SOPs/ INSTRUCTION MANUAL

1) To find the concentration of the given mixture, consisting of two liquids A and B, by viscosity measurement.

Apparatus: Ostwald's viscometer, thermostat, stop watch, thermometer stop watch, thermometer etc.

Theory: By plotting the values of viscosity of solutions against their concentrations, we get a curve from which the concentration of the unknown solution is determined. Curves of various forms are obtained and usually viscosity curves of simple solutions are sagged, i.e., fall below the straight line connecting the viscosities of their components.

Procedure: Prepare a number of solutions by mixing the two and B in different proportions. The solutions made up with 90%, 80%, 70%, 60%, 50%, 40%, 30%, 20%, 10%, of A by volume. The time of flow for each solution is noted by means of Ostwald's Viscometer as described in the preceding experiment. The time of flow for the unknown solution is also measured as usual. The density of each solution as well as the unknown solution is also determined, if we are to calculate the absolute viscosity of the liquid.

Observations: Room temperature = T ^{o}C

Percentage of components		Time of flow in seconds	Percentage of components		Time of flow in seconds
A	B		A	B	
90%	10%	….	40%	60%	….
80%	20%	….	30%	70%	….
70%	30%	….	20%	80%	….
60%	40%	….	10%	90%	….
50%	50%	….	Unknown solution	-	….

Calculations: A curve is plotted between the concentration of one component, say A and time of flow in seconds. We see that a straight line is obtained. The composition of the unknown solution is calculated locating and marking the point on straight line corresponding to its measured time of flow. A perpendicular is drawn from that point on the concentration axis from which the composition of the unknown solution can be read directly.

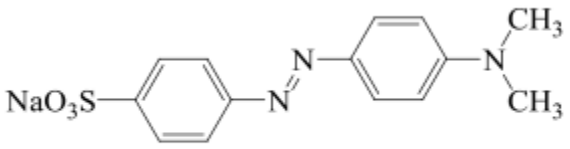
Result: The composition of the given mixture is ....% A and ....% B.

Precautions:

(i) The viscometer should be held vertical while performing the experiment.
(ii) The temperature should be maintained constant, as the viscosity is greatly influenced by temperature.
(iii) Same volume of water and the liquid should be taken in the viscometer.
(iv) The time of flow between the marks *x* and y in the viscometer should be about 2-3 minutes.

2)Preparation a pure sample of methyl orange from N, N-dimethyl aniline and sulphanilic acid

Theory: In this experiment the azo dye methyl orange is prepared by a electrophillic substitution with arenediazonium salts (diazo coupling).

