

**ACHARYA N.G.RANGA AGRICULTURAL UNIVERSITY  
RAJENDRA NAGAR, HYDERABAD**



**PRACTICAL MANUAL**

**MANURES, FERTILIZERS AND AGRICULTURAL CHEMICALS**

**Course No. : SSAC 321**

**Credits: 2+1 = 3**

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**JANUARY 2012**

## **CERTIFICATE**

**Certified that this is a bonafide record of practical work done by**  
\_\_\_\_\_ **I.D.No.** \_\_\_\_\_ **in course No. : SSAC 321**  
**Manures, Fertilizers and Agrochemicals during the *First/ Second* Semester**  
**of 20\_\_ to 20\_\_ .**

**Signature of Course-in-charge**

### Particulars of the experiments conducted

<b>Expt. no.</b>	<b>Title of the experiment</b>	<b>Date of submission</b>	<b>Page no.</b>	<b>Signature</b>	<b>Remarks</b>
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## LIST OF THE EXPERIMENTS

1. Sampling of organic manures, fertilizers and pesticides for chemical analysis.
2. Quick tests for identification of known fertilizers.
3. Identification of anion and cation in unknown fertilizer.
4. Estimation of nitrogen in FYM and UREA.
5. Estimation of water soluble  $P_2O_5$  in single super phosphate by pembedon's method.
6. Estimation of potassium in MOP/SOP by flame photometer method.
7. Pesticide control order.
8. Study of pesticide formulations and physical tests for different formulations.
9. Iodometric titrations -determination of iodine present in the given sample solution.
10. Standardization of sodium thiosulphate solution by iodimetry.
11. Determination of purity of endosulfan by iodimetry.
12. Determination of purity of metasystox by acid –base neutralization method.
13. Determination of purity of malathion by iodometry.
14. Determination of copper content in copper oxychloride.
15. Determination of sulphur content in sulphur fungicide (elemental sulphur).
16. Compatibility of fertilizers with insecticides and fungicides.

**Expt. No.:**1

**Date:**

**SAMPLING OF ORGANIC MANURES, FERTILIZERS AND PESTICIDES  
FOR CHEMICAL ANALYSIS**

**Aim:** To collect a representative sample of manure / fertilizer / pesticide for chemical analysis.

**Objectives:**

1. To ensure the guaranty of nutrients present in manures and fertilizers.
2. To detect adulteration in chemical fertilizers / pesticides
3. To calculate the doses of fertilizers based on nutrient composition for different crops and soils.
4. To check the per cent active ingredient present in given pesticide
5. To check the pesticide residues present in soil samples.

With regard to analysis of samples it is done by different methods like volumetric, gravimetric, spectroscopic and chromatographic methods. Success of any of these methods depends on type of sample obtained for analysis and also storage of sample after collection is equally important. If the sample is stored under improper conditions, it may lead to changes in physical and chemical properties.

**I. Method of manure sample collection:**

The organic manure sample like FYM / poultry manure / vermicompost / oil cakes *etc.* are taken for analysis. The sample is drawn from the pit or heap covering all sides and it should be representative sample of the heap. If the sample is moist, it should be dried under shade.

**Sampling procedure:** Sampling should be done with sampling probe [Description of sample probe: Probe has dimensions of 3 feet length, hollow tube made of stainless steel or brass, a sharpened end at one end with a slught of 3-4 cm diameter up to a length of 15-20 cm.]. Sampling probe is an instrument used for sampling of manures. The probe is pushed across the manure pit and draw the representative samples and make it as a composite sample by adopting quartering method. Desired sample is taken in an air tight glass bottle. Bottle is kept in a cloth bag and insert a label with full details.

## II. Method of fertilizer / pesticide sample collection (Solid formulations)

The solid form of fertilizers (Ex: urea / DAP / SSP/ MOP etc.) or pesticides (granules / dusts / wettable powders) are marked in gunny or polythene bags. These bags should be stored properly as some of them are hygroscopic in nature and form lumps or clods and gives wrong results. The instruments used for the collection (sharp edge needle, plastic buckets etc.) should be free from contamination.

