SOPs/ INSTRUCTION MANUAL

1) Preparation of polymers such as urea-formaldehyde resin

Chemicals Required :Urea ,Formaldehyde, concentrated Sulphuric acid

Theory : The two important classes of amino resins are the condensation products of urea and melamine with formaldehyde. They are considered together here because of the similarity in their production and applications. In general, the melamine resins have somewhat better properties but are higher in price. Chemistry and Production: And which condensed with two more urea molecules to form a liner polymer. Urea is dissolved in aqueous formaldehyde in a throw-away container . Acidification of this solution initiates condensation polymerization, and a hard, white, thermosetting polymer is formed within a few minutes . After washing, the properties of this substance can be investigated .If a mold is used, the experiment could be extended to show the formation of plastic articles made in a mold by condensation polymerization. The reaction is a condensation polymerisation in which water is eliminated as the hydrogen atoms from the ends of one amino group from each of two urea molecules combine with the oxygen atom from a formaldehyde molecule .The remaining –CH2– group from the formaldehyde molecule then forms a bridge between two neighboring urea molecules, as shown below .This process, repeated many thousands of times, forms long chains of urea and formaldehyde molecules linked in this way . Sometimes the second hydrogen atom on an amino-group will also react with a formaldehyde molecule, producing a branch in the chain, and chains may even become cross-linked to each other .Eventually a random three-dimensional network of cross-linked chains is formed, giving a rigid structure and thus a hard, inflexible material .The product has many cross links :Because the tangle of cross- linked chains is almost impossible to separate, the material does not melt on heating, although it will eventually break down at high temperature, decomposing and giving off small molecules such as steam and nitrogen, leaving a charred mass which is largely carbon.
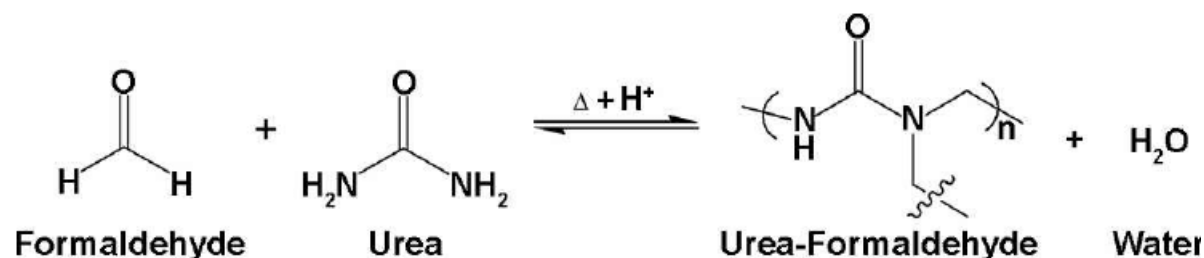
Procedure : Dissolve 10 g of urea in 20 mL of formaldehyde solution (formalin $= 37 - 40$)% , in the disposable container . Add about 1 mL of concentrated sulfuric acid H_2SO_4 a drop at a time, using a dropping pipette, and stir steadily .Within a minute the solution begins to go milky and eventually a hard, white solid is formed which is difficult to remove from the container. A lot of heat is evolved .Show that the polymer is hard by poking the material with a spatula. Wash the polymer thoroughly before passing around the class, as it is likely to be contaminated with unreacted starting materials .Alternatively, pass it round in the container with the lid screwed on.

Make a plastic mold from the simple shape and line it with aluminum foil. Make another urea-formaldehyde solution as above, but, immediately after adding the acid, pour some of the solution into the mold and allow it to polymerize, Remove from the mold when solid. Hold a sample of the polymer from the mold with tongs and heat in a Bunsen flame .It will char but not melt, showing that it is a thermosetting polymer.

2) To find the surface tension of methyl alcohol, ethyl alcohol and n hexane at room temperature and then calculate the atomic parachors of carbon, hydrogen and oxygen.

Apparatus : Stalagmometer, pyknometer, three liquids, beakers etc.

