

# POLYMERS

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## Introduction

Natural polymers (from the Greek *poly* meaning “many” and *meros* meaning “parts”) are found in many forms such as horns of animals, tortoise shell, shellac (from the *lac* beetle), rosin (from pine trees), asphalt, and tar from distillation of organic materials. One of the most useful of the natural polymers was rubber, obtained from the sap of the *hevea* tree. (Rubber was named by the chemist Joseph Priestley who found that a piece of solidified latex gum was good for rubbing out pencil marks on paper. In Great Britain, erasers are still called “rubbers”.) Natural rubber had only limited use as it became brittle in the cold and melted when warmed. In 1839, Charles Goodyear discovered, through a lucky accident, that by heating the latex with sulfur, the properties were changed making the rubber more flexible and temperature stable. That process became known as *vulcanization*.

The first synthetic polymer, a phenol-formaldehyde polymer, was introduced under the name “Bakelite”, by Leo Baekeland in 1909. Its original use was to make billiard balls. Rayon, the first synthetic fiber was developed as a replacement for silk in 1911.

Although many polymers were made in the following years, the technology to mass produce them was not developed until World War II, when there was a need to develop synthetic rubber for tires and other wartime applications and nylon for parachutes. Since that time, the polymer industry has grown and diversified into one of the fastest growing industries in the world. Today, polymers are commonly used in thousands of products as plastics, elastomers, coatings, and adhesives. They make up about 80% of the organic chemical industry with products produced at approximately 150 kg of polymers per person annually in the United States.

## RUBBER, A Natural Polymer

Natural latex is found in the inner bark of many trees, especially those found in Brazil and the Far East. The white sticky sap of plants such as milkweed and dandelions is also a latex. Latex will turn into a rubbery mass within 12 hours after it is exposed to the air. The latex protects the tree or plant by covering the wound with a rubbery material like a bandage.

Natural rubber is a polymer of isoprene (2-methyl-1,3-butadiene, see Figure 1) in the form of folded polymeric chains which are joined in a network structure and have a high degree of flexibility (See Figure 2). Upon application of a stress to a rubber material, such as blowing up a balloon or stretching a rubber band, the polymer chain, which is randomly oriented, undergoes bond rotations allowing the chain to be extended or elongated (See Figure 3). The fact that the chains are joined in a network allows for elastomeric recoverability since the cross-linked chains cannot irreversibly slide over one another. The changes in arrangement are not constrained by chain rigidity due to crystallization or high viscosity due to a glassy state.

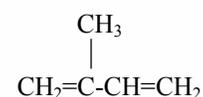


Figure 1. 2-methyl-1,3-butadiene

Since latex will solidify in air, a stabilizer is added to prevent polymerization if the latex is to be stored or shipped in liquid form. The stabilizer is usually 0.5 to 1% ammonia. When the ammonia is removed by evaporation or by neutralization, the latex will solidify into rubber.