### Sampling of fertilizers can be done in two ways:

**1. Bulk sampling:** When sample is drawn from bulk material, there should not be lumps or clods. They should be pressed and entire material is spread on the hard cement floor and sample is drawn at random, mixed thoroughly by quartering method, stored in clean, air tight container.

### 2. Sampling from gunny bags / packages / lots:

Sampling should be done with sampling probe. This method is followed both for fertilizers and solid formulations of pesticides. It is not possible to draw samples from all the bags of lot. Sampling should be done rapidly by using a sampling probe [It is appropriate instrument to be used by quality control inspectors for collection of representative samples. Probe consists of stainless steel single tube with sharpened end with a length of 60 cm having a hollow tube with 5-8 cm with diameter 1.5 cm ].The probe is inserted diagonally and draw the sample from bag . The probe is inserted when the fertilizer is in good physical condition otherwise the entire material in bag is broken by wooden hammer and then the fertilizer is sampled.

**Drawing the samples from bag:** The sampling probe is used for collection of sample from one end or one corner to another and pushed diagonally and when filled with fertilizer, the probe is withdrawn and fertilizer is emptied in a container, iron polythene sheet or on a clean hard surface and made into one composite sample depending on lot size.

For straight fertilizers-500 grams, complex fertilizers -700 grams, micronutrients -1500 grams are drawn for test sample by quartering method. Test sample is divided in 4 parts.

1<sup>st</sup> part is taken to fertilizer control laboratory, Faridabad

2<sup>nd</sup> part –fertilizer control order

3<sup>rd</sup> -part remains with dealer

#### 4<sup>th</sup> part –quality control inspector

The number of samples to be drawn depends on the number of bags in each lot and it is as follows.

<b>Number of bags in each lot</b>	<b>Number of samples to be drawn</b>
< 10	1
11-100	2
101-200	3
201-400	4
401-600	5
601-800	6
801-1000	7
1001-1300	8
1301-1600	9
1601-2000	10

#### **Weight of one sample**

Approximate weight of one sample of fertilizer is as follows.

- i. For straight micro-nutrient fertilizers 100 g
- ii. For chelated micro-nutrient fertilizers  
and mixtures of micronutrients 50 g or the maximum  
packing size of  
similar quantity.
- iii. For other fertilizers and mixtures of fertilizers 400 g

#### **Preparation of Composite sample**

Spread the composite sample on a level, clean land surface, flatten it out and divide it into four equal parts. Remove any diagonally opposite parts. Mix the two remaining parts together to form a cone. Flatten out the cone and repeat the operation of quartering till a representative sample of required weight is obtained.

#### **Preparation of test sample and reference sample**

- i. The composite sample obtained above shall be spread out on a clean, hard surface and divided into three approximately equal portions each of the weight as specified above. Each of these samples shall constitute the test sample.

- ii. Each test sample shall be immediately transferred to a suitable container. The slip with detailed description may be put inside the sample bag. Each bag shall also be properly labelled.
- iii. Each test sample container shall then be sealed with the seals of the inspector, if possible seal of the manufacturer/importer/dealer or purchaser as the case may be, may also be affixed.
- iv. Out of three samples collected, one sample so sealed shall be sent to the Incharge of the Laboratory notified by the State Government. Another sample shall be given to the manufacturer or importer or dealer or the purchaser, as the case may be. The third sample shall be sent by the inspector to his next higher authority for keeping in safe custody. Any of the latter two samples may be sent for referee analysis.

### **III. Method of sample collection for liquid formulations:**

Generally this is followed for liquid formulations (emulsified concentrates) of pesticides.

In case of liquid formulations, these are packed in small containers of 100 mL / 250 mL / 500 mL / 1000 mL capacities. They are properly packed and sealed in large cartons. As per ISI specifications, liquid formulations are collected as given below.

- If box contains 3-15 bottles, select 3 bottles at random
- If box contains 16-40 bottles, select 4 bottles at random
- If box contains 41-65 bottles, select 5 bottles at random
- If box contains 66-110 bottles, select 10 bottles at random

After selection of bottles, they are neatly packed, labeled properly and sent to the nearest pesticide testing laboratory for analysis.