Theory : The parachor, [P] of a liquid of molecular weight M and density D is given by-

$$[P] = \frac{M\gamma^{1/4}}{D}$$

Where, γ = surface tension.

First, the surface tension and density of each liquid is determined by means of a stalagmometer and pyknometer as usual. Thus, the value of parachor of each liquid can be calculated. Atomic parachors of carbon, hydrogen and oxygen can be found as follows:

a) $[P_{EtOH}] = [P_{MeOH}] - [P_{CH_2}]$
b) $[P_{hexane}] = 6[P_{CH_2}] = 2[P_H]$
c) $[P_{CH_2}] - 2[P_H] = [P_C]$

Procedure: First tht stalagmometer is washed and dried. It is then filled with water and clamped vertically.

The number of drops is counted between the two marks x and y.

Similarly, the stalagmometer is washed with every liquid in turn and the number of drops is counted. The densities of each liquid are also measured by pyknometer, as usual. The room temperature is also noted.

Observations:

a) Weight of empty pykonmeter = W_1 g
b) Weight of pyknometer + water = W_2 g
c) Weight of pyknometer + CH_3OH = W_3 g
d) Weight of pyknometer + C_2H_5OH = W_4 g
e) Weight of pyknometer + n-hexane   = W_5 g

Liquid	Number of drops	Average number of drops	Liquid	Number of drops	Average number of drops
Water CH$_3$OH			C$_2$H$_5$OH n-hexane		

Let the molecular weights of CH$_3$OH, C$_2$H$_5$OH and n-hexane be M$_1$, M$_2$, M$_3$ respectively.

Calculations:

Density of methyl alcohol $= \dfrac{W_3 - W_1}{W_2 - W_1}$

Density of ethyl alcohol $= \dfrac{W_4 - W_1}{W_2 - W_1}$

Density of n-hexane $= \dfrac{W_5 - W_1}{W_2 - W_1}$

(Density of water is taken to be unity).

The surfaee tension of each liquid is calculated. Once the values of surface tension are known, the parachor values can be calculated, as all the factors, viz., molecular weight, density and surface tension are known in the expression,

$$[P] = \frac{M \gamma^{1/4}}{D}$$

The atomic parachors of C, H and O can then be calculated, as explained in the theory of this experiment.

Result: The parachor values of methyl alcohol, ethyl alcohol and n-hexane are

..., ..., and ..., respectively, while the atomic parachors of C, H and O are ..., ...,

and ..., respectively.

Precautions:

  (i)   The stalagmometer should be held vertically.

(ii)   The stalagmometer should be absolutely clean from any greasy matter.

(iii) The rate of flow of the liquid should be about 15-20 drops per minute.

(iv)   The drops should be allowed to fall off from the stalagmometer tip under their own weight and should not be pushed away by the kinetic flow

3)Synthesis of Benzopinacol from benzophenone.

Theory: This experiment was designed to provide experience in preparing compounds using a two-step synthetic scheme. The first step is a light-catalyzed radical reaction and the second-step is an elimination/rearrangement reactions. The first step is the conversion of benzophenone to bezopinacol using a photochemical reaction. Photochemical reactions often involve free radical species which are compounds with an unpaired electron. Free radical compounds are very reactive and are involved in reactions such as hydrogen abstraction, radical coupling, and polymerizations. The second step of this reaction series is the acid catalyzed dehydration of benzopinacol followed by rearrangement to benzopinacolone. This reaction illustrates the concept of carbocation rearrangement to a more stable carbocation. In this reaction, a tertiary carbocation rearranges to a more stable carbocation.

