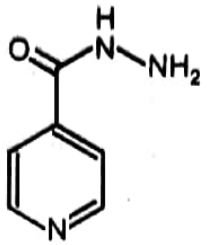
Experiment No. 7

Aim: To carry out the assay of Isonicotinic acid hydrazide (Isoniazid) Tablets IP.



Requirements:

Apparatus:

Volumetric flask, Measuring cylinder, Analytical balance, Weight box, Beaker, Burette, Conical flask and UV Spectrometer.

Chemicals:

Isoniazid tablets, Potassium bromate, Methyl red, Dimethyl formamide, Sodium carbonate, Sodium thiosulfate, Starch solution.

Principle:

The reaction is between isoniazid and potassium bromine, but solution of bromine is not stable. Therefore, a small amount of potassium bromide is added to the acidified solution of isoniazid, which is slowly titrated with potassium bromated. The reaction between $KBrO_3$, KBr, HCl, liberate bromine and this bromine oxidizes isoniazid. At the end point when HCl get deflected, changes colour from red to yellow.

Preparation and Standardization of standard solutions:

1. **Preparation of Potassium Bromate, 0.0167 M:**

Dissolve 2.783 gm of potassium bromate in sufficient water to produce 1000 ml.

2. **Preparation of Sodium Thiosulphate, xM:**

Dissolve 248x g of Sodium thiosulphate and 2x g of sodium carbonate in sufficient water to produce 1000 ml.

3. **Standardization of Potassium Bromate:**

Transfer the volume of about 30 ml of potassium bromate into a glass stoppered flask, add 3 ml of potassium iodide, following by 3 ml of hydrochloric acid. Allow to stand for five minutes at room temperature, then titrate the liberated iodine with standard sodium thiosulphate by using 3 ml of starch solution as an indicator. Collect for a blank on the same quantities of the same reagents. Each ml of 0.1 N sodium thiosulphate is equivalent to 0.002784 gm of potassium bromate.

Procedure:

1. Assay Method by Volumetric Analysis:

- Weigh and powder 20 tablets.
- Weigh accurately quantity of the powder equivalent to 0.4 g of isoniazid and dissolve in water, filter and wash the residue with sufficient water to produce 250 ml.
- Add 50 ml of water to 50 ml of the resulting solution, 20 ml of hydrochloric acid and 0.2 g of potassium bromide.
- Titrate slowly with 0.0167 M potassium bromate with continuous shaking using 0.05 ml of methyl red solution as indicator, until the red colour disappears.
- Each ml of 0.0167 M potassium bromate is equivalent to 0.003429 g of $C_6H_7N_3O$.

2. Assay Method by UV-Spectroscopic Method:

- Weigh about 80 mg of isoniazid and add 150 ml of dimethyl formamide, then add sufficient water to produce 500 ml.
- Dilute 5 ml to 100 ml with water and mix. Measure the absorbance of the resulting solution at the maximum at about 367 nm.
- Calculate the content of $C_8H_7N_3O_5$ taking 750 as the specific absorbance at 367 nm.

Result:

The given sample contains ..... mg of isoniazid.