Information should be enclosed to pesticide or fertilizer sample, which contains the following information.

1. Common Name :
2. Trade Name :
3. Company manufacturing pesticide / fertilizer:
4. Date of manufacturing :
5. Per cent nutrient (or) :  
Per cent active ingredient :
6. Lot No / Batch No. :

7. Name of the dealer / manufacturer:
8. Date of sample collection :
9. Name of the person who collected sample :
10. Signature of dealer / manufacturer :
11. Signature of collected person / inspector :

Among the three samples packed one part of the sample is sent for analysis to nearest pesticide / fertilizer testing laboratory for analysis. The second part of the sample is given to the dealer or manufacturer from whom the sample was collected. The third part is retained by the inspection / person who collected the sample for producing in the court in case of any legal problem.

The sample received by the laboratory is given a code number and is entered in a register with the information present in information sheet. As far as possible the samples are to be analyzed immediately. Otherwise, the sample is stored in a refrigerator. Just before analysis, the sample is kept in over at 4 °C and is brought back to room temperature for analysis. After analysis, the report is signed by the analyst and sent in duplicate to the person who collected the sample / inspector for taking necessary action.

**General precautions in drawing sample:**

1. Sample should not be taken from place exposed to sun
2. Sampling instrument as well as materials should be free from contamination
3. To draw representative sample, sampled material is thoroughly mixed
4. The sample should be kept in dry, clean, glass bottle or screw capped polythene bottles or thick walled polythene bags which may accommodate 400-600 grams of material
5. The bottles are properly sealed and inserted in a cloth bags and it is supported with a label.

**Terminology used in fertilizer sampling:**

**Consignment:** This term is used consisting of different kinds of fertilizers namely urea, SSP, DAP etc., which will have maximum capacity of 2000 Metric tones.

**Lot:** Lot consists of stacked material of one kind namely urea. Maximum size of lot is 2000 bags or 40 Metric tones.

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**Expt. No. : 2**

**Date:**

## **QUICK TESTS FOR IDENTIFICATION OF KNOWN FERTILIZERS.**

The radicals  $\text{NH}_4^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{++}$  that are present in fertilizers are known as basic radicals whereas  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{PO}_4^{3-}$  and  $\text{NO}_3^-$  are acid radicals. The basic radicals react with reagents, giving precipitates. The acid radicals may be classified into two categories.

**Class A:** Radicals can be identified by volatile products obtained on treating with acids.

**Class B:** Those identification dependent on the reaction in solution

### **Class A is divided into 2 categories**

- 1) Gases evolved with dilute acids: e.g.  $\text{CO}_3^{2-}$ ,  $\text{HCO}_3^-$ ,  $\text{SO}_3^-$ , sulphide ( $\text{S}^-$ ), thiosulphite ( $\text{S}_2\text{O}_3$ ), Nitrite ( $\text{NO}_2^-$ ) are the examples
- 2) Gases evolved with conc.  $\text{H}_2\text{SO}_4$ : e.g.  $\text{Cl}^-$ ,  $\text{Br}^-$  and  $\text{NO}_3^-$

### **Class B is divided into 2 categories**

- 1) The precipitation reactions in solution  $\text{SO}_4^{2-}$  and  $\text{PO}_4^{3-}$
- 2) Oxidation and reduction reactions in solution, eg. Manganate, chromate and permanganate

**Radical:** It is a group of atoms which maintains its identity in the chemical reaction.

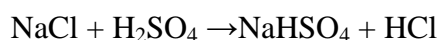
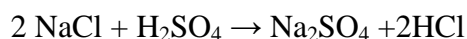
**Tests for identification of acid radicals:** The common acidic radicals found in fertilizers are  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{PO}_4^{3-}$  and  $\text{NO}_3^-$ .