Procedure: To a clean test tube were added benzophenone (2.0 g, 0.011 mol), 2-propanol (10 mL), and concentrated acetic acid (1 drop). The tube was then sealed with a parafilmcovered cork. The cork was secured to the test tube using more parafilm and wire. The test tube was taken to the roof of the chemistry building and exposed to sunlight for 5 days. The resulting crystalline product, benzopinacol, was collected and air dried using a Büchner funnel. The white crystalline product was analyzed.
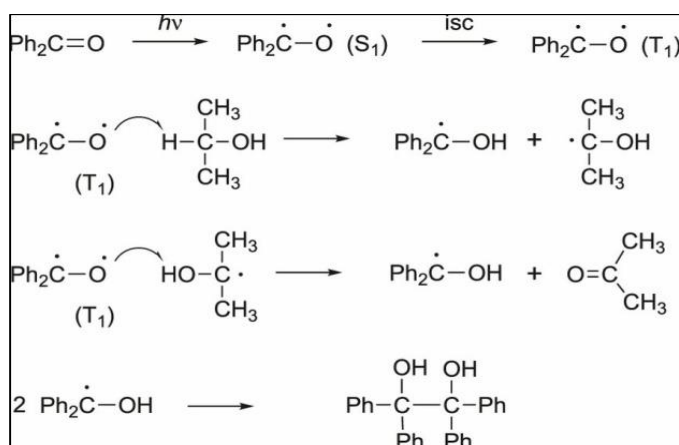
4) To determine the critical micelle concentration of soap by surface tension measurement.

Apparatus: Stalagmometer, pyknometer, three liquids, beakers etc.

Theory: Addition of soaps and other surface active agents lowers the surface tension of water to an appreciable extent. If the solution is not too dilute, the surface tension of the mixture varies directly with logarithm of concentration of the added substance. However, In case of soap and other substances undergoing association, the curve shows a break which corresponds to critical micelle concentration (CMC).

Procedure: Prepare a 0.2 M solution (stock solution) of potassium laurate (soap) of molecular weight 238.4 in distilled water. Then, by successive dilution of the stock solution, prepare 0.1, 0.05, 0.025, 0.02, 0.01, 0.001 M solution of potassium laurate. Determine the surface tension of each of the above solutions by drop fall method.

Calculations: Now plot a curve between surface tension (ordinate) and logarithm of the concentration of potassium laurate (abscissa). A break in the curve so obtained gives the critical micelle concentration (CMC) of the soap.

Result: The critical micelle concentration of soap is ……....

Precautions:

(i)   The stalagmometer should be held vertically.
(ii)   The stalagmometer should be absolutely clean from any greasy matter.
(iii)   The rate of flow of the liquid should be about 15-20 drops per minute.

The drops should be allowed to fall off from the stalagmometer tip under their own weight and should not be pushed away by the kinetic flow

5) Preparation of acetanilide by green chemistry

Theory: Acetylation of aromatic primary amines 1 is generally carried outby using acetic anhydride. Acetic acid in presence or absence of zinc dust can also be used for the conversion of aniline to acetanilide. However, the method requires high temperature, gas trap and 1.5 h refluxation. In view of the above and wide applicability of microwave irradiation in chemical reaction enhancement2, we report here the microwave-induced synthesis of anilides from some substituted primary aromatic amines using acetic acid and in the absence of zinc dust resulting higher yields, clear reaction products and easier workup

Procedure: A solution of amine (0.054 mol) in glacial acetic acid (10.0 ml) was taken in a 100-ml Erlenmeyer flask fitted with a funnel as a loose top. The reaction mixture was then irradiated under microwave (17-30% power) for 2 min. Then an interval of I min was allowed to avoid excessive evaporation of the solvent. This protocol was repeated until reaching an overall irradiation time of 10-20 min. It was then cooled and the resulting solution was poured in ice-cold water. The resulting solid was filtered, washed with water and recrystallized from alcohol.

6) **Study the inversion of cane sugar by potentiometer**

Apparatus: - Polarimeter, Sodium vapor lamp, Cane sugar, 100ml flasks, Distilled water.

Theory: - A beam of light whose vibrations occur only in one plane is said to be plane polarized light. The plane in which these vibrations occur is known as plane of vibrations of the polarized light while the plane perpendicular to it is called the plane of polarization.

The sugar solution has the power of rotating the plane polarized light through a certain angle and hence the sugar is said to be optically active. If the analyzer is turned to the right i.e. clockwise, the optically active substance is said to be dextrorotatory and if the analyzer is turned to the left i.e. anticlockwise, the optically active substance is said to be laevorotatory. The instrument of measuring angles of rotation is called POLARIMETER.

