Background
In this experiment you will synthesize three polymers: linear polyester, Glyptal polyester, and nylon. Your instructor will demonstrate the synthesis of polystyrene from the monomer styrene. These are all polymers you use in your daily life. Polymers are very large molecules that exist in chains composed of smaller repeating units called monomers. Figure 1 shows polystyrene. The “n” subscript in the figure represents a large number and signifies that the unit in brackets repeats that many times. The “n” can be in the thousands or tens of thousands! Remember that hydrocarbons are chemicals that contain only hydrogen and carbon atoms. You will compare all four polymers in this lab.

![Figure 1: Polystyrene](image)

Both linear and cross-linked polymers will be prepared in this lab. Linear polymers have the monomers repeated in just one line. Cross-linked polymers are branched. Linear polymers have just two ends. Cross-linked have more than two ends.

![linear and cross-linked polymers](image)

Figure 2 shows the synthesis of linear polyester from phthalic anhydride and ethylene glycol.

![Figure 2: Preparation of linear polyester](image)

If more than two functional groups (reactive sites) are present in the monomer, the monomers may connect to each other by cross-linking to form a branched three-dimensional molecule instead of a linear...
molecule. Such polymers are usually more rigid than the linear kind, and are useful in making paints and coatings. Glyptal is prepared using glycerol and is depicted in Figure 3.

![Figure 3: Structure of Glyptal](image)

Reaction of adipoyl chloride with a diamine leads to a linear polyamide by a condensation reaction depicted in Figure 4. A condensation reaction occurs when the monomers join together and release a small molecule as a byproduct. For example let’s say the monomer H-X joins with a second monomer W-OH to make the polymer XWXWXW… What was left over? The H and OH were left over and they make HOH or water as a byproduct.

![Figure 4: Synthesis of nylon 6-6](image)

**Procedure**

Safety: Many of the compounds in this lab have strong odors – use caution when smelling them. Keep all samples away from flames unless told to test small amounts for chemical behavior when heated. Place all discards in the waste bottle in the hood.

**Polystyrene Demonstration** by Instructor – will show you how to weigh using weigh paper. Examine the polystyrene and write your observations on your lab report. You can touch it.
**Polyesters**

You and your partner will make either linear or Glyptal polyester. A pair of students near you will make the other one and you will all share. Your instructor will assign your group the correct one.

Place 1 gram of phthalic anhydride AND 0.05 grams of sodium acetate into a test tube. (Both solids in the one tube) Look at the sample test tube to see a sample of 0.4mL. If you are making the linear polyester, add 0.4mL of ethylene glycol directly to the test tube. If you are making the Glyptal polyester, add 0.4mL of glycerol directly to the test tube. Clamp the test tube so that it points away from you and gently heat it with a Bunsen burner. Heat until the solution gently bubbles, then continue heating for 5 more minutes gently. If the mixture begins to turn amber or black you are heating too much. Let cool for several minutes. Examine the two polymers and write down your observations on your lab report. Put these two test tubes next to the waste jar in the hood. Do not attempt to clean them.

**Nylon – WEAR GLOVES DURING THIS PART**

Pour 10mL of 5% aqueous solution of hexamethylenediamine (1,6-hexanediamine) into a 50 mL beaker. Add 10 drops of 20% sodium hydroxide solution from the dropper bottle. Carefully add 10mL of a 5% solution of adipoyl chloride in cyclohexane to the solution by pouring it down the wall of the slightly tilted beaker (like pouring beer). Two layers will form, and there will be an immediate formation of the polymer film at the liquid-liquid boundary. Do NOT stir. Hook the mass at the center and slowly raise the wire so the polyamide forms continuously, producing a string that can be pulled out for many feet. You can touch the nylon while wearing gloves. The strand will break if you pull too fast. Lay the string on a paper towel to dry. Try again, and again, and pull as much out as possible. Measure all your strings and get a total length in centimeters (cm). If you can get one string to go the length of the bench, I’ll give you extra credit. I must see it to receive the extra credit. When you are all done pulling out strings, with a piece of wire vigorously stir the remainder of the solution to make as much nylon as you can. Pour any remaining liquid into the waste jar. Write all your observations on your lab report. Put your nylon strings in the container for solid waste, not with the liquids.

**Clean up**

Wash your hands. Wipe off your lab bench surface. Make sure all liquids were put into the waste jar. Make sure all solids were put in the solid waste container. **Nothing** but the weigh paper goes in the trash or drain. Make sure you wash your hands before you leave.

**Instructor Directions for Polystyrene**

The glassware is hard to clean, thus only the instructor will prep this polymer. Do this in the hood. Styrene vapor is dangerous. Place 12 mL styrene monomer in a 100mL beaker and add 0.35 g benzoyl peroxide. Heat on a hot plate until yellow (setting 5). When the color disappears and bubbles appear, immediately take the beaker off the heat using tongs. After the reaction subsides, put the beaker back on and heat more until the liquid becomes syrupy. With a stir rod draw out a long filament. If it can be cleanly snapped after a few seconds of cooling, the polystyrene is ready. If the filament does not break, heat more. Pour the polymer on a watch glass that is lightly coated with stopcock grease. After cooling, the polymer can be pried up with a spatula and touched.
I. Observations of the polymers – color, hard or soft, smelly, brittle, bendable, oily, etc.

Linear polyester –

Glyptal -

Nylon -

Polystyrene -

Length of your nylon strings in centimeters: ________________ cm

II. Drawing  Draw the polymer formed from the following monomer. Draw at least 5 monomers in your polymer.

III. Questions:

1. Are these polymers linear or cross-linked?
   a. Linear polyester __________  c. nylon ______________________
   b. Glyptal ________________  d. polystyrene __________________

2. Carefully look at the synthesis of nylon 6-6 in Figure 4. When the monomers join, 2 atoms are left over and make a small molecule byproduct. What are these two atoms? ________________

3. Circle the functional groups in ibuprofen.
1. There are 6 carbons that repeat in nylon 6-6. Clearly number them 1-6 in this image.

\[
\begin{align*}
\text{H} & \quad \text{N} & \quad \text{O} & \quad \text{H} \\
\text{O} & \quad \text{N} & \quad \text{O} & \quad \text{N}
\end{align*}
\]

2. The repeating unit in a polymer is called this: __________________________

3. Polymers often contain carbon and hydrogen. What do we call such compounds? ______________

4. Which one of the polymers made today contains only carbon and hydrogen? ______________

5. Polystyrene is commercially manufactured from petroleum and is one of the most used plastics today. It is used in CD and DVD cases, smoke detector cases, packing foam materials, disposable razors, yogurt containers and foam drink cups. It does not degrade, is largely not recyclable, and is found all over the world on beaches as washed up trash. Give one example of polystyrene in your house - ____________________________________________

6. A most common polyester is PET (polyethylene terephthalate). Polyester threads are woven into fabrics, yarns, cushions, and polyesters fibers are used as filler for pillows and comforters. Polyesters are also used to make "plastic" bottles, films, canoes, liquid crystal displays (LCD’s), holograms, and filters. Give one example of polyester is your house - ____________________________________________

7. Glyptal is largely used in paints, adhesives, varnishes, waxes, resins, and coatings. It was first produced and sold by GE in the 1920’s. Give a possible example of glyptal in your house - ____________________________________________

8. Nylon was first produced in 1935 by DuPont. Nylon is a silky feeling material and was first used in toothbrushes and women’s hose. It was intended to be a replacement for silk that can be expensive. The military uses nylon in vests and parachutes. Nylon is now used in carpets, tires, ropes, airbags, hoses, musical strings and smooth waterproof fabrics. Give one example of nylon in your house - ____________________________________________

9. Label these photos as polystyrene, linear polyester, glyptal, or nylon.

<table>
<thead>
<tr>
<th>Image 1</th>
<th>Image 2</th>
<th>Image 3</th>
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<tbody>
<tr>
<td><img src="image1.png" alt="Polystyrene" /></td>
<td><img src="image2.png" alt="Linear Polyester" /></td>
<td><img src="image3.png" alt="Glyptal" /></td>
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