Polymer name	Monomer(s)	Polymer	Use
Polyethylene	$\text{CH}_2=\text{CH}_2$ (ethene)	$-\text{CH}_2-\text{CH}_2-$	Most common polymer. Used in bags, wire insulation, and squeeze bottles
Polypropylene	$\text{CH}_2=\text{CH}$ $\quad  $ $\quad \text{CH}_3$ (1-propene)	$-\text{CH}_2-\text{CH}-$ $\quad  $ $\quad \text{CH}_3$	Fibers, indoor-outdoor carpets, bottles
Polystyrene	$\text{CH}_2=\text{CH}$ $\quad  $  (styrene)	$-\text{CH}_2-\text{CH}-$ $\quad  $ 	Styrofoam, molded objects such as tableware (forks, knives and spoons), trays, videocassette cases.
Poly(vinyl chloride) (PVC)	$\text{CH}_2=\text{CH}$ $\quad  $ $\quad \text{Cl}$ (vinyl chloride)	$-\text{CH}_2-\text{CH}-$ $\quad  $ $\quad \text{Cl}$	Clear food wrap, bottles, floor covering, synthetic leather, water and drain pipe
Polytetrafluoroethylene (Teflon)	$\text{CF}_2=\text{CF}_2$ (tetrafluoroethene)	$-\text{CF}_2-\text{CF}_2-$	Nonstick surfaces, plumbing tape, chemical resistant containers and films
Poly(methyl methacrylate) (Lucite, Plexiglas)	$\text{CO}_2\text{CH}_3$ $\quad  $ $\text{CH}_2=\text{C}$ $\quad  $ $\quad \text{CH}_3$ (methyl methacrylate)	$\text{CO}_2\text{CH}_3$ $\quad  $ $-\text{CH}_2-\text{C}-$ $\quad  $ $\quad \text{CH}_3$	Glass replacement, paints, and household products
Polyacrylonitrile (Acrilan, Orlon, Creslan)	$\text{CH}_2=\text{CH}$ $\quad  $ $\quad \text{CN}$ (acrylonitrile)	$-\text{CH}_2-\text{CH}-$ $\quad  $ $\quad \text{CN}$	Fibers used in knit shirts, sweaters, blankets, and carpets
Poly(vinyl acetate) (PVA)	$\text{CH}_2=\text{CH}$ $\quad  $ $\quad \text{OOCCH}_3$ (vinyl acetate)	$-\text{CH}_2-\text{CH}-$ $\quad  $ $\quad \text{OOCCH}_3$	Adhesives (Elmer's glue), paints, textile coatings, and chewing gum
Natural rubber	$\text{CH}_3$ $\quad  $ $\text{CH}_2=\text{C}-\text{CH}=\text{CH}_2$ (2-methyl-1,3-butadiene)	$\text{CH}_3$ $\quad  $ $-\text{CH}_2-\text{C}=\text{CH}-\text{CH}_2-$	Rubber bands, gloves, tires, conveyor belts, and household materials
Polychloroprene (neoprene rubber)	$\text{Cl}$ $\quad  $ $\text{CH}_2=\text{C}-\text{CH}=\text{CH}_2$ (2-methyl-1,3-butadiene)	$\text{Cl}$ $\quad  $ $-\text{CH}_2-\text{C}=\text{CH}-\text{CH}_2-$	Oil and gasoline resistant rubber
Styrene butadiene rubber (SBR)	$\text{CH}_2=\text{CH}$ $\quad  $  $\text{CH}_2=\text{CH}-\text{CH}=\text{CH}_2$	$-\text{CH}_2-\text{CH}-\text{CH}_2-\text{CH}-\text{CH}-\text{CH}_2-$ 	Non-bounce rubber used in tires

Table 1. Addition Polymers



Figure 2. Schematic sketch of a typical elastomeric network.

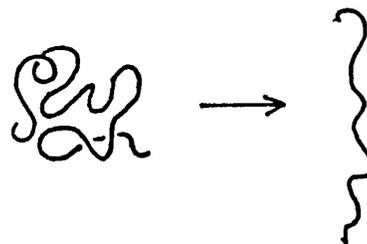


Figure 3. Elastic deformation of a rubber chain.

## Make a Rubber Ball from Rubber Latex

### Safety Precautions

Wear safety goggles at all times in the laboratory.

When noting the odor of the rubber latex, cup your hand and waft the vapors toward you. Do not take a deep breath of the material.

Make sure the rubber ball is submerged under water when squeezing out the excess liquid to prevent it squirting onto anyone.

### Materials needed

Rubber latex  
Vinegar  
Stirring rod  
Paper cup  
Measuring devices

### Procedure

Measure 20 mL of rubber latex into a paper cup.

Carefully, smell the material. Describe its odor.

Pour a few drops of latex onto the palm of your hand. Spread it out using your finger. Observe any changes that take place.

Add 20 ml of water to the latex in the cup and stir.

Add 20 mL of vinegar to the cup and stir the mixture.

Remove the mass from the cup and stirring rod with your fingers. Carefully squeeze the mass while washing it *underwater*. Form the mass into a ball by repeatedly rotating it and squeezing it.

Dry the rubber with a paper towel.

Drop the rubber on the floor and describe what happens.