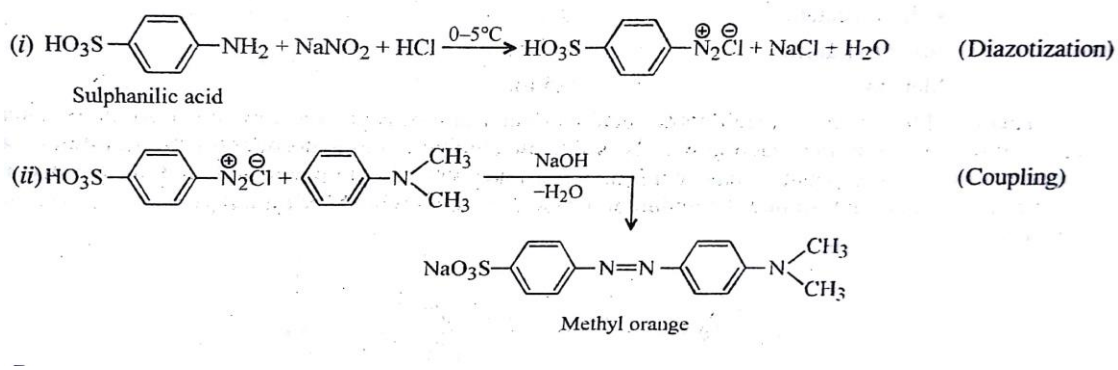


***p*-Dimethylamino-Azobenzenesulfonic Acid (Methyl Orange)**

Methyl orange is a pH indicator and due to its clear color change it is very often used in titrations. Methyl orange changes color at the pH of a mid-strength acid and is usually used in titrations for acids. Unlike a so called universal indicator, methyl orange does not have a full spectrum of color change, but has a sharper end point. Dyes are used to give colors to substances, especially fabrics. Chromopohores, functional groups that absorb light, give color to these dyes. The most common chromophores are azo, nitro, and carbonyl groups. Auxochromes , functional groups that increase the intensity of the color, are also important parts of dyes. The most common chromophores are hydroxyl, amino, sulfonate, and carboxylate groups.Azo dyes have a nitrogen to nitrogen double bond as their chromophore. These dyes are created by taking a diazonium salt and adding it to a strongly activated aromatic system.

In this experiment, you will synthesize methyl orange, an azo dye, by a diazonium coupling reaction with diazotized sulfanilic acid and N,N-dimethylaniline.

Chemical Reaction:-



Apparatus-100 ml conical flask, 250 ml beaker, ice bath, glass rod, Buchner funnel, water pump.

Chemicals

Sulphanilic acid = 7 g

Dimethyl aniline = 4 g

Anhydrous sodium carbonate = 2 g

Sodium nitrite = 2 g

Con. HCL = 8 ml

Dil. HCL = 20 ml

Sodium chloride = 10 g

10% NaOH solution = 50 ml

Procedure

Diazotized Sulfanilic Acid

1. Dissolve 0.29 g of anhydrous sodium carbonate in 12.5 mL of water. Use a 50 mL Erlenmeyer flask.

2. Add 1.0 g of sulfanilic acid monohydrate to the solution and heat it until it dissolves. A small amount of suspended material may render the solution cloudy.

3. Gravity-filter the still hot solution and rinse the used filter paper with a little (0.5 1.0 mL) hot water.

4. Cool the filtrate to room temperature, add 0.375 g of sodium nitrite, and stir until solution is complete.

5. Pour this mixture, while stirring, into a 100-mL beaker containing 8 mL of ice water to which 1.25 mL of concentrated hydrochloric acid have been added; add HCl dropwise to maintain a temperature of 0-5^{0}C.

The diazonium salt of sulfanilic acid should soon separate as a finely divided white precipitate. Keep this suspension cooled in an ice bath until it is to be used.

Methyl Orange

1. In a test tube, mix together 0.7 mL of dimethylaniline and 0.5 mL of glacial acetic acid.

2. Add this solution dropwise to the cooled suspension of diazotized sulfanilic acid in the 100-mL beaker. Stir the mixture vigorously. In a few minutes, a red precipitate of helianthin should form. Keep the mixture cooled in an ice bath for about 15minutes to ensure completion of the coupling reaction.

3. Add 7.5 mL of a 10% aqueous sodium hydroxide (NaOH) solution. Do this slowly and with stirring, as you continue to cool the beaker in an ice bath. Check with litmus or pH paper to make sure the solution is basic. If it is not, add more base.

4. Heat the mixture to boiling with a Bunsen burner for 10 to 15 minutes to dissolve most of the newly formed methyl orange. When all (or most of it) the dye is dissolved, add 2.5 g of sodium chloride, and cool the mixture in an ice bath. The methyl orange should recrystallize. Filter using a Buchner funnel.

5. To purify the product, transfer the filter cake and paper to a large beaker containing about 40 mL of boiling water. Maintain the solution at a gentle boil for a few minutes, stirring it constantly.

Note: Not all the dye will dissolve, but the salts with which it is contaminated will dissolve.

6. Remove the filter paper and allow the solution to cool to room temperature. Cool the mixture in an ice bath, and when it is cold, collect the product by vacuum filtration, using a Buchner funnel. Allow the product to dry, weight it, and calculate the percentage yield.

