AN ADDITION REACTION

This demonstration illustrates the addition of bromine to the double bond in cyclohexene, but not to cyclohexane or benzene.

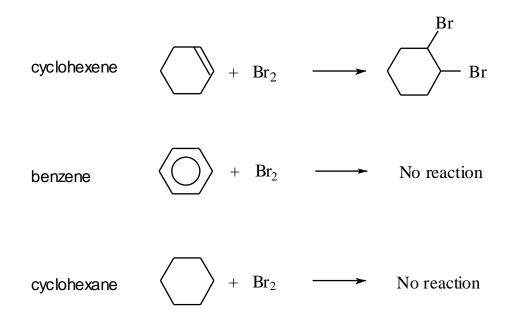
- **EQUIPMENT** perspex test tube rack
 - 3 x 25 cm test tubes
 - 10 mL measuring cylinder
 - light box

REAGENTS

- cyclohexene (25 mL)
- cyclohexane (25 mL)
- Toluene (25 mL)
 - bromine in DCM (2%, 30 mL)
- **PREPARATION** Pour 25 mL of each of the first three of the above reagents into separate labelled test tubes.
 - Provide the bromine solution in a stoppered reagent bottle.
 - Set up light box with the test tubes in their rack.
 - Provide measuring cylinder.

PROCEDURE Add bromine solution (10 mL) to each test tube.

RESULTS An addition reaction occurs when bromine is added to cyclohexene, as indicated by the loss of colour of the bromine solution. There is no colour loss with benzene or cyclohexane.



OXIDATION OF ALCOHOLS

Four alcohols, when mixed with potassium dichromate are oxidised to different extents.

EQUIPMENT .

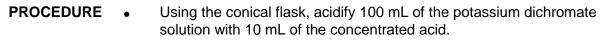
- perspex test tube rack
- 3 test tubes, 25 cm long
- measuring cylinder, (10 mL)
- 250 mL conical flask
- light box
- thermometer, (0-100°C)
- water bath
- bunsen burner



- ethanol, CH₃CH₂OH (25 mL)
 - n-butanol, CH₃CH₂CH₂CH₂OH (25 mL)
 - 2-butanol, CH₃CH₂CH(OH)CH₃ (25 mL)
 - tert-butanol, $(CH_3)_3COH$ (25 mL)
 - potassium dichromate, $K_2 Cr_2 O_7$ (0.2 M, 100 mL)
 - sulfuric acid, H_2SO_4 (18 M, 10 mL)

PREPARATION

- Provide 100mL of 0.2M potassium dichromate solution in a 250mL conical flask.
- Provide 10mL of 18M sulphuric acid in a stoppered 10mL measuring cylinder
- Place 25 mL of each alcohol into a separate, labelled test tube.
- Set up the light box with test tubes and rack .



- This process will heat up the solution..
- Add 10 mL of the potassium dichromate solution to each test tube.

RESULTS Ethanol is oxidised: The colour changes to olive green.

3CH₂CH₂OH + 2Cr₂O₇²⁻ + 16H⁺ ζ 3CH₂COOH + 4Cr³⁺ + 11H₂O

n-butanol is oxidised: After some 20 seconds the colour changes to greenish brown.

 $3CH_{3}CH_{2}CH_{2}CH_{2}OH + 2Cr_{2}O_{7}^{2} + 16H^{+} \zeta 3CH_{3}CH_{2}CH_{2}COOH + 4Cr^{3+} + 11H_{2}O$ 2-butanol is oxidised: After 30 seconds the colour changes to blue-green. $3CH_{3}CH_{2}CH(OH)CH_{3} + Cr_{2}O_{7}^{2} + 8H^{+} \zeta 3CH_{3}CH_{2}C(O)CH_{3} + 2Cr^{3+} + 7H_{2}O$ tert-butanol (CH₃)₃COH is not oxidised: No colour change takes place.

High Risk Demonstration: 1- Refer to HIRAC 2- Set up in Red Tray



AN ALDOL CONDENSATION REACTION

To observe the formation of dibenzalacetone or dicinnamalacetone.

