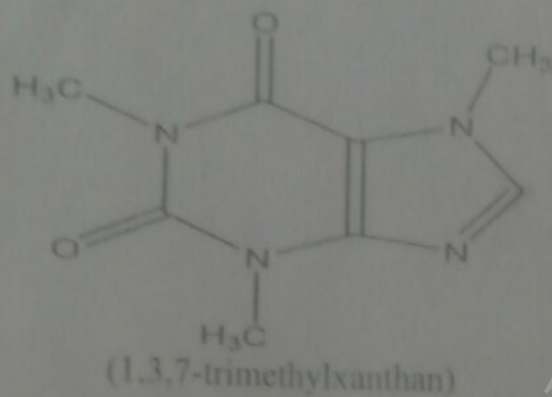


## Experiment No. 6

1. Objective : Isolation of caffeine from Tea leaves

2. Introduction : Caffeine occur in tea leaves in 1-4.8%, chemically it is 1,3,7-trimethylxanthan.



Being soluble in boiling water and using chloroform as a solvent. It can be crystallize as long silky needles from aqueous solution. It is present in tea and coffee extract and is known to process stimulant activity on central nervous system.

### 3. Requirements

- ❖ Tea leaves
- ❖ Chloroform
- ❖ Lead acetate
- ❖ Litharge
- ❖ Dilute sulphuric acid
- ❖ Animal charcoal

### 4. Procedure

#### Method - 1

- I. Take 50 gms of tea leaves in 25 ml of water in a 500 ml flask fitted with an water condenser. Boil it for 30 mins.
- II. Filter the hot solution and wash the residue with boiling water.
- III. Add basic lead acetate solution to the filtrate with constant stirring till complete precipitation is obtained.
- IV. Filter the hot solution and add dilute sulphuric acid till the whole of lead is removed as lead sulphate.
- V. Filter the lead sulphate precipitate by filtration again.
- VI. Add half gm of charcoal.
- VII. Concentrate the solution to half.
- VIII. Filtrate the resulting solution.
- IX. Extract caffeine from the filtrate by adding 75 ml of chloroform in a separating funnel (add 25 ml of chloroform 3 times).
- X. Distill of chloroform using distillation flask and water bath.

- XI. Dissolve the residue of distillation flask in small amount of distill water.  
XII. Cool the solution in ice bath long silky needle shape crystal are separated in water. Filter them out.

### 5. Observation

- > Appearance - Colorless, long silky needle shape
- > Yield; about 0.7 gm
- > Melting point 235 - 237 °C

### 6. Result

Yield of caffeine in given sample is ----- %

(Preparation of standard lead acetate solution: Basic lead acetate solution is obtained by boiling lead acetate solution with excess of litharge and filtering the resulting solution.)

### Method 2:

- i. Weigh exactly 6 gms of tea leaves, transfer it 100 ml conical flask.
- ii. Add 25 ml of dichloro methane and 5 ml of 0.2 N NaOH to it and stir the content for 10 mins and keep the flask aside for 5 mins.
- iii. Decant the upper NaOH layer and filter the organic layer over a funnel using filter paper. Add more dichloro methane if needed.
- iv. Wash the residue with dichloro methane (5 ml) four times.
- v. Transfer the filtrate along with the washing to a dry separating funnel. Keep it standing for 5 - 10 mins.
- vi. Separate the organic layer into a 100 ml beaker / china dish and evaporate the solvent on boiling water bath.
- vii. Complete evaporation of the solvent leaves behind a dry green powder (crude caffeine).

### Purification

- i. Dissolve green powder in least amount of propanol. Gently boil it.
- ii. Transfer the clear solution into 50 ml conical flask and add 2 ml of hexane.
- iii. Cool the solution in ice bath. Filter the crystalline precipitate (pure caffeine) and dry it in air.

### Observation

- > Appearance
- > Yield
- > MP

### Test for caffeine

Warm a few mg of caffeine with  $K_4[Fe(CN)_6]$  and  $HNO_3$  - a Prussian blue color is obtained.