

**Laboratory Task 1****12 points****Preparation of 2-iodobenzoic acid****[Approx. time: 1 hr]**

This laboratory task involves preparation of 2-iodobenzoic acid from 2-aminobenzoic acid. The procedure consists of diazotization of 2-aminobenzoic acid followed by reaction with KI (in H<sub>2</sub>SO<sub>4</sub>).

**Procedure**

- 1) Quantitatively transfer the given sample of solid 2-aminobenzoic acid into a 100 mL beaker placed in the ice-bath. Add 7.2 mL of H<sub>2</sub>SO<sub>4</sub> (2.6 M) (labelled H<sub>2</sub>SO<sub>4</sub>) and mix the contents thoroughly for 1 minute with the help of a glass rod. Cool the solution for 5 minutes.
- 2) Using a measuring cylinder, measure out 4.4 mL of supplied cooled NaNO<sub>2</sub> solution from the vial placed in the ice-bath.
- 3) With the help of a dropper, slowly add the cooled NaNO<sub>2</sub> solution to the acid solution with constant gentle stirring using a glass rod to obtain an almost clear solution (3-5 minutes).
- 4) Remove the beaker from the ice bath and then slowly add 9.4 mL of KI solution from the stoppered tube, with stirring.
- 5) Get hot water from the laboratory expert. Keep the beaker in hot water for 5 minutes.
- 6) Filter the crude product and wash it thoroughly with distilled water (10 mL). Collect the washings along with the main filtrate.
- 7) Neutralize the combined filtrate by gradually adding the given solid Na<sub>2</sub>CO<sub>3</sub> until effervescence ceases. Dispose of the filtrate in the appropriate plastic bucket.

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**Purification of the crude product**

Place the funnel containing the precipitate on a 100 mL conical flask. Pour about 15 to 20 mL of the supplied NaHCO<sub>3</sub> solution (using test tube) over the filter paper so as to dissolve the precipitate completely.

- 8) Add the supplied charcoal powder to the filtrate and mix it thoroughly. Filter the solution to remove charcoal.
- 9) Add dilute H<sub>2</sub>SO<sub>4</sub> gradually to the filtrate till effervescence ceases. Filter the purified product. Use 10 -15 mL distilled water to wash the precipitate. Keep the filter paper with the product on a watch glass.
- 10) Cover the product with the same funnel and hand over the product to the laboratory expert for drying (for a minimum of one hour).

Towards the end of the practical session have the product weighed by the laboratory expert and record the same.