## **I. CHLORIDES :**

1. **Action with heat:** Generally chloride fertilizers don't undergo any change. However, hydrated chlorides like  $\text{MgCl}_2$  and  $\text{ZnCl}_2$  undergo reaction by giving off HCl on heating.
2. **Test with Conc.  $\text{H}_2\text{SO}_4$ :** Take a little amount of chloride solution in a test tube and add conc.  $\text{H}_2\text{SO}_4$  and heat it. Reaction is vigorous even in cold condition. On warming decomposition is complete with the evolution of HCl. This is a colourless gas.

It gives fumes with moist air which is recognized when blue litmus paper is kept at the mouth of the test tube and it turns to red. A glass rod dipped in ammonia when

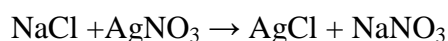
exposed at the mouth of the test tube, dense white fumes of  $\text{NH}_4\text{Cl}$  are formed which have pungent odour



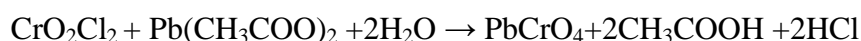
- 3. Test with  $\text{MnO}_2$  and conc.  $\text{H}_2\text{SO}_4$ :** Take equal quantities of chloride salt and  $\text{MnO}_2$  in a test tube and mix them well. To it add con.  $\text{H}_2\text{SO}_4$  and warm it. By this reaction greenish yellow colour gas of chlorine. This is recognized by its pungent[suffocating] odour. It bleaches the moist litmus paper.



- 4. Action with  $\text{AgNO}_3$ :** To a solution of chloride, add  $\text{AgNO}_3$  and mix it well. A white curdy precipitate of  $\text{AgCl}$  is formed. It turns to violet when exposed to sunlight. This white precipitate insoluble in  $\text{HNO}_3$  and soluble in  $\text{NH}_3$ .



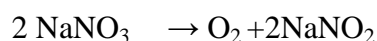
- 5. Chromyl chloride test:** Mix dry chloride fertilizer with an equal quantity of potassium dichromate in the dry test tube and few drops of conc.  $\text{H}_2\text{SO}_4$ . Gently warm the contents, reddish brown fumes of chromyl chloride is evolved. Dissolve the vapours in a test tube containing water. Acidify the brown solution with little acetic acid and lead acetate. Then yellow precipitate of lead chromate is formed.



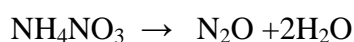
## II. NITRATES:

- 1. Action with heat:** Nature of the reaction depends on the cation

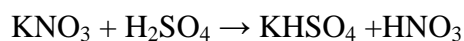
**i) Nitrates of alkali metals:** They yield  $\text{O}_2$  gas leaving only solid nitrite on heat



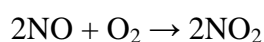
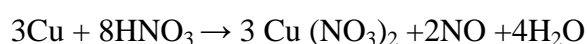
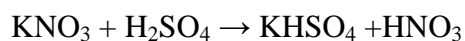
**ii) Ammonium nitrate:** On heating gives nitrous acid and steam



- 2. Action with conc.  $\text{H}_2\text{SO}_4$ :** To the salt add conc.  $\text{H}_2\text{SO}_4$  and heat it, reddish brown vapours with pungent odour is liberated. These reddish vapours are nothing but nitric acid vapours. They yield nitrates by decomposition on heating

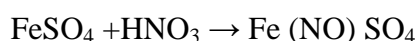
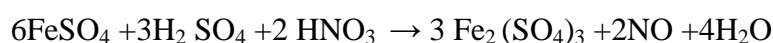
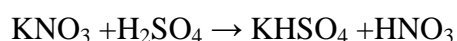


- 3. Action with Conc. H<sub>2</sub>SO<sub>4</sub> and Cu turnings :** Take a solid fertilizer sample in a test tube. To it add Cu turnings and conc.H<sub>2</sub>SO<sub>4</sub> and heat the contents. Reddish brown fumes of nitrates are released conspicuously and the solution turns to blue due to the formation of cupric nitrate.



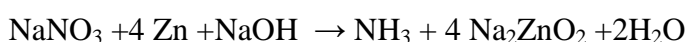
This test is also know as Cu test for nitrates

- 4. Brown ring test:** Add slowly conc.H<sub>2</sub>SO<sub>4</sub> along the sides of the test tube containing the saturated solution of ferrous sulphate and nitrate. A brown ring is formed at the junction of the two layers. The compound formed is ferrous nitroso sulphate, Fe(NO)SO<sub>4</sub> .