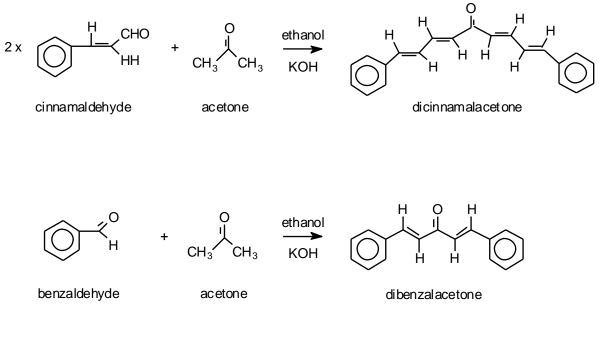
- EQUIPMENT 400 mL beaker
 - stirring rod
 - light box

REAGENTS

- cinnamaldehyde [or benzaldehyde], C₆H₅CHCHCHO [or C₆H₅CHO] (20 mL)
- ethanol, CH_3CH_2OH (125 mL)
- potassium hydroxide, KOH (2 M [4 M], 75 mL)
- acetone, CH_3COCH_3 (10 mL)
- **PROCEDURE** Dissolve the cinnamaldehyde (or benzaldehyde) in the ethanol in the beaker on the light box.
 - Stir in the potassium hydroxide to achieve a homogeneous solution.
 - Add the acetone.

RESULTS The solution will remain clear for thirty seconds, after which time bright

yellow crystals will appear throughout the solution.



- Refer to HIRAC
- Set up in Red Tray

THE "PROOF TEST"

The	ignition	of aun	powder	with ethanol.
1110	ignition	or guin	pomaor	with otherior.

EQUIPMENT	 3 x evaporating basins, 8 cm diameter 3 x 50 mL conical flasks, with stoppers 3 x sample tubes with lids insulating mat taper and matches 	
REAGENTS	 gun powder (15 g) (Ask Marjan for gun powder) absolute ethanol, CH₃CH₂OH (300 mL) 	
PREPARATION	 Using the sample tube, weigh out 5 g of gun powder. Stopper the tube. Set up the basin on the insulating mat. Provide gun powder, taper, matches and safety glasses. Place ethanol in the flask and seal with the stopper. 	
PROCEDURE	 Put the gun powder into the basin. Use sufficient ethanol to cover the powder completely. Repeat using a mixture of 75% ethanol:water. Repeat using a mixture of 50% ethanol:water. 	
	Remove any flammable substance within a 3 metre radius. Warn the audience to avoid looking directly at the basin.	
<u>E</u>	 Light the taper and standing well back ignite the alcohol at arm's length. 	
RESULTS	In tests 1 and 2, the ethanol will burn off quietly. Then, suddenly, with a blinding flash and a puff of greenish white smoke, the combustion of the gur powder will take place. In the third test with 50% ethanol:water the gun powder will not ignite.	

- Refer to HIRAC
- Set up in Red Tray

POLYMERISATION: THE NYLON ROPE

The synthesis of nylon can be successfully and conveniently performed with reagents contained in a 250 mL beaker.

- EQUIPMENT 2 x 250 mL beakers
 - tongs
 - rotating rod on stand

REAGENTS

• sebacoyl chloride (1 mL)



- Hexane (50 mL)
- 1,6-diaminohexane (1.2 g)
- anhydrous sodium carbonate (2 g)
- distilled water
- **PREPARATION** Perform this demonstration in a fume hood.
 - Using one of the beakers, dissolve 1 mL of sebacoyl chloride in 50mL of hexane
 - Fill the other beaker with 50mL mL distilled water.
 - Add 1.2 g of 1,6-diaminohexane and 2 g of sodium carbonate.
 - Mix well.
 - Set up the solutions and the special apparatus.
 - Provide tongs.
- **PROCEDURE** Pour the hexane solution carefully on top of the aqueous solution, to form a floating layer.
 - A nylon membrane will form at the interface of the two solutions.
 - Using the tongs, take hold of it and pull it up.
- **RESULTS** The lifting of the membrane will cause it to collapse into a rope. If you draw the rope over the roller and wind it up, new material will continuously form as long as both solutions are present.
- CAUTION Both 1,6-diaminohexane and sebacoyl chloride are irritants to the respiratory system.

DEHYDRATION OF SUCROSE

Sulfuric acid is added to a beaker containing sugar. After a short period, a reaction occurs in which a black foam is produced.

