

ORGANIC POLYMERS: THE SYNTHESIS OF NYLON

Objectives:

- To synthesize Nylon 6,10 from hexamethylene diamine and sebacoyl chloride.
- To determine the length of the Nylon formed

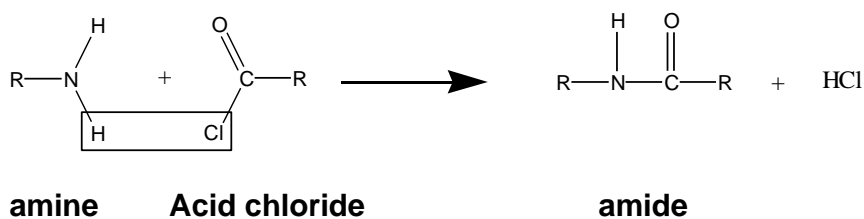
Safety Concerns CAUTION: All chemicals are hazardous to the skin and eyes. Safety goggles and gloves are required during the experiment!!!

WASTE MANAGEMENT: Pour all waste from synthesis and solubility tests into container labeled Polymer waste located in the hood.

Background -

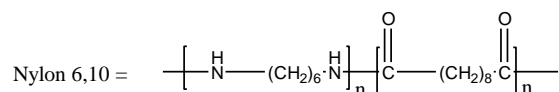
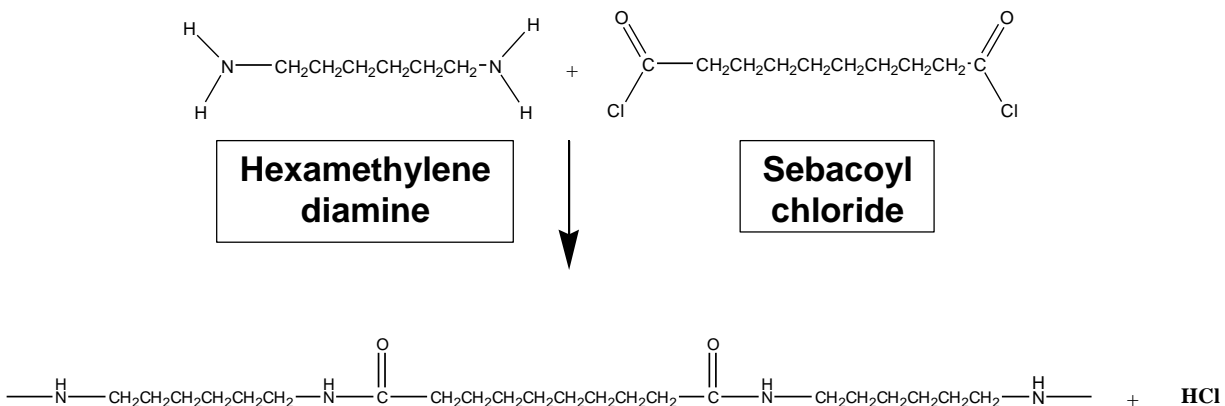
Synthesis of an Amide

By using a carboxylic acid chloride, a more reactive carboxylic acid derivative, the rate of reaction can be increased. In this reaction, HCl is the by-product.