This brown compound is stable in cold condition. On warming it gets decomposed giving off nitric oxide. This test is not dependable, in the presence of iodides and bromides .

- 5. Diphenyl amine test :** Take few drops of nitrate solution in a watch glass, add one or two drops of diphenyl amine reagent , the solution changes to blue colour
- 6. Al or Zn with NaOH:** Boil the solution of nitrates with Zn and NaOH. Ammonia is liberated. Ammonia can be recognized by its smell or when moist red litmus paper is exposed to the vapours of ammonia turns to blue or glass rod dipped in HCl when exposed to the vapour dense white fumes of NH<sub>4</sub>Cl are formed.

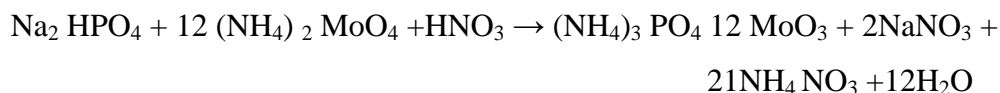


### III. SULPHATES:

- 1. Action with conc. H<sub>2</sub>SO<sub>4</sub> and heat :** No action
- 2. Action with BaCl<sub>2</sub>:** When a solution of sulphates is treated with BaCl<sub>2</sub> solution, a white precipitate of BaSO<sub>4</sub> will be formed which is insoluble in water.

#### IV. PHOSPHATES :

1. **Action of heat:** No reaction
2. **Action with ammonium molybdate:** Take few ml of the phosphate solution in a test tube and then add ammonium molybdate solution  $[(\text{NH}_4)_2\text{MoO}_4]$  followed by conc.  $\text{HNO}_3$  and warm the solution ( $50^\circ\text{C}$ ). On warming the solution turns into yellow colour and the canary yellow precipitate  $[(\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3]$  is formed. .



3. **Action with  $\text{AgNO}_3$ :** If phosphate reacts with  $\text{AgNO}_3$ , yellow precipitate of silver orthophosphate is formed. It is soluble in  $\text{NH}_3$  and dilute  $\text{HNO}_3$



This precipitate is soluble in  $\text{CH}_3\text{COOH}$  and mineral acid and insoluble in  $\text{NH}_3$ .

4. **Action with  $\text{MgSO}_4$  and  $\text{Mg}(\text{NO}_3)_2$  mixture:** Phosphate solution gives white crystalline precipitate magnesium ammonium phosphate with  $\text{MgSO}_4$  and  $(\text{MgNO}_3)_2$  mixture. The precipitate is soluble in mineral acids including acetic acid and insoluble in  $\text{NH}_3$ .
5. **Action with  $\text{FeCl}_3$  and  $\text{CH}_3\text{COONa}$  :** On addition of  $\text{FeCl}_3$  drop by drop to the phosphate solution and heated with sodium acetate or acetic acid which gives yellow precipitate of ferric phosphate which is soluble in dilute mineral acid but insoluble in dilute  $\text{CH}_3\text{COOH}$ .

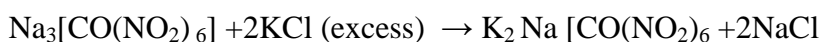
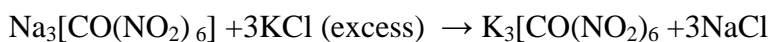
#### 2.2. Identification of basic radicals in fertilizers

##### I. POTASSIUM:

Many salts of potassium are commonly known and all are soluble in water viz.,  $\text{KCl}$ ,  $\text{K}_2\text{SO}_4$ ,  $\text{K}_2\text{Cr}_2\text{O}_7$ ,  $\text{KMnO}_4$ ,  $\text{KBr}$ ,  $\text{KI}$ ,  $\text{KCN}$  etc.,. Out of these salts  $\text{KCl}$  and  $\text{K}_2\text{SO}_4$  are widely used as fertilizers to supply K. Potassium hydrogen tartarate and potassium perchlorate are sparingly soluble in water. Prepare a solution of the given potassium salt ( $\text{KCl}$ ) in water and carryout the following tests.

