**Exp.8: Preparation of Acetanilide**

**Principle:**

The freshly redistilled aniline, is almost a colorless oily liquid which being practically insoluble in water. Therefore, before carrying out the ‘acetylation’ aniline has got to be made soluble in the aqueous medium. It can be accomplished by adding requisite amount of concentrated HCL whereby the highly reactive amino function easily takes up a proton from the dissociation of HCL in water, get protonated to yield aniline hydrochloride that is water-soluble. Subsequently, the soluble form of aniline is reacted with acetic anhydride in the presence of sodium acetate. The acetate ion obtained from the hydrolysis of the salt (sodium acetate) helps to sustain the acetylation reaction in the forward direction to yield acetanilide completely.



Acetanilide

Equations:



**Uses:**

1. It possesses antipyretic and analgesic activities.

2. It is invariably used in the manufacture of other medicinal e.g., sulphonamide ;besides dyes.

3. It is also employed as a stabilizer for H2O2 solution.

4. It finds its application as an additive to cellulose ester varnishes.

**Chemicals Required:**

1. Aniline: 10 ml (Freshly redistilled to have almost a colourless product).

2. Acetic anhydride: 13 ml.

3. Sodium acetate (crystalline): 16.5 g

4. Concentrated Hydrochloric acid (12 N) : 9 ml.

**Procedure:**

**1.** Transfer 10 ml of aniline is a 500 ml beaker and add to it 9 ml of concentrated hydrochloric acid and 25 ml of distilled water. Stir the contents of the beaker thoroughly with a glass rod till the whole of aniline undergoes dissolution.

**2.** Dissolve in a separate 100 ml beaker 16.5 g of sodium acetate in 50 ml of distilled water.

**3.** To the clear solution of aniline (1), add 13 ml of acetic anhydride, in small lots at intervals, with constant vigorous stirring until a perfect homogeneous solution is obtained.

**4.** Immediately pour the solution obtained from (3) into the sodium acetate solution (2). Shake the contents thoroughly with the help of a glass rod and immerse the beaker containing the reactants in an ice-bath.

***5.*** Beautiful shining crystals of Acetanilide separate out which may be filtered at the Büchner funnel by applying suction, washed with enough cold water, squeeze out the excess of water by pressing with an inverted glass stopper. Transfer the crude product onto a watch glass with the aid of a stainless-steel spatula and finally dry it in an electric oven previously maintained at 80°C. The yield of crude acetanilide (mp 113–114°C) is approximately 12g.

**Test for anilides**

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| **Tafels Test** |  |
| To sample, add conc. H2SO4 and shake add pinch of potassium dichromate. A red or violet colour is obtained. Leave for a few minutes, the colour changes to green. | A red or violet colour |

**Notes:**

1. Warming might be necessary for completing the solution process.

2. Operations with aniline, hydrochloric acid, and acetic anhydride should be performed in a well-ventilated hood. Avoid the contact of these compounds with skin.

3. As the amount of acetic anhydride is equimolar to the aniline, use of good quality reagent is essential (the acetic anhydride can be slowly hydrolyzed to acetic acid by the air moisture in a not well-sealed bottle).

4. The product can partially precipitate from a cold reaction mixture.

5. Cooling can be completed with ice-water bath. Loss of a well performed recrystallization is about 20 %.