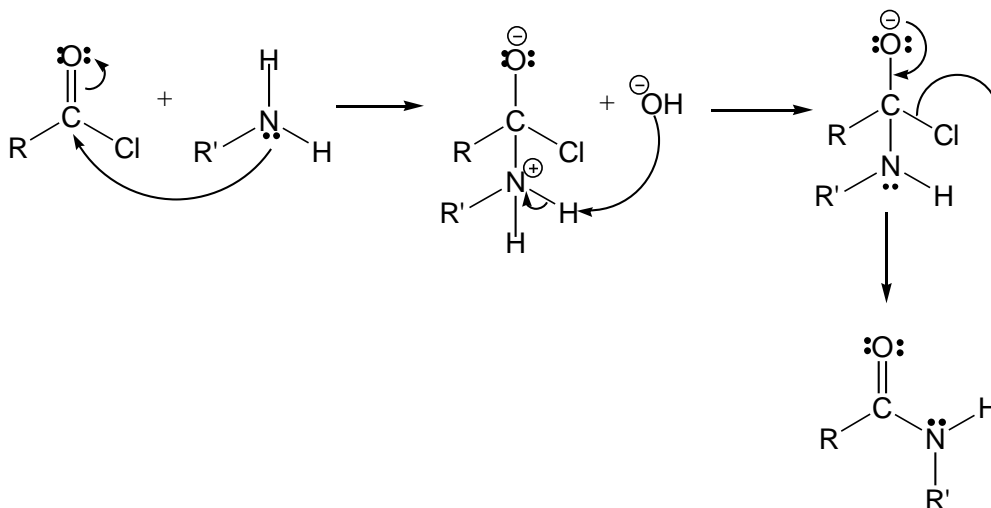
Synthesis of Nylon 6,10

- In order to make a polyamide, such as Nylon 6,10, the amine molecule must have a $-\text{NH}_2$ group at each end, and the acid chloride must have a $-\text{COCl}$ group at each end.
- The diamine and the diacid chloride bond together, end-on-end, to form very long chains.
- Nylon 6,10 is made from hexamethylene diamine (the diamine) and sebacoyl chloride (the diacid chloride).

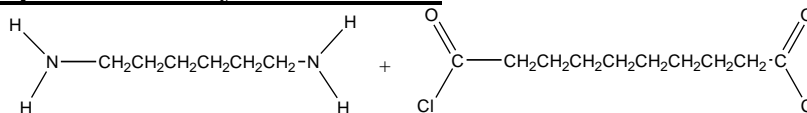


- Since HCl is a by-product of this reaction, we use a diamine solution prepared in an equimolar equivalent of aqueous NaOH. The NaOH reacts with the HCl to form salt and water.
- This prevents the HCl from causing side reactions, such as the possible addition of HCl to a C=C if one is present in the molecule.

Mechanism

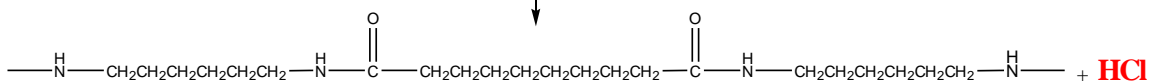


Chemical Equation and Physical Constants



hexamethylenediamine
 MF: C₆H₁₆N₂
 MW: 116.20 g/mol
 d: 0.93 g/mL
 Hazards: CORROSIVE
 SKIN/LUNG IRRITANT

sebacoyl chloride
 MF: C₁₀H₁₆Cl₂O₂
 MW: 239.14 g/mol
 d: 1.121 g/mL
 Hazards: CORROSIVE
 INGESTION HAZARD



Nylon 6,10
 poly(hexamethylene sebacamide)
 MF: C₁₆H₃₂Cl₂N₂O₂
 MW: 331.34 g/mol

Summary: Nylon's outstanding characteristic in the textile industry is its versatility. It can be made strong enough to stand up under the punishment tire cords must endure, fine enough for sheer, high fashion hosiery, and light enough for parachute cloth and backpacker's tents. Nylon is used both alone and in blends with other fibers, where its chief contributions are strength and abrasion resistance. Nylon washes easily, dries quickly, needs little pressing, and holds its shape well since it neither shrinks nor stretches.

Nylon is produced by melt spinning and is available in many different forms. The fiber has outstanding durability and excellent physical properties. Nylons are semi-crystalline polymers. The amide group $(-\text{CO}-\text{NH}-)$ provides hydrogen bonding between polyamide chains, giving nylon high strength at elevated temperatures, toughness at low temperatures, combined with its other properties, such as stiffness, wear and abrasion resistance, low friction coefficient and good chemical resistance. These properties have made nylons the strongest of all synthetic fibers in common use. Because nylons offer good mechanical and thermal properties, they are also a very important engineering thermoplastic. For example, 35% of total nylon produced is used in the automobile industry.