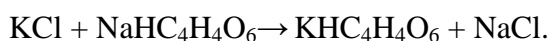
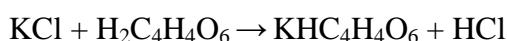
1. **Action of sodium cobaltinitrite solution:** Take 1 ml of conc.  $\text{KCl}$  solution in a test tube and add concentrated sodium cobalt nitrite solution. Shake the contents and a yellow orange precipitate of potassium cobaltinitrite is formed, which is insoluble in

dilute acetic acid. The precipitate is formed slowly in dilute solutions hence concentrated solutions should be used. Warming hastens the precipitation.



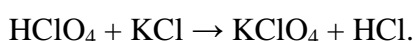
As salts of ammonia yield similar results, it is essential to eliminate ammonium salts prior to a test for potassium.

- 2. Action with tartaric acid:** A little solution of potassium salt and equal quantity of tartaric acid is added to a test tube. A white crystalline precipitate of potassium acid tartarate *i.e.* potassium hydrogen tartarate is formed. Take concentrated solutions the precipitation is soluble in mineral acids and alkalies, but insoluble in 50 per cent alcohol. The test is similar in the case of ammonium salts and hence they need elimination.



The use of sodium hydrogen tartarate solution (saturated) is preferable to tartaric acid. Precipitate formation is promoted by vigorous shaking.

- 3. Action with perchloric acid:** To a concentrated solution of potassium salt, add a little quantity of perchloric acid. A white crystalline precipitate of potassium perchlorate is formed



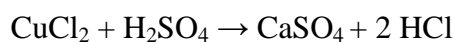
This alcoholic solution should not be heated as a danger explosion may result.

- 4. Action with  $\text{AgNO}_3$  and solid sodium cobaltinitrite:** To a little quantity of potassium salt add a drop of 0.05 per cent  $\text{AgNO}_3$  and a small quantity of sodium cobaltinitrite are added when a yellow precipitate of dipotassium silver hexanitrocobaltate  $\text{K}_2\text{AgCo}(\text{NO}_2)_6$  is obtained.

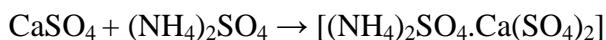
## II. CALCIUM

The calcium salts used as a fertilizer are super phosphate, dicalcium phosphate, calcium ammonium nitrate and as amendments of gypsum, lime *etc.* The nitrates and chlorides of calcium are soluble in water. Carbonates are more soluble in dilute acids while sulphates of calcium are sparingly soluble in water.

- 1. Action with dilute  $\text{H}_2\text{SO}_4$ :** To a small volume of calcium salt solution add equal quantity of dilute  $\text{H}_2\text{SO}_4$  when a white precipitate of calcium sulphate is formed.

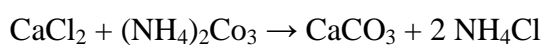


The precipitate is appreciably soluble in water and more so in acids. It is also soluble in hot ammonium sulphate solution due to the formation of a complex salt.

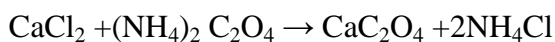


**2. Action with CaSO<sub>4</sub> salt:** Add to a solution of Ca salt- calcium sulphate solution produces no precipitate even on heating.

**3. Action with ammonium carbonate solution:** To the small quantity of ammonium carbonate. A white amorphous precipitate of CaCO<sub>3</sub> changes to crystalline on boiling, soluble in warm acid. The precipitate is also soluble in water containing carbonic acid in excess owing to the formation of soluble bicarbonate.

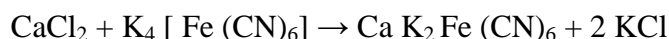


**5. Action with ammonium oxalate solution:-** Add a little of ammonium oxalate to a solution of calcium salt a white precipitate of calcium oxalate formed readily from the concentