C 142 (Expt. No. 5)

NAME : _____ BATCH : _____

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Synthesis of Methyl Orange

IUPAC name : 4-Dimethylaminoazobenzene - 4' – sulfonic acid sodium salt

AIM

To synthesize methyl orange (an azo dye) from sulfanilic acid and N,N-dimethylaniline, using a diazonium coupling reaction.

MATERIALS

Sulfanilic acid, Na_2CO_3, $NaNO_2$, concentrated HCl, N,N-dimethylaniline, glacial acetic acid, NaOH, NaCl.

PROCEDURE

1. Dissolve **1g** of sulfanilic acid in **12.5 ml** of 2.5% Na_2CO_3 solution in a 250 ml beaker. Do not warm the mixture, as this may decompose the Na_2CO_3. Test one drop of the solution to make sure it is alkaline. If not, add a small amount (1-2 ml) of Na_2CO_3 solution, and check the pH again.
2. Cool the solution to room temperature. Add **0.5 g** of $NaNO_2$, and stir.
3. Pour this solution, **slowly**, into a beaker containing crushed ice and **1 ml** of concentrated HCl.

   A white precipitate of the **diazonium salt** separates out. Keep this solution cold in the ice bath at all times. This now contains the diazonium salt, which will decompose if it becomes warm.

4. Add **1.25 ml** of the **supplied solution A** (containing 0.75 ml of N,N-dimethylaniline and 0.5 ml of glacial acetic acid, which results in the formation of the **dimethylaniline acetate salt).**
5. Add **this solution A** to the cold suspension of diazotized sulfanilic acid, **with constant stirring**. A dull reddish-purple mass should appear.

6.** **VERY SLOWLY** add **9 ml** of **2.0 M** NaOH solution, with **constant stirring,** to produce an **orange colored sodium salt.** Stir the solution and add **~ 5 g NaCl**.

7. Heat the mixture to boiling. Cool to room temperature, and then cool in an ice bath. **The dye separates out as orange crystals.**

8. Filter the crystals, wash with ethanol (**1 ml**). Dry the crystals.

9. **Weigh the recrystallized sample. Calculate the percent yield.**

****_Note_ :**

(a) The NaOH solution should be added, **a few ml** at a time.

(b) The addition should take 10 – 15 minutes.

(c) **The actual coupling does not occur until NaOH is added.**

(d) If NaOH is added too quickly, then free dimethylaniline will separate out as an oily phase, leaving an equivalent amount of the diazonium salt unreacted. This excess salt decomposes to brown tar on warming to room temperature, and thus contaminates the otherwise beautiful crystalline orange dye.

RESULTS

Yield = …….. g

% Yield = …….. %