Result

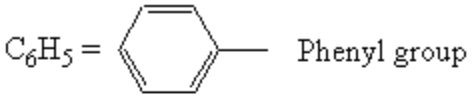
….g of methyl orange are obtained as deep reddish orange crystals.

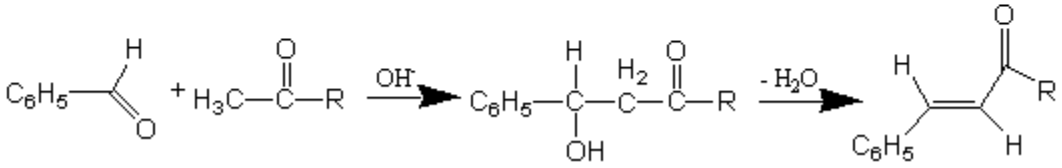
Expected yield = 12.30g

3) Preparation of Dibenzalacetone (1,5-Diphenyl-1,4-pentadien-3-one).

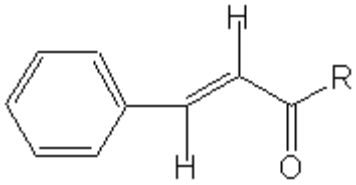
Chemicals Required: Benzaldehyde, acetone, Ethanolic sodium hydroxide.

Theory and Chemical Equation: An aldol condensation is a reaction that is named based on the type of product formed when two aldehydes (or ketones), in the presence of dilute base, yields a molecule having both aldehyde (ald-) and alcohol (-ol) functional groups. The aldol products are β-hydroxyaldehydea (or β-hydroxyketonea). This reaction is used extensively for the synthesis of new C-C bonds and to make larger organic molecules. In every case, the product results from the addition of one molecule of an aldehyde (or ketone) to a second aldehyde (or ketone) in such a way that the α-carbon (in the form of an enolate ion) of the first becomes attached to the carbonyl carbon of the second. This reaction is depicted below.



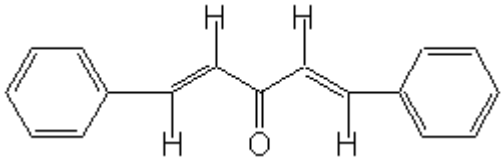


Although a β-hydroxyaldehyde (or a β-hydroxyketone) is produced in an aldol condensation, the ultimate product of these reactions (as shown above) is usually the α,β-unsaturated aldehyde (or ketone) and a separate molecule of water. Upon heating, the β-hydroxy aldehyde (or ketone) product of an aldol condensation easily undergoes dehydration to yield an α,β-unsaturated aldehyde (or ketone). Conjugation of the newly formed double bond with the carbonyl group (or of the benzene ring, as shown below) stabilizes the unsaturated product and provides the thermodynamic driving force for the dehydration process.



Showing the α,β-unsaturated Aldol Condensations product.

In the present case, crossed aldol condensation involving an aromatic aldehyde is referred to as a Claisen-Schmidt condensation. The Claisen-Schmidt condensation always involves dehydration of the mixed aldol condensation product to yield a chemical in which the double bond (produced during dehydration) is conjugated to both the aromatic ring and the carbonyl group. Because this aromatic aldehyde lacks α-hydrogens, only one product is can be formed, rather than a mixture of four different compounds, as long as the concentration of the second aldehyde is carefully controlled. We will prepare the dibenzalacetone: 1,5-diphenyl-1,4-pentadien-3-one. The equilibrium is shifted toward the product because the compound precipitates from the reaction mixture as it is formed.



1,5-diphenyl-1,4-pentadien-3-one

Procedure: In a 50-mL round-bottom flask, containing a curved magnetic stirring bar (clam shell shaped), attach a condenser column for reflux. Combine the benzaldehyde (2.40 mL; use pipetor) with 0.90 mL of acetone (reagent grade; use pipetor) in the round-bottom flask. To this reaction mixture, you will then need to add 25 mL of aqueous ethanolic sodium hydroxide (formulation is given below).