Factors affecting angle of rotation:- The angle through which a substance in the pure or dissolved state, rotates the plane of polarized light depends on

1. The nature of substance in respect of nature and number of asymmetry centers in its molecule.
2. Nature of solvent.
3. Concentration of solute.
4. Temperature of measurement.
5. Length of optical path in the substance.
6. Wavelength of light used.

The angle of optical rotation is approximately inversely proportional to the square of wavelength. The angle of optical rotation, α of a substance is related to the length of optical path , l conventionally measured in decimeters and density of substance , d in g/ml by following proportionality

$$\alpha = constant.l.c$$

The constant in this equation is characteristic of substance and depends on temperature of measurement, t and wavelength, λ of the light used. It is denoted by the symbol $[\alpha]^t_\lambda$ and is called Specific Rotation of substance at t^o for wavelength λ.

Specific rotation $[\alpha]^t_\lambda = \alpha/lc$

where: $[\alpha]$ = specific rotation; α = observed rotation; c = concentration of the solution of an enantiomer; l = length of the tube (Polarimeter tube The value of specific rotation is measured in units of angle degree ml per gram per decimeter. $(\deg\ cm^3 g^{-1} dm^{-1})$

For solutions, concentration is used in place of density.

$[\alpha]^t_\lambda = (\alpha/lc)*100$

Where c is concentration in units of g/ml.

Specific rotation $[\alpha]^t_\lambda = \alpha/lc$

For a pure substance in solution, if the color and path length are fixed and the specific rotation is known, the degree of rotation can be used to determine the concentration. In case of inversion of cane sugar the rate of reaction depends on the concentrations of sucrose, water and of hydrogen ion acts as a catalyst. The concentration of water may be regarded as constant when a large excess is used and H+ ion remains constant during the reaction. Under these conditions the reaction is first order in sucrose.

If A0 = the initial angular rotation, At = the value at time t, and the final value is A∞ then (A0-A∞) is proportional to the original concentration of sucrose and (At-A∞) is proportional to concentration of sucrose at time t. Substituting these values in the standard equation for a first order reaction we have

$$k = \frac{2.303}{t} log \left(\frac{A_0 - A_\infty}{A_t - A_\infty} \right)$$

Procedure: Take 50 ml each of 20% sugar solution and N-HCl in separate dry 100 ml conical flasks. Cork them loosely and put them into a thermostat at room temperature. Now set up a polarimeter. 50 ml of the acid solution is quickly mixed with 50 ml of the sugar solution and the stop watch is started. This mixing reduces the concentration of acid as well as of sugar to half their initial values. The polarimeter tube is rinsed with this solution and then filled with it. The angle of rotation and also the time at which the observation is taken, are recorded. Record subsequent readings after 5, 10, 15, 20 and 30 minutes and so on. The remaining mixed solution is placed again in the thermostat. The final reading can be taken after 48 hours with the mixture preserved in the thermostat. For quicker operation, the remaining mixture may be loosely corked and heated in a water bath at about 60° for about an hour taking care that loss of water by evaporation is avoided. Sugar solution may be charred or discoloured if the temperature of bath goes above 60 °C. The observations may be recorded as follows:

Data Recordings:

Time in minutes (t)	0	5	10	15	25	40	60	120	Final reading ∞
Rotationin degrees (r)									

Calculation: The change in rotation is proportional to the amount of sugar decomposed. Suppose r_o is the rotation at the beginning of the experiment, while, and are the rotations after time and final reading after complete inversion of cane-sugar respectively. Then: a, the initial concentration of sugar present is proportional to r_o-r_∞ and (a-x), the amount of sugar present at time t, is proportional to r_t-r_∞ Substituting these values corresponding to a and (a-x) in the first order integrated rate equation, we get

$$k = \frac{2.303}{t} log \left(\frac{A_0 - A_\infty}{A_t - A_\infty} \right)$$