made; rubbers of various kinds; poly(ethylene) and poly(propylene); plexiglass; foams; adhesives; … The list is endless. Life itself depends on biological polymers like DNA, RNA and proteins.

Certrain simple organic chemicals that possess suitable functional groups (an atom or group of atoms covalently bonded in a specific order that confers unique chemical properties on the molecule) can be easily transformed from liquids into high molecular weight solids. The liquid or liquids are monomer units that can be thought of as building blocks from which the polymer is made. In this experiment, two types of polymers will be formed: poly(methylmethacrylate) — trade name Plexiglass, also known as Lucite or Perspex — and Nylon-6,6.

Poly(methylmethacrylate) is formed by a process known as addition polymerization (a stepwise addition of monomers to a lengthening chain with no loss of atoms in the process). The functional group that permits one monomer to connect with another is the carbon-carbon double bond, which is the distinguishing feature of the class of organic compounds called alkenes. The structures of the monomer and polymer are shown below.



Nylon-6,6 is formed by a process known as condensation polymerization (bifunctional monomers react with each other in such a way that the elements of a small molecule such as water or HCl are eliminated). In the case of nylon, the functional groups that permit the monomers to connect to one another are (a) the acyl chloride functional group (carbon connected to chlorine and double bonded to oxygen in the first monomer) and (b) the amine functional group (nitrogen connected to carbon and two hydrogen atoms in the second monomer). The structures of the monomers and polymer are shown below.



Note: n = repeating units of atoms in [ ].

The objectives of this experiment are to learn how to measure and mix reactive chemicals, arrange for control of reaction temperature, and clean crude products by removing spent reagents.

Pre-lab Your Table of Properties should have entries for methyl methacrylate, benzoyl peroxide, hexane, adipoyl chloride, and 1,6-hexanediamine (including hazards of all of these).

Experimental Protocols

[There is a lot of waiting while methyl methacrylate polymerizes. Work on making nylon after starting this polymerization.]

1. Polymerization of methyl methacrylate

Heat water in an almost full 250 mL beaker covered with a watch glass using a hot plate. Adjust the heating rate to maintain a water bath temperature between 60-70° C (stir to measure). As the beaker heats, obtain a clean, dry, disposable small test tube that is a little taller than the beaker. Find a cork (not made of rubber) that fits the neck. Temporarily store the test tube in a 50 mL Erlenmeyer flask (to hold it upright).

Weigh approximately 0.12 g of benzoyl peroxide on a piece of weighing paper using the method demonstrated in lab— NO METAL SPATULA! (The compound is explosive and metal can trigger the reaction).
Partially fold the boat so as to make a pour spout and gently tap it until all the powder falls into the test tube stored in the Erlenmeyer flask. Using a funnel, pour methyl methacrylate (monomer) from the stock bottle into the test tube until the test tube is just over ½ full. Be careful that the tube does not overflow while using a thin stirring rod to thoroughly mix the contents until all the solid dissolves. Put the cork in loosely (make sure it is NOT sealed) and put the tube back in the Erlenmeyer flask.

Put the test tube into the hot water bath and make sure that the cork is **very loose**! Let the tube incubate for about 20 - 30 minutes. Maintain the temperature the water bath between 60 – 70 °C. If it gets too cool, the polymerization takes forever, and if it gets too hot, lots of bubbles form in the plastic. During this time the polymerization starts, and the solution starts to become viscous toward the end of the incubation time. At this point, objects can be placed into the polymerizing mixture to embed them (*i.e*. paper clip, plant seed or leaf, rocks,…). Replace the cork loosely. Let the tube incubate for another 25 - 35 minutes. The polymerization can be observed by looking for a change in the index of refraction that grows upward from the bottom of the test tube.

Finally, once the plastic is solid, turn off the hot plate and remove the tube from the water bath. Let the test tube with plexiglass cool to room temperature. Wrap it in several layers of paper towel and step on it hard enough to break the glass. **Be careful** not to hurt yourself! Dispose of glass fragments in the box at the front of the lab.

2. Preparation of Nylon-6,6

Caution: Both monomer solutions are skin irritants so handle them carefully and wear goggles. Place 30 mL of adipoyl chloride solution (4% by volume in a solvent of hexanes) in a 250 mL beaker. To a second 250 mL beaker, add 30 mL of hexamethylenediamine solution (IUPAC name 1,6-hexanediamine; 5% by volume in water). Add 1 ml of 10% NaOH solution to the hexamethylenediamine solution and mix well. The purposes of the NaOH are to make sure that all the amine functional groups are unprotonated (electrically uncharged) and to react with protons from the HCl that will be released when the polymerization reaction occurs.

Tilt the beaker containing the hexamethylenediamine at a 45° angle and gently pour all of the adipoyl chloride solution onto the aqueous surface of the hexamethylenediamine. Return the beaker to its upright position. Water and hexanes do not mix. The polymerization takes place at the organic/aqueous interface where the monomers dissolved in each of the liquids meet – in other words, the nylon polymer forms only at the interface between the solutions.

Use a small wire with a hook at the end to gently snag the nylon at the middle of the interface and pull it slowly straight up out of the solution. Drape the end of the nylon over the outside of a 400 mL beaker. Slowly rotate the 400 mL beaker to form a continuous coil of nylon around the beaker until no more nylon is present at the interface. Rinse the nylon coil on the beaker with 40 mL of 50% ethanol/water and then with flowing tap water. Slowly peel the strand of nylon off of the beaker and squeeze dry between two paper towels.

Report Follow “Guidelines for writing the Introduction section” in your lab syllabus. Present the main goals of the experiment in your own words.

Introductions often include pertinent background material whose purpose is to set the stage for the work about to be described. Look up one piece of background information related to the main topics in this protocol. Incorporate your findings into your introduction. Some suggestions:

1) Another protocol to make plexiglass and/or nylon-6,6 [just summarize how the alternate protocol resembles and differs from current protocol]
2) How does modern life depend on polymers? [Pick a polymer, give the structure, and tell how modern society uses the polymer]
3) Contrast addition polymerization with condensation polymerization (you’ll need to consult a text)

Remember to cite the source in American Chemical Society (ACS) format. The direct link on our website which can help with proper citation format is:
http://www.linfield.edu/chem/assets/files/Courses/CHEM%20321/ACSQuickRefGuide.pdf

To cite an internet source, use the following format:
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In addition, answer the following enrichment questions at the end of your report:

1. Circle and name all of the functional groups in all the monomers and in both of the polymers.

2. What could account for the change in physical properties of the products compared to those of the starting materials? (Hint: describe the change in chemical structure and guess how that might affect physical properties.)