This demonstration should be performed in a fume hood.

EQUIPMENT • 200 mL beaker

REAGENTS

- sucrose, $C_{12}H_{22}O_{11}$
- sulfuric acid, H_2SO_4 (18 M, 5-10 mL)
- **PROCEDURE** Fill about one-third of the beaker with sugar (sucrose).
 - Add the sulfuric acid carefully and do NOT stir.
 - Stand at a safe distance.
- **RESULTS** After a short time a violent reaction will occur, in which a "carbon snake" is produced. Care must be taken as sulfur dioxide is in the fumes. Also, handle the snake with gloves as it still contains sulfuric acid.

 $C_{12}H_{22}O_{11}(s) \zeta 12C(s) + 11H_2O$

The sulfuric acid is acting as a dehydrating agent.

- Refer to HIRAC
- Set up in Red Tray

OPTICAL ACTIVITY: ROTATION OF LIGHT

Two solutions are observed to rotate plane polarised light in opposite directions, thus indicating that they are optically active.

- **EQUIPMENT** glass cylinders on an extended, flat base, about 50 mL
 - 2 sheets of polarising material: -
 - sandwiched between perspex about 10 x 10 cm
 sandwiched between perspex about 50 x 50 cm
 - small watch glasses to cover the cylinders
 - small labels for the watch glasses
 - tissues

REAGENTS • R(+)-limonene

- S(-)-limonene
- deionised water
- and if desired also: L-menthol, D-menthol sucrose, C₁₂H₂₂O₁₁
- **PREPARATION** Place each of the concentrated limonenes into a separate cylinder.
 - Place water into a third one.
 - Do not fill the cylinders to more than three-quarters.
 - If desired, make up also the following solutions:
 100 g of a menthol in 200 mL of ethanol
 100 g of sucrose in 200 mL deionised water
 - Place each solution into a separate cylinder.
 - Label the watch glasses and cover the solutions with these.
 - Place the larger of the perspex/polaroid sandwiches on the over-head projector.
 - Set the vessels containing the solutions on a tray alongside the projector.
 - Provide the smaller of the perspex/polaroid sandwiches and the tissues.

CAUTION Avoid spillage onto the overhead projector - There is a risk of a short circuit and corrosion of the perspex surface and polarising material.

- **PROCEDURE** Dim the lecture theatre lights.
 - Place the solutions and the water over the polarising material on the projector.
 - Hold the other polarising material horizontally above the cylinders.
 - Rotate the sheet until the extinction of the light transmitted by the water is at a maximum.
 - From here, slowly continue to rotate the sheet clockwise and anticlockwise.
- **RESULTS** All solutions will prove to be optically active. They will differ from each other by the degree and the direction in which they rotate light. Water is inactive.

CANNED HEAT: ALCOHOL GELS

Two clear liquids, when mixed together, produce a solid gel which may then be ignited.

		-
EQU	JIPMEN	

- metal can, with a capacity of 500 mL, or 500 mL beaker
- matches (optional)

REAGENTS
A

- ethanol, CH₃CH₂OH (95%, 300 mL)
- calcium acetate-1-water, $Ca(C_2H_3O_2)_2H_2O$ (40 g)

PREPARATION • Prepare 100 mL saturated calcium acetate solution by placing 40 g calcium acetate-1-water in a stoppered flask with 100 mL of water.

- Shake occasionally over a period of a few days.
- Add more solid if it all dissolves.
- Decant the saturated solution from the solid that settles in the bottom.
- **PROCEDURE** Pour 300 mL ethanol and 60 mL of the saturated calcium acetate solution simultaneously into the clean, dry beaker or metal can.
 - Do not stir.
 - Wait 10 seconds and invert the beaker.
- **RESULTS** A gel will form and remain in place when the container is inverted. It may be ignited if the demonstration is done in the metal can (glass may shatter due to the heat). The flame may be seen in a darkened room.

The gel forms due to the reduction of solubility of calcium acetate when ethanol is added. The calcium acetate precipitates rapidly, forming a network of solid throughout the liquid, which entraps the liquid within it, forming a gel.

AN UNSATURATION TEST

Bromine water is discoloured in the presence of a common unsaturated (non-aromatic) compound.