Polymer name	Monomers	Polymer	Use
Polyamides (nylon)	$\begin{array}{c} \text{O} \quad \text{O} \\ \parallel \quad \parallel \\ \text{HO}(\text{C})_n(\text{CH}_2)_n(\text{C})_n\text{OH} \\ \text{H}_2\text{N}(\text{CH}_2)_n\text{NH}_2 \end{array}$	$-\text{C}(\text{CH}_2)_n\text{C}-\text{NH}(\text{CH}_2)_n\text{NH}-$	Fibers, molded objects
Polyesters (Dacron, Mylar, Fortrel)	$\begin{array}{c} \text{O} \quad \text{O} \\ \parallel \quad \parallel \\ \text{HO}(\text{C})_6(\text{C})_6\text{OH} \\ \text{HO}(\text{CH}_2)_n\text{OH} \end{array}$	$-\text{C}(\text{C}_6\text{H}_4)_2\text{C}-\text{O}(\text{CH}_2)_n\text{O}-$	Linear polyesters, fibers, recording tape
Polyesters (Glyptal resin)	$\begin{array}{c} \text{O} \quad \text{O} \\ \parallel \quad \parallel \\ \text{C} \quad \text{C} \\ \diagdown \quad \diagup \\ \text{C}_6\text{H}_4 \quad \text{C}_6\text{H}_4 \\ \diagup \quad \diagdown \\ \text{C} \quad \text{C} \\ \parallel \quad \parallel \\ \text{HOCH}_2\text{CH}(\text{OH})\text{CH}_2\text{OH} \end{array}$	$\begin{array}{c} \text{O} \\ \parallel \\ \text{C} \\ \diagdown \quad \diagup \\ \text{C}_6\text{H}_4 \quad \text{C}_6\text{H}_4 \\ \diagup \quad \diagdown \\ \text{C} \quad \text{C} \\ \parallel \quad \parallel \\ \text{COCH}_2\text{CH}(\text{O})\text{CH}_2\text{O}- \end{array}$	Cross-linked polyester, paints
Polyesters (Casting resin)	$\begin{array}{c} \text{O} \quad \text{O} \\ \parallel \quad \parallel \\ \text{HO}(\text{C})=\text{C}(\text{C})=\text{C}(\text{C})\text{OH} \\ \text{HO}(\text{CH}_2)_n\text{OH} \end{array}$	$-\text{C}(\text{C})=\text{C}(\text{C})-\text{O}(\text{CH}_2)_n\text{O}-$	Cross-linked with styrene and benzoyl peroxide, fiberglass boat resin, casting resin
Phenol-formaldehyde (Bakelite)	$\begin{array}{c} \text{OH} \\   \\ \text{C}_6\text{H}_5 \\ \text{CH}_2=\text{O} \end{array}$	$\begin{array}{c} \text{OH} \quad \text{OH} \\   \quad   \\ \text{CH}_2-\text{C}_6\text{H}_3-\text{CH}_2-\text{C}_6\text{H}_3-\text{CH}_2 \\   \quad   \\ \text{CH}_2 \quad \text{CH}_2 \end{array}$	Mixed with fillers, molded electrical cases, adhesives, laminates, varnishes
Cellulose acetate (cellulose is a polymer of glucose)	$\begin{array}{c} \text{CH}_2\text{OH} \\   \\ \text{C}_6\text{H}_9\text{O}_5 \\   \\ \text{OH} \\   \\ \text{OH} \\   \\ \text{CH}_3\text{COOH} \end{array}$	$\begin{array}{c} \text{CH}_2\text{OAc} \\   \\ \text{C}_6\text{H}_9\text{O}_5 \\   \\ \text{OAc} \\   \\ \text{OAc} \end{array}$	Photographic film
Silicones	$\begin{array}{c} \text{CH}_3 \\   \\ \text{Cl}-\text{Si}-\text{Cl} \quad \text{H}_2\text{O} \\   \\ \text{CH}_3 \end{array}$	$\begin{array}{c} \text{CH}_3 \\   \\ -\text{O}-\text{Si}-\text{O}- \\   \\ \text{CH}_3 \end{array}$	Water-repellent coatings, temperature-resistant fluids and rubber
Polyurethanes	$\begin{array}{c} \text{CH}_3 \\   \\ \text{C}_6\text{H}_4-\text{N}=\text{C}=\text{O} \\   \\ \text{N}=\text{C}=\text{O} \\   \\ \text{HO}(\text{CH}_2)_n\text{OH} \end{array}$	$\begin{array}{c} \text{CH}_3 \\   \\ \text{C}_6\text{H}_4-\text{NHC}(\text{O})-\text{O}(\text{CH}_2)_n\text{O}- \\   \\ \text{NHC}(\text{O})-\text{O}(\text{CH}_2)_n\text{O}- \\   \\ \text{O} \end{array}$	Foams, rigid and flexible, fibers

Table 2. Condensation Polymers

## BAKELITE, The First Synthetic Polymer

Bakelite, a phenol-formaldehyde polymer, was the first completely synthetic plastic, first made by Leo Baekeland in 1907. Baekeland and an assistant started their research in 1904 looking for a synthetic substitute for shellac. Bakelite was commercially introduced in 1909. Bakelite was first used to make billiard balls, but, later, was used to make molded insulation, valve parts, knobs, buttons, knife handles, many types of molded plastic containers for radios and electronic instruments, and more.

### Safety Precautions

Wear safety goggles at all times in the laboratory.

Formalin is an irritant to the skin, eyes, and mucous membranes.

Phenol is toxic via skin contact. It is listed as a carcinogen.

Glacial acetic acid is an irritant and can cause burns on contact.

Work under a hood and wear gloves and protective clothing when working with these materials.

### Materials needed

Phenol-formaldehyde reaction mixture (freshly prepared solution should be available. The reaction mixture is made by mixing 25 g 36-40% formaldehyde + 20 g phenol + 55 mL glacial acetic acid.)

