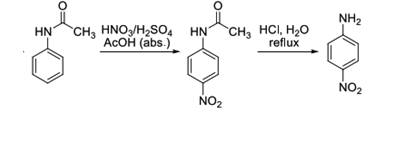
**EXP.3**

**Synthesis of *p*-nitroaniline from aniline based on protection/deprotection of amine group**

The nitration of aniline is difficult to carry out with nitrating mixture (a mixture of cone. H2SO4,and cone. HN03) since —NH2 group gets oxidized which is not required. So the amino group is first protected by acylation to form acetanilide which is then nitrated to give p-nitroacetanilide as a major product and o-nitroacetanilide as a minor product. Recrystallization from ethanol readily removes the more soluble ortho-compound and the pure p-nitroacetanilide is obtained. The chemical equation can be written as:



**Deprotection of amine group procedure:**

1. In a 50 mL Erlenmeyer flask mix the moist, crude *p*-nitroacetanilide with 3 mL of water and 4 mL of concentrated hydrochloric acid. Reflux the mixture gently for 15–20 minutes. The material gradually dissolves and an orange-colored solution is formed.
2. When the hydrolysis is completed add 6 mL of cold water and cool the mixture to room temperature. Crystals of the product may separate.
3. Pour the *p*-nitroaniline hydrochloride slowly, stirring thoroughly, into a mixture of 4 mL of concentrated aqueous ammonia, 15 mL of water, and 5–6 g of chipped ice. The mixture must be distinctly alkaline at the time of the mixing; test with indicator papers, and add a little more ammonia if necessary.
4. Collect the orange-yellow precipitate of *p*-nitroaniline with suction and wash it with cold water. Recrystallize the product from a large volume of hot water; about 30 mL of water will be required per gram of material.
5. The yield is 0.5–0.8 g. Prepare a sample of the purified product.