EQUIPMENT • 1 L flask

•

- bunsen burner
- tongs
- stopper
- bacon

REAGENTS

300 mL of bromine water



- Quickly open a large (e.g. 1 L) flask containing the bromine water and drop in strips of well-done (crispy) bacon.
- Shake the flask.

RESULTS

The colour of bromine disappears.

Bromine adds to the unsaturated aldehyde $CH_2 = CH - CHO$ (the bacon odour).

 $CH_2 = CH - CHO + Br_2 \zeta CH_2Br - CHBr - CHO$



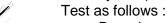
PREPARATION OF ACETYLENE

Acetylene is generated, then used to discolour bromine water.

- **EQUIPMENT** Set up the apparatus as in the diagram below
- REAGENTS
- calcium carbide, CaC_2
- sand
- water

PROCEDURE

- Prepare some acetylene by adding water to calcium carbide in the apparatus shown below.
- Collect several test tubes of gas (after displacing the air from the system).



- Burn the gas in the test tube.
- Shake an aqueous solution of bromine.

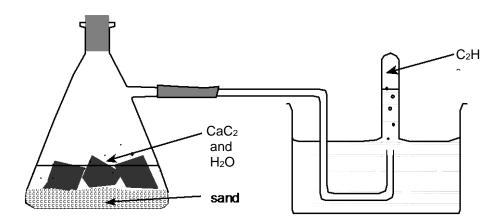
RESULTS

The gas produced burns with a smoky flame.

Bromine adds to the triple bond of acetylene, decolourising the bromine water.

Note the distinctive odour of acetylene.

 $C_2H_2 + 2 Br_2 \zeta C_2H_2Br_4$



THE DEHYDRATION OF FORMIC ACID BY SULFURIC ACID

Concentrated sulfuric acid is added to formic acid to produce carbon monoxide which burns in air with a blue flame.

- **EQUIPMENT** 1 x 250 mL Erlenmeyer flask
 - 1-holed rubber stopper to fit flask
 - glass tubing
 - matches
 - taper
- **REAGENTS** sulfuric acid, H₂SO₄ (18 M, 50 mL)
 - formic acid, $HCO_{2}H$ (50 mL)

CAUTION Carbon monoxide is a colourless, odourless gas that is very toxic. Carry out this demonstration in a fume hood.

- **PREPARATION** Form a nozzle out of one end of the glass tubing.
 - Make a 90° bend about half way along the length of the tubing.
 - Insert the glass tubing into the stopper.

PROCEDURE

- Add 50 mL of sulfuric acid to 50 mL of formic acid and insert rubber stopper/glass tubing assembly into the neck of the flask.
- When sufficient gas has been generated, ignite the gas with a lighted taper.

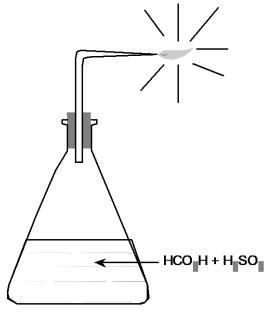
RESULTS

The gas liberated burns in air with a blue flame.

 $HCO_{2}H(I) \longrightarrow CO(g) + H_{2}O(I)$

The carbon monoxide is produced by the dehydration of formic acid.

- Refer to HIRAC
- Set up in Red Tray



THE "CARBON SNAKE"

Sulfuric acid is added to 4-nitroacetanilide, then stirred and heated. A violent reaction occurs, in which a black foam is produced.

EQUIPMENT

- 10 mL measuring cylinder
- tall 250mL beaker
- small insulating mat with 5 cm diameter hole
- large insulating mat
- retort stand with ring
- Meker burner and matches
- 20 cm stirring rod
- portable fume hood

REAGENTS

- 4-nitroacetanilide, $O_2N-C_6H_4-NH-CO-CH_3$ (10 g)
 - sulfuric acid, H_2SO_4 (18 M, 6 mL)

PREPARATION • Set up the portable fume hood so that the flexible duct leads into the fume cupboard.

- Place the retort stand on the large insulating mat in the fume hood.
- Place the burner on the foot of the stand.
- Use the retort ring to support the small mat over the burner.
- Weigh out fresh 4-nitroacetanilide (10 g).
- Place it in the tall beaker and set over the hole in the mat.
- Using the measuring cylinder, provide sulfuric acid (6 mL).
- Lay out stirring rod and matches.

