

## **Aim of the experiment**

Systematic extraction, isolation, purification and identification of **non-metallic poison** by various analytical techniques.

## **Requirements**

- a) **Test sample:** Food curry (divided in four parts) spiked with sodium salts containing chloride, thiosulfate, nitrate and nitrite, respectively.
- b) **Materials:** Beakers, test-tubes, test-tube holder, test-tube, stands, Dropper, Pipette, Mortar-pestle, filter paper sheet.
- c) **Instruments:** Hot plate
- d) **Chemicals:** Hydrochloric acid, ferric chloride, sulfanilic acid, alpha-naphthylamine, zinc dust.

## **Theory and Principle**

- a. Non-metals are the elements which form negative ions by accepting or gaining electrons.
- b. The qualitative analysis of anions involves the detection and identification of radicals present in inorganic salts
- c. Chlorine is a halogen, an element belonging to Group 7 of periodic table (Chloride: halide ion). Halide ions are detected using silver nitrate solutions.
- d. Sodium thiosulfate with ferric chloride reacts to form ferrous thiosulfate (dark violet).
- e. 1-naphthylamine and sulfanilic acid is used for detection of nitrate and nitrite where reductase reduces nitrate to nitrite and reacts with sulfanilic acid to form diazonium salt. This salt on reaction with 1-naphthylamine forms an azo dye.

## **Procedure**

### **Extraction of poison:**

- a) Add 50 ml of hydrochloric acid (for digestion of sample) to the sample matrix in beaker along with equal amount of water.
- b) Keep the beaker(s) on hot plate to heat for 20-30 minutes (or till digestion was completed) at 110 degrees celsius.
- c) During heating HCL can be added if loss of volume is observed.
- d) The beaker(s) is then left to cool. Further, digested sample is filtered and filtrate can be used for anion analysis.

### **Reagent Preparation:**

a) Preparation of alpha-naphthylamine solution: 0.5 g  $\alpha$ -naphthylamine to be dissolved in 5N acetic acid by gently heating and made up to 100 ml. 5N acetic acid solution can be prepared by adding distilled water to 28.7 ml glacial acetic acid (17.4 N) to give a final volume of 100 ml. OR

Prepared by boiling 0.3 gm of 1-naphthylamine with 70 ml of water, filtering or decanting from the small residue and mixing with 30 ml of glacial acetic acid.

b) Preparation of sulfanilic acid solution: Prepared by dissolving 1 gm of sulfanilic acid in 30% warm acetic acid and volume made up to 100 ml.

**Sample Preparation:** Acidified solution of extract was prepared by adding few drops of acetic acid to be used for nitrate and nitrite test.

**Detection of anions:**

Test sample	Procedure	Observation	Inference
S1	A few drops acidified solution of the extracted sample was taken in a test-tube + 1 drop of sulfanilic acid + 1 drop of $\alpha$ -naphthylamine + few mg of zinc dust was added	Red colour will obtain.	Indicates presence of nitrate.
S2	A few drops acidified solution of the extracted sample was taken in a test-tube + 1 drop of sulfanilic acid + 1 drop of $\alpha$ -naphthylamine	Red colour will obtain.	Indicates presence of nitrite
S3	2 ml extracted sample was taken in a test-tube + Few drops of ferric chloride solution is added. On standing colour disappears.	Dark Violet Colour appears.	Indicates presence of thiosulphate.
S4	Extracted sample was taken in a test-tube + Silver nitrate solution (1:1) Precipitate is soluble in dilute nitric acid but soluble in dilute ammonia / potassium cyanide / sodium thiosulphate	White precipitate forms.	Indicates presence of chloride

**Conclusion:**

On the basis of observation, it can be concluded that the test sample \_\_\_\_\_ respectively.