Hydrochloric acid, HCl, concentrated

150-mL beaker

stirring rod

### Procedure

Under a hood, measure 25 mL of the phenol-formaldehyde reaction mixture into a 150-mL beaker. Place the beaker on a white paper towel.

Add 10 mL of concentrated hydrochloric acid, *slowly*, with stirring.

Add additional hydrochloric acid, dropwise, with stirring. (You will need approximately 2 mL of HCl.) As the polymerization point is reached, a white precipitate will form and dissolve. At the point where polymerization begins, the white precipitate will not dissolve.

Continue to stir as the plastic forms and becomes pink in color.

Wash the plastic well before handling.

Describe the properties of the plastic.

## RAYON, The First Synthetic Fiber

What may be considered as the first precursor to Rayon, Louis Marie Hilaire Bernigaut, Comte de Chardonnet worked on a process of producing threads of an "artificial silk" made from collodion in 1884. Chardonnet's silk was finally marketed in 1891, but was short-lived after a young lady's ball gown when up in a puff of smoke after being touched by the lighted cigar of her escort. Charles F. Cross and Edward J. Bevan patented their formula for viscose, a cellulose polymer, in 1892 and were soon manufacturing items of their new Viscoid. The first viscose thread was made by Charles Topham Jr. and commercially produced in 1899. Rayon, made from regenerated cellulose in 1926, marketed by Du Pont, was finally used as a replacement for silk.

Recycling code	Polymer and structure	Uses
 <b>PETE</b>	$-\text{O}-\text{CH}_2-\text{CH}_2-\text{O}-\text{C}(=\text{O})-\text{C}_6\text{H}_4-\text{C}(=\text{O})-$ Poly(ethylene terephthalate) (PET)	Bottles for soft drinks and other beverages
 <b>HDPE</b>	$-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-$ High-density polyethylene	Containers for milk and other beverages, squeeze bottles
 <b>V</b>	$-\text{CH}_2-\underset{\text{Cl}}{\text{CH}}-\text{CH}_2-\underset{\text{Cl}}{\text{CH}}-$ Vinyl/poly(vinyl chloride)	Bottles for cleaning materials, some shampoo bottles
 <b>LDPE</b>	$-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-$ Low-density polyethylene May have some branches	Plastic bags, some plastic wraps
 <b>PP</b>	$-\text{CH}_2-\underset{\text{CH}_3}{\text{CH}}-\text{CH}_2-\underset{\text{CH}_3}{\text{CH}}-$ Polypropylene	Heavy duty microwaveable containers
 <b>PS</b>	$-\text{CH}_2-\underset{\text{C}_6\text{H}_5}{\text{CH}}-\text{CH}_2-\underset{\text{C}_6\text{H}_5}{\text{CH}}-$ Polystyrene	Beverage/foam cups, toys, window in envelopes
 <b>Other</b>	All other resins, layered multimaterials, some containers	Some ketchup bottles, snack packs, mixture where top differs from bottom.

Table 3. Recycling codes for plastics

### **Safety Precautions**

Wear safety goggles at all times in the laboratory.

Ammonia fumes are irritating eyes, and mucous membranes. Work in a well ventilated area.

### **Materials needed**

Copper(II) sulfate pentahydrate,  $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$   
Ammonia,  $\text{NH}_3$ , concentrated  
Sulfuric acid,  $\text{H}_2\text{SO}_4$ , 5% solution  
Filter paper  
Funnel  
Plastic dropper  
Stirring rod

### **Procedure**

Dissolve 5.0 g of copper(II) sulfate pentahydrate in 25 mL of deionized water.

Working under a hood, add concentrated ammonia solution dropwise, with stirring, to form a light blue-green copper(II) hydroxide,  $\text{Cu}(\text{OH})_2$ , precipitate persists. Allow the precipitate to settle for a few minutes. The remaining liquid should be light blue or colorless. If necessary, add an additional drop or two of the ammonia solution to check for completeness of precipitation of the copper(II) sulfate. Avoid an excess of ammonia.

Collect the precipitate by gravity filtration. Wash the solid with two 5 mL portions of cold water.

Transfer the precipitate to a 150-mL beaker. The filtrate can be discarded.

Under a hood, add 25 mL of concentrated ammonia solution to the beaker. Stir to dissolve the solid.

Obtain two pieces of filter paper. Tear them into pieces and add them to the dark blue copper-ammonia solution. Stir to dissolve the filter paper.

Obtain 100 mL of 5% sulfuric acid in a 250-mL beaker.

Fill a plastic dropper with the blue cellulose-copper-ammonia solution. Place the tip of the dropper below the surface of the 5% sulfuric acid solution and squirt its contents into the solution. Dark blue threads will form which will slowly bleach to white. Repeat with the rest of the cellulose-copper-ammonia solution.