PROCEDURE • Turn on the ventilation of the fume hood and fume cupboard.

- Light the burner.
- Add the acid to the acetanilide and stir.
- It is essential that this mixing be thorough and that all of the 4-nitroacetanilide is wetted by the acid.
- With agitation and heating the mixture turns dark and syrupy.
- Within one or two minutes, the mixture will boil and smoke.
- The stirring rod should be taken well away from the reaction mixture, otherwise the formation of a well-shaped product is jeopardised by the collision of the evolving foam with the rod.

CAUTION SULFUR TRIOXIDE! Agitation should cease when smoke evolution becomes distinct.

RESULTS Amid effervescent hissing and smoke evolution a long, black body of stiff, spongy foam will shoot up from the vessel, reaching a length of about 1 metre. This is the "Carbon Snake"! When the smoke abates, the "snake" will probably stand quietly towering over the vessel. On rare occasions, however, the "snake" will form with such speed and energy that it leaps completely out of the vessel. It is therefore advisable to be "on the ready" to catch it in mid-air if necessary.

This reaction is complex, involving sulfonation, cyclisation and finally dehydration. The mechanism for it is not known.

- Refer to HIRAC
- Set up in Red Tray

PHENOL-FORMALDEHYDE POLYMERISATION

Concentrated hydrochloric acid is added to a mixture of aqueous formaldehyde, glacial acetic acid, and phenol. After approximately one minute the solution turns pink and solidifies into a cake-like material.

- EQUIPMENT 250 mL beaker
 - 100 mL graduated cylinder
 - stirring rod
 - gloves, plastic or rubber

REAGENTS

• formaldehyde, HCHO (formalin, 34M) (25 mL)



- glacial acetic acid, CH₃COOH (17 M, 55 mL)
- phenol, C_6H_5OH (20 g)
- hydrochloric acid, HCI (10 M, 55 mL)
- **PROCEDURE** In a 250 mL beaker, mix 25 mL of aqueous formaldehyde, 55 mL of glacial acetic acid, and 20 g of phenol.
 - Quickly add 55 mL of concentrated hydrochloric acid while stirring.
 - After about a minute, the solution will turn pink and solidify.
 - If not stirred, the resulting polymer is friable, but continuous stirring during polymerisation results in a solid mass more resistant to crumbling.

CAUTION

All compounds used in this demonstration should be handled in a fume cupboard. Plastic or rubber gloves should be worn.

Formaldehyde vapours are extremely irritating to mucous membranes. Skin contact can cause dermatitis. Extended exposure to high concentrations of vapour can cause chronic effects such as laryngitis, bronchitis, conjunctivitis, or skin problems. Moreover, mixing of formaldehyde with hydrogen chloride can result in the generation of a potent human carcinogen.

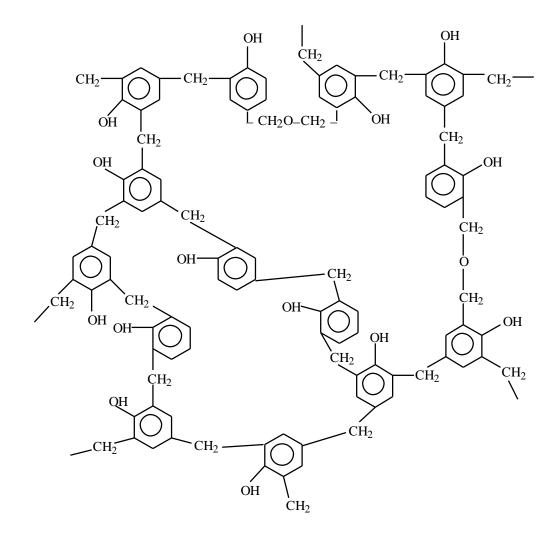
Phenol is toxic and causes burns. It can be absorbed rapidly through the skin. Prolonded inhalation can have chronic effects.

Glacial acetic acid and concentrated hydrochloric acid can produce skin burns, eye irritation, and irritation of the respiratory tract.