The reaction mixture is stirred at room temperature for 30 min. During this time a yellow solid precipitates from the reaction mixture. If a precipitate does not form, there is usually a heavy yellow oil, which can often be solidified by stirring it vigorously with a spatula or stirring rod. Be patient and let it stir for an hour more, if necessary. If no solid forms, then you will need to repeat the reaction from the beginning. However, as a last resort, you could draw off the basic aqueous layer (is this the upper or lower layer?) and stir the remaining oil vigorously, cooling it ice water (do not add this water to the reaction mixture), which usually allows for solid formation.

The crude product is collected using vacuum filtration in a Buchhner funnel. Any remaining solid in the reaction container can be washed out with a small quantity of cold water (water does not dissolve your product). Wash the precipitate with a small amount of water.

Transfer the solid product to a beaker and add about 20 mL of 5% acetic acid in ethanol. Stir the suspension, which will not dissolve, and filter again through a Buchner funnel. Wash the solid, in the funnel, with a small amount of cold ethanol.

The dry product from the previous lab period will be recrystallized by using about 20 mL of hot, boiling 95% ethanol. After dissolving, crystals should reform as soon as the solution cools to

room temperature (without the aid of ice). You should be careful to dissolve your solid dibenzalacetone in a minimum volume of the hot (boiling) 95% ethanol. Many students use too much solvent, thinking that the product must be dissolved in warm (not hot) 95% ethanol. If too much ethanol is used, and no crystals form, it will be necessary to reduce the volume to about 15 mL by evaporation. (Alternatively, ethyl acetate can be use as the recrystallization solvent if you choose.)

Collect the crystallized solid using filtration using a Buhner funnel as before. Since you are usually a volatile organic solvent for crystallization, it is not necessary to dry your sample overnight. Simply allow your solid material to dry in the Buhner funnel for 10-15 min, with vacuum on. This will allow air to continue to be drawn across your solid and evaporate your solvent.

Determine the mass of your dry product and determine percent yield.

4) To prepare 5-nitrosalisylic acid from salicylic acid by Nitration reaction.

Chemicals Required: Salicylic acid -2 g, Acetic acid – 10 ml and Calcium nitrate tetrahydrate-3 g.

Chemical equation and theory: Nitration on salicylic acid occurs by placing a nitro group on the aromatic ring system via an electrophilic aromatic substitution reaction. Here calcium nitrate is used as the nitrating agent in presence of acetic acid. Two groups -CO_2H and -OH in salicylic acid complement each other since they both direct the entering nitro group to the 5^{th} position. The 5^{th} position and the 3^{rd} position are both electronically favored since the -CO_2H group is meta directing and the -OH group is ortho-para directing. The nitro group is attached at the 5^{th} position, and not at the 3^{rd} position, due to steric effects. We can also use anhydrous nitric acid or concentrated nitric acid and concentrated sulphuric acid as nitrating reagent.

***Reaction*:**

Mechanism:

1.

2.

3.

Procedure: 3 g of calcium nitrate tetrahydrate is dissolved in 10 ml of acetic acid within a 100 ml conical flask by gently heating on a water bath. 2 g of salicylic acid is added and the reaction mixture is heated on a boiling water bath (below 80°C) for few minutes. A dark red solution is formed. Then the dark- red colored reaction mixture is poured into a 100 ml beaker containing 20ml of ice cold water.

A turbid dark red colored solution forms which is kept in a refrigerator and after 4-5 hours yellow crystals of 4-Nitrosalicylic acid separates out. The crude product is filtered at the suction pump, washed with cold water and dried. Yellow precipitate of 4-Nitrosalicylic acid is obtained with yield of 1.32 g, m.p. 234°C.