Wash the rayon well with water before handling.

Record your observations of the rayon.

## **POLYSTYRENE, An Addition Polymer**

Polystyrene is used to make many types of containers such as videocassette cases, compact disk jewel boxes, tableware (forks, knives and spoons), and cafeteria trays. A foamed form of polystyrene is used to make coffee cups, grocery store meat trays, and building insulation.

To minimize fumes in the laboratory, this procedure uses a styrene casting resin.

### **Safety Precautions**

Wear safety goggles at all times in the laboratory.

Styrene is harmful by inhalation. It is an irritant to the eyes and respiratory system. Work in a well ventilated area.

Methyl ethyl ketone is an irritant. Work in a well ventilated area.

### **Materials needed**

Styrene casting resin  
Casting resin catalyst, methyl ethyl ketone  
Half of a small petri dish  
Stirring rod  
Optional: A clean dry coin, such as a penny

### **Procedure**

Measure 15 mL of styrene casting resin into a paper cup.

Add 5 drops of catalyst to the resin. Stir well.

Pour the resin mixture into a clean half of a petri dish. Allow the mixture to sit, undisturbed, for approximately one hour or until hard.

If desired, a clean, dry penny can be added to the resin after it has set for about 5 minutes. To avoid air bubbles, coat the penny with a small amount of the mixed resin before adding it to the resin mixture in the petri dish.

## **NYLON, A Condensation Polymer**

Nylon was the result of research directed by Wallace Hume Carothers at du Pont. The research team was interested in duplicating the characteristics of silk. Nylon gained rapid acceptance for use in stockings and in making parachutes. Carothers, however, was subject to bouts of depression and in 1937, shortly before du Pont placed nylon stockings on the market, Carothers committed suicide by drinking cyanide.

### **Safety Precautions**

Wear safety goggles at all times in the laboratory.

The materials in this experiment are considered toxic. They are irritants to the eyes and mucous membranes. Wear gloves and work in a well ventilated area.

### **Materials needed**

Hexamethylenediamine (1,6-hexanediamine), 5% aqueous solution  
Sebacoyl chloride (or adipyl chloride), 5% solution in cyclohexane  
Sodium hydroxide, NaOH, 20% aqueous solution

Beaker, 50 mL  
 Forceps  
 Stirring rod.

### Procedure

Pour 10 mL of hexamethylenediamine solution into a 50 mL beaker.

Add 10 drops of 20% sodium hydroxide solution. Stir.

Carefully add 10 mL of sebacoyl chloride solution by pouring it down the wall of the tilted beaker. Two layers should be evident in the beaker and there should be an immediate formation of a polymer film at the interface between the two liquids.

Using forceps, pick up the mass at the center and slowly draw out the nylon, allowing a “rope” to be formed. Use a stirring rod to wrap the rope and continue to pull it slowly from the mixture interface.

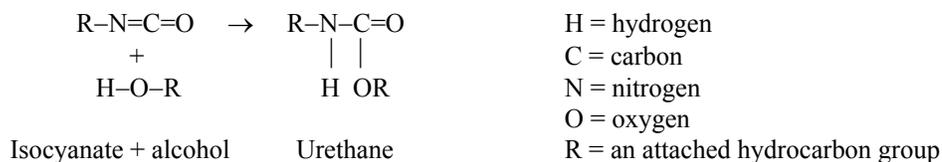
Wash the nylon well with water before handling.

Stir any remaining solutions left in the beak to form additional polymer. Wash the nylon well with water.

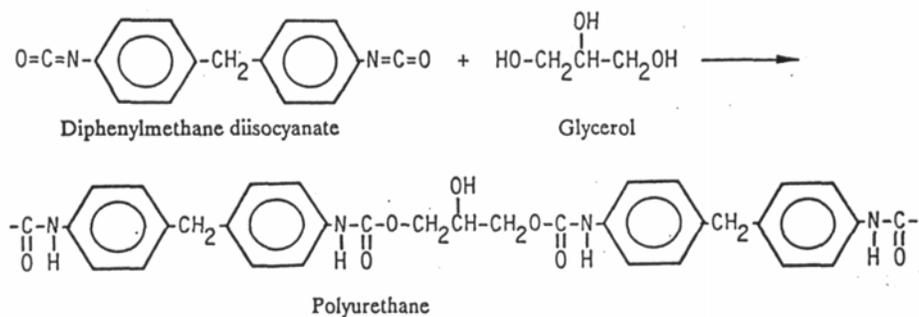
Commercially, nylon is made by mixing the components in bulk. Nylon thread, rod, and sheet are made by melting and forming or extruding the polymer.

## POLYURETHANE, A Foamed Condensation Polymer

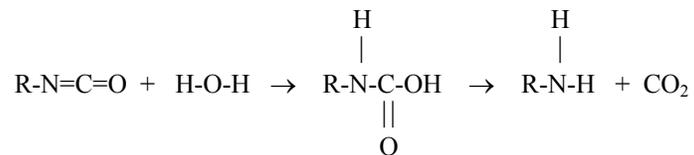
Polyurethane is formed from a two-part liquid mixture that, when mixed in equal amounts, produces a rigid foam that can be used to make castings of objects, materials for insulating or soundproofing, or other uses. This procedure uses preprepared monomer solutions to produce the foam. Part A consists of a polymeric diol or triol (glycerol is commonly used), a blowing agent, a silicone surfactant, and a catalyst. Part B contains a polyisocyanate (diphenylmethane diisocyanate). Upon mixing, a polymerization reaction occurs in three directions leading to a large molecule that is rigidly held into a three-dimensional structure. At the same time, the small amount of water present causes a decomposition of some of the isocyanate and the evolution of carbon dioxide which results in the foaming. The blowing agent, a low boiling liquid, is vaporized by the heat of the reaction. The carbon dioxide, along with the blowing agent, create gas bubbles in the viscous mixture as the foam sets into a rigid mass. The cell size and structure of the foam is controlled by the silicone surfactant. A generalized reaction scheme is shown below:



The actual reaction of the diphenylmethane diisocyanate to form polyurethane with glycerol is:



The generalized reaction forming the carbon dioxide is:



### Safety Precautions

Part B, of the polyurethane system, contains diphenylmethane diisocyanate (MDI), which is toxic as well as an irritant to the skin and eyes. It may cause an allergic response. Avoid breathing the vapor. Keep the container closed when not in use. Wear gloves.

Although the diphenylmethane diisocyanate is the least toxic of the diisocyanate compounds, DO NOT PERFORM THIS PREPARATION UNLESS THE ROOM HAS ADEQUATE VENTILATION.

### Materials Needed

Polyurethane foam system parts A and B (Available from Flinn Scientific or from craft/hobby shops under the name Craft Cast)

Paper cup, 5 oz

Stirring rod

Paper towel or newspaper

Disposable rubber gloves

Optional: food color

### Procedure

Spread a paper towel or a piece of newspaper flat on the bench top.

Measure 10 mL of polyurethane part A. Pour it into a 3 or 5-ounce paper cup.

Optional: If desired, the polyurethane can be colored by the addition of food color. Add 2 drops of food color to the polyurethane monomer in the paper cup. Stir well.

Measure 10 mL of polyurethane part B. Pour it into the paper cup containing the part A.

Place the paper cup of liquid in the center of the paper towel. Stir the liquid thoroughly until the mixture is uniform in color. Remove the stirring rod. Does the cup feel warm? Describe what occurs as the mixture expands.

Do not touch the foam. It is very sticky and it may contain some unreacted material. It will take about 5 to 10 minutes to surface set and up to 24 hours to cure.

## A Laboratory Preparation of Polyurethane Foam

### Safety Precautions

Wear safety goggles at all times in the laboratory

Toluene diisocyanate is toxic. It is an irritant to the skin and eyes. Avoid breathing the vapor. It may cause an allergic response. Work in a hood. Keep the container tightly closed. Wear gloves.

DO NOT PERFORM THIS PREPARATION UNLESS THE ROOM HAS A PROPERLY OPERATING FUME HOOD AND HAS ADEQUATE VENTILATION.

In the interest of safety, it is strongly recommended that a commercial product such as the polyurethane foam system or Craft Cast be used instead of preparing the polyurethane as described below.

### Materials Needed

Mixture A: 350 g of castor oil, 100 g of glycerol, 50 drops of stannous octoate (stannous 2-ethylhexanoate), 50 drops of Dow- Corning 200 silicone oil, and 150 drops of water. Place in a bottle, cap it and shake well. Use within 12 hours.

toluene diisocyanate (toluene-2,4-diisocyanate)

### Procedure

Work in a fume hood.