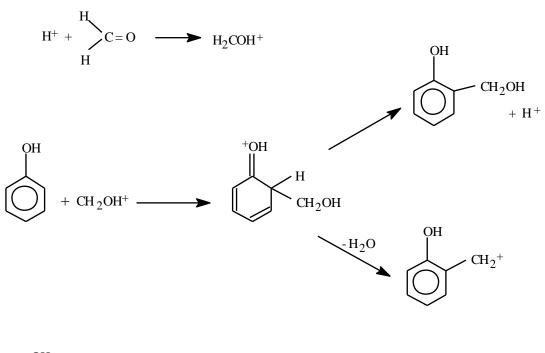
DISPOSAL The product contains acid and probably unreacted formaldehyde or phenol; before handling it should be washed thoroughly with dilute aqueous sodium hydroxide solution and then water. The washed polymer should be discarded in a waste container.

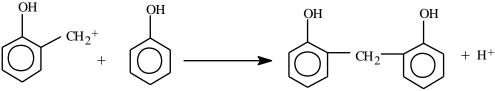
- Refer to HIRAC
- Set up in Red Tray

DISCUSSION In general, phenol-formaldehyde resins are condensation polymers consisting of aromatic rings linked by methylene groups. The product linkages occur primarily in the ortho or para position to the phenolic hydroxyls. In addition, ether linkages are found under conditions of excess formaldehyde and neutral pH.



In this demonstration, prepared with a molar excess of formaldehyde and under acidic conditions, the product resin is formed by reactions that lead mostly to methylene bridges. The reaction sequence can be illustrated as follows:





All possible bridges do not form, because all phenolic rings do not lie in the same plane. These gaps in the resin weaken the polymer. The presence of occluded reaction materials and by-products, such as water, causes further weakening.

POLYSTYRENE

Clear, liquid styrene is mixed with an initiator, benzoyl peroxide, and the mixture is then heated in a boiling water bath. The styrene slowly polymerises to form a transparent solid.

EQUIPMENT

- 2 x 800 mL beakers
- stirring rod
- 11 cm filter funnel
- test tube, 16 mm x 150 mm, with stopper
- hot plate
- gloves, plastic or rubber

REAGENTS

- alumina, Al_2O_3 (3 g)
- benzoyl peroxide, $(C_6H_5CO)_2O_2(0.5 \text{ g})$

styrene, C_eH_eCH=CH₂ (20 mL)

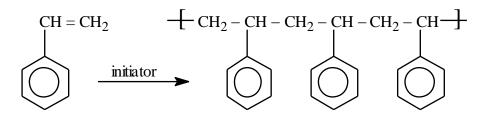
- To remove the inhibitor from the styrene, place 20 mL of styrene in a small beaker and add 2-3 g of alumina.
 - Stir the mixture well to form a slurry.
 - Filter the mixture through filter paper supported in a small funnel.
 - Place the styrene in a test tube.
 - Add 0.5 g of benzoyl peroxide; stopper and shake the tube well to dissolve the initiator.
 - Remove the stopper and heat the tube in a boiling water bath for 10-20 minutes.
 - The styrene will become viscous with heating.
- CAUTION The demonstation should be performed in a fume cupboard. Plastic or rubber gloves should be worn. Styrene is flammable, and it is irritating to the eyes and mucous membranes. In high concentrations, it can be narcotic.

Benzoyl peroxide (dibenzoyl peroxide) is highly flammable and shock sensitive. It should not be stored in screw-capped bottles, because the friction produced by opening the bottle could cause an explosion.

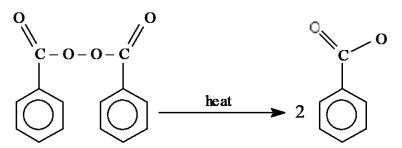
DISPOSAL Although the polymer will ignite in a direct flame, it is otherwise inert and should be discarded in a waste container.

- Refer to HIRAC
- Set up in Red Tray

DISCUSSION Free radical polymerisation of styrene can be carried out by bulk, solution, suspension, or emulsion techniques. The reaction equation can be represented as:



The arrangement of styrene in the polymer is considered to be "head-totail". The initiator, benzoyl peroxide, is cleaved by heat into two benzoyl radicals:



Polystyrene is one of the major commercial plastics widely used in appliances, toys, food containers, packaging, etc. It is a good electrical insulator. Many copolymers of styrene with other monomers have been investigated, and some, such as styrene-acrylonitrile copolymers, are commercially important.