Experimental Procedure- (Synthesis of Nylon)

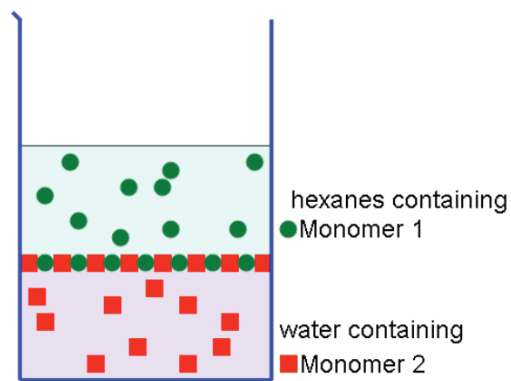
Reagent Preparation (mix in hood):

1. Dissolve 1 mL of the sebacyl chloride in 50 mL of hexane. Label Solution **SC**
2. Dissolve 2.5 mL of 1,6-hexanediamine in 25 mL of 3% sodium hydroxide. Label solution **diamine**.

Nylon Synthesis (read the directions and then try to do the steps)

Length:

1. Take 10 mL of the diamine solution and place in a beaker (50mL, 200mL or 400mL)
2. Carefully and gently pour 20 mL of the SC solution over the diamine solution so as to form two layers. This can be accomplished either by pouring down the side of the beaker or by using a funnel held just above the surface of the other solution.
3. Reach to the interface with tweezers, grasp the polymeric film, and **SLOWLY** withdraw it from the beaker. Using a glass stir rod or a large test tube, **WIND** the nylon rope until one of the reagents is exhausted. **SLOW** pulling will help to avoid incorporating of the solutions into the nylon rope. You can overlap the rope on the glass rod.



4. Using a squirt bottle, wash the product first with water and then with Acetone to speed the drying as well as to remove more of the reagents.
5. Measure the rope when you are done (you may want to go out into the hallway).
6. How many meters of nylon rope did you get from your 30 mL of reagent?

Mass:

1. Pour 5 mL of the diamine solution into a beaker (50mL, 200mL or 400mL)
2. Slowly pour 10 mL of the SC solution into the beaker containing the diamine solution. A white film should form at the interface of the two layers.
3. Reach into the beaker with forceps and grasp the film in the center of the beaker. Slowly pull straight up. Be careful not to let the thread of nylon touch the sides of the beaker. Pull the nylon from the beaker and wrap the thread around a **large test tube**.
4. Rotate the tube, **counting revolutions**, until no more nylon can be obtained.
5. **Record** the number of revolutions of the nylon, along with the circumference and diameter of the test tube used in the data section of your laboratory notebook.

6. Wash the nylon with water and then with acetone to hasten the drying process. Press the washed nylon between paper towels until no more water can be removed.
7. Finally, calculate the length of the nylon string produced in your experiment using the following formula:
Nylon produced (meters) = Diameter of test tube * π * # test tube revolutions
(Where $\pi = 3.14$)
8. After thoroughly drying your nylon, **obtain its mass** (weight it on the balance) and calculate your final % yield.

Nylon Synthesis Data

Part A. Length

Length of Nylon Rope: _____ (meters)

Part B. Mass

Diameter of beaker used for wrapping the nylon: _____ (meters)

Circumference of the beaker ($c = \pi d$): _____ (meters)

Number of turns of nylon around the beaker: _____ (turns)

Length of Nylon Rope: _____ (meters)

Weight of DRY Nylon Rope: _____ (grams)

Sebacyl chloride amount used in grams: _____ (grams)

1,6-hexanediamine amount used in grams: _____ (grams)

Limiting reagent in reaction mixture: _____

Show your work:

Theoretical Yield of Nylon: _____ (grams)

Actual Yield of Nylon: _____ (grams)

Percent yield of Nylon Rope: _____ (%)

Weight of Nylon rope / meter: _____ (grams / meters)