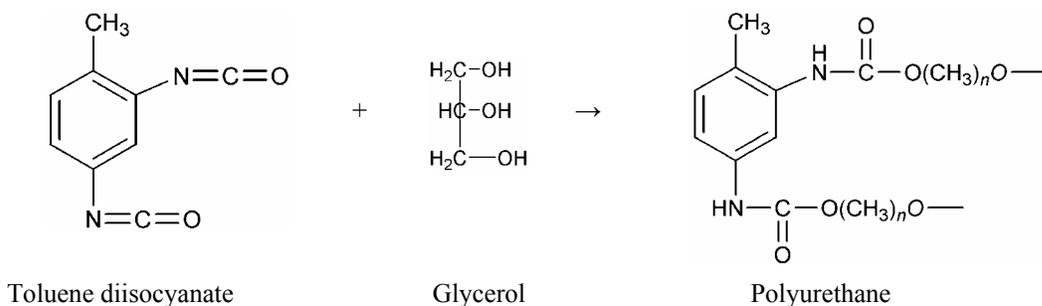
Pour 17 mL of mixture A (shake well before using) into a disposable cup.

Add 10 mL of toluene diisocyanate.

Place the cup on a piece of paper towel or newspaper. Stir the mixture rapidly and thoroughly with a stirring rod until a smooth and creamy mixture is obtained. When the mixture begins to bubble, stop stirring, foaming will be spontaneous. DO NOT BREATHE THE FUMES.

After foaming stops, allow to cool and set thoroughly before handling. The polyurethane will be firm after several hours. It will shrink noticeably upon standing.

The structure of the polyurethane is:



### Reference

Pavia, Donald L., Lampman, Gary M., and Kriz, George S. Jr., **Introduction to Organic Laboratory Techniques, A Contemporary Approach**, 2nd Ed., Saunders College Publishing, Philadelphia, 1982, page 389.

## **SODIUM POLYACRYLATE, A Copolymer**

Superabsorbants were originally developed by the United States Department of Agriculture in 1966. This material consisted of a grafted copolymer of hydrolyzed starch-polyacrylonitrile (polyacrylonitrile is commonly known as Acrilan, Orion, or Creslan). The intended use was for additives for drilling fluid in off-shore secondary oil recovery operations and as agricultural thickeners. These materials were followed by synthetic superabsorbants that are polyacrylic and polyacrylonitrile based. Some of these materials are capable of absorbing up to 2000 times their weight of distilled water.

When a starch-hydrolyzed polyacrylonitrile superabsorbant is mixed with glycerin or ethylene glycol, the resulting firm gel has a rubbery texture and is very strong and resilient. This material can absorb about 300 to 400 times its weight in distilled water and can "grow" many times its original size. This material was formed into various shapes and sold under names such as "Grow Creatures". The process is reversible and, on standing in air, the grow creature will shrink almost to its original size on drying. It can be grown and dried many times.

A useful application of "Super Slurper" is in the liners of Pampers and other disposable diapers. Under this application, the polymer gel can absorb up to 90 times its weight in liquid.

Super-absorbent material, sold under the name "Water Grabber" or "Water Lock" is available from garden supply stores and can be found in the garden department of some variety stores.

### **Safety Precautions**

There are no safety hazards with the materials used in this experiment.

### **Materials Needed**

Sodium polyacrylate  
Sodium chloride, NaCl  
Paper cup, 5 oz  
Stirring rod  
Disposable diaper  
Newspaper

### **Procedure**

Measure a level  $\frac{1}{2}$  teaspoon of superabsorbent material and place the material in a 5 oz paper or plastic cup.

Add 100 mL of water to the cup.

Record your observations.

Add  $\frac{1}{2}$  teaspoon of salt, sodium chloride, to the mixture. Record your observations.

### **Sodium polyacrylate from a disposable diaper**

To obtain the superabsorbant material from a disposable diaper: Obtain an ultra-absorbent diaper. Spread out some newspaper on a table top or the floor.

Slowly, take the diaper apart. Observe the different layers. What is the function of each layer?

Shake the cotton well to remove any superabsorbent material. Throw the waste cotton into the trash. Rub the outside liner of the diaper to remove any superabsorbent material adhering to the plastic.

Transfer the powder and cotton on the newspaper to a 5 oz. plastic or paper cup. Remove any loose cotton, leaving the powder in the cup. Add 50 mL of water to the powder in the cup and stir. Observe the material in the cup for about a minute. Record your observations.

Testing a disposable diaper: Open a disposable diaper on the bench top.

Add water, 100 mL at a time, until the diaper does not hold additional water.

Record the amount of water held by the diaper.

## Some Inorganic Polymers

Some examples of Inorganic Polymers: (Ref.: H. R. Allcock, **Scientific American**, **230**, 66, March 1974)

Glass: made of rings and chains of repeating silicate units. Glass fibers (fiberglass) are used in textiles and in many construction materials.

Bricks, concrete and ceramics are three-dimensional polymers.

Polyphosphazenes: general formula  $(\text{NPCl}_2)_n$ . Formed from reacting  $\text{NH}_4\text{Cl}$  with  $\text{PCl}_5$  at  $250^\circ\text{C}$  for several hours. Can be substituted to alter properties.