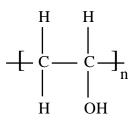
"Slime": Gelation of Poly(vinyl alcohol) with Borax

Two clear, colourless liquids are mixed and almost immediately form a gel. The gel can be formed into a ball, but if left unhandled, it will flatten and run.

EQUIPMENT	 250 mL beaker stirring hot-plate, with stir bar thermometer, -10 °C to +110 °C 100 mL disposable plastic cup, or beaker flat-bladed wooden stirrer
REAGENTS	 distilled water (50 mL) poly(vinyl alcohol), [-CH₂CH(OH)-]_n (2.5 g). The polymer should be >99% hydrolysed (average molar mass at least 105 gmol⁻¹) aqueous solution of sodium tetraborate, Na₂B₄O₇ (4% by weight, 5 mL). (To prepare 100 mL of solution, dissolve 7.6 g of Na₂B₄O₇.10H₂O [borax] in 100 mL of distilled water.)
PREPARATION	 Pour 50 mL of distilled water into the 250 mL beaker. Place a stir bar in the beaker, and set the beaker on the stirring hot plate. Warm the water to a temperature not to exceed 90 °C.
PROCEDURE	 While stirring the warm water, slowly sprinkle 2 g of poly(vinyl alcohol) on the surface of the water. This procedure prevents the formation of a sticky mass of polymer that is difficult to dissolve. Pour the poly(vinyl alcohol) solution and 5 mL of sodium borate solution together into a plastic cup and stir the mixture vigorously with the wooden stirrer. Gel formation begins almost immediately, and a material, "slime". Knead the gel into an elastic ball. Hold the ball in the palm of your hand and tip your hand. The ball will stretch out into a long column. Attempt to stretch the column abruptly; it will break.
DISPOSAL	The plastic cup and wooden spoon used to prepare the gel should be discarded in a solid-waste container. The gel can be discarded in a solid- waste container, or by mixing with plenty of water and flushing the mixture

down the drain with more water.

DISCUSSION Poly(vinyl alcohol) is a polymer with a structure composed of repeating vinyl alcohol units:



The average value of n for the poly(vinyl alcohol) used in this demonstration is at least 2300.

Sodium borate hydrolyses in water to form a boric acid-borate ion buffer with a pH around 9.

 $B_4O_7^{-2}(aq) + H_2O(I) + 1 HB_4O_7^{-1}(aq) + OH^{-1}(aq)$

In this demonstration, the borate ions react with the hydroxyl groups of the polymer to form cross links possibly with the elimination of water. These cross links probably involve hydrogen bonds that break and reform under flow. Such relatively weak cross-linking is necessary to produce the viscoelastic gel and the interesting physical properties of "slime". The capability of poly(vinyl alcohol) to engage in extensive hydrogen bonding is suggested by its high solubility in water.

CHROMATOGRAPHY

Separation and purification are demonstrated using column chromatography.

EQUIPMENT • glass chromatography column, 2cm diameter

- chromatography silica for gravity column (63-200 μm particle size, 22g)
- measuring cylinder, (100mL)
- 250 conical flask
- three 100mL beakers
- sample vial and pasteur pipette

REAGENTS



- hexane, CH₃(CH₂)₄CH₃ (90mL)
 - dichloromethane, CH₂Cl₂ (90mL)
 - diethyl ether, CH₃CH₂OCH₂CH₃ (20mL)
 - ferrocene, FeCp₂ (20mg)
 - acetylferrocene, Fe(AcCp)Cp (20mg)

PREPARATION • Mix the solvents

- Place a small plug of cotton wool at the bottom of the column and half fill with solvent
- Pour in the silica and allow to settle
- Mix the ferrocene and acetylferrocene in a sample vial
- Just before the lecture, run the solvent so it is ½ cm above the top of the silica
- **PROCEDURE** Dissolve the compound mix in 1mL of dichloromethane and load into the column
 - Carefully add more solvent to the column
 - Elute into beakers
- **RESULTS** The compounds quickly separate, the yellow ferrocene eluting first followed by the red acetylferrocene.