## Polymeric Sulfur

### Materials Needed

Sulfur

### Safety Precautions

It is recommended that a hot plate be used in heating the sulfur in this preparation. This will prevent the sulfur from igniting and burning to form toxic sulfur dioxide, a strong irritant. Keep the beaker of sulfur covered with a watch glass to minimize sulfur vapors escaping from the beaker.

### Procedure

Heat ordinary sulfur to a temperature between  $140$ - $170^\circ\text{C}$ . The sulfur will turn red and become viscous above  $159^\circ\text{C}$ . At  $175^\circ\text{C}$ , the viscosity of the molten sulfur begins to decrease.

Pour the hot melt into water in a thin stream. When cool, it forms rubbery fibers of the Polymeric sulfur. The polymer is not stable at room temperature and it will eventually harden, returning to its normal yellow color, when the molecules reform into eight-membered rings.

## SILLY PUTTY, An Inorganic Polymer

Silly Putty is a silicone polymer marketed by Binney & Smith Inc. Easton, PA 18042, and with similar products marketed under other names by other companies. It comes packaged in small egg-shaped containers and is usually pink in color. Some forms of Silly Putty contain phosphorescent material that will allow it to glow in the dark.

Silly Putty is a non-Newtonian fluid which has dilatant properties. That is, it tends to dilate (or expand) when sheared resulting in an increased viscosity under stress. For this reason, Silly Putty has some unique properties:

- Under low stress, such as slowly pulling the Silly Putty apart, the putty flows forming thin strands.
- Under high stress, such as a sharp pull, the putty breaks.
- If rolled into a ball and dropped, the putty will bounce.
- If the ball of putty is placed on a table top and hit with your hand, the ball will hardly be deformed. If hit with a hammer, the putty will shatter. Yet, if you squeeze gently, the ball will flatten.
- If you stuff the putty through a tube, it will swell as it emerges from the open end. This is known as die-swell. (This works well with freshly prepared putty as the putty tends to harden with age.)

### The Preparation of Silly Putty

#### Materials needed

dimethyldichlorosilane,  $\text{SiCl}_2(\text{CH}_3)_2$   
diethyl ether, anhydrous,  $(\text{C}_2\text{H}_5)_2\text{O}$   
sodium hydrogen carbonate,  $\text{NaHCO}_3$ , saturated aqueous solution  
boric oxide,  $\text{B}_2\text{O}_3$  sometimes called boric acid, fused)  
sodium sulfate,  $\text{Na}_2\text{SO}_4$ , anhydrous  
safety shield  
flask, 250-mL, and 125-mL  
separatory funnel  
test tube, Pyrex, 25 x 250 mm  
stirring rod

#### Safety Precautions

Wear safety goggles at all times in the laboratory.

Dimethyldichlorosilane reacts violently with water to produce heat and toxic and corrosive fumes (HCl). Avoid skin contact and use it only in a fume hood.

Ether is extremely flammable, avoid the use of flames in the laboratory during this procedure.

When washing the ether solution with sodium hydrogen carbonate, carbon dioxide is released building up pressure in the separatory funnel. Vent the funnel often.

#### Procedure

This procedure must be carried out in a fume hood or in an area with excellent ventilation and behind a safety shield.

To a 250-mL flask add 20 mL of dimethyldichlorosilane and 40 mL of anhydrous ether. Hydrolyze the solution by adding 40 mL distilled water DROPWISE, allowing the reaction to cease before adding additional water. (If water is added too rapidly, the reaction will proceed vigorously and may spatter from the flask. Avoid inhaling any vapors from the flask.)

After the water addition is complete, transfer the reaction mixture to a separatory funnel and drain off and discard the water layer. Wash the remaining ether solution with 10 mL portions of saturated sodium hydrogen carbonate, venting often, until it is no longer acid (and no more  $\text{CO}_2$  forms), then wash with 10 mL of distilled water. Drain the ether layer into a 125-mL flask containing 2 grams  $\text{Na}_2\text{SO}_4$  and allow to dry for 15-20 minutes.

Filter the solution into a 100-mL beaker and transfer to a water bath or steam bath to evaporate the ether.

Determine the mass of the residual silicone oil. Add about 5% of the silicone oil's mass of boric oxide, and stir for a few minutes. Transfer the mixture to a Pyrex test tube and heat in an oil bath at 200°C for three hours. Allow the silly putty to cool.

NOTE: Initially, the silly putty will be a clear, colorless to slightly yellow material. It will cure within one week to a white solid with properties closer to the commercially available Silly Putty. As it is used, it will pick up foreign matter and become gray in color slowly improving its properties.

Reference: Armitage, D. A., et. al., J. Chem. Educ., 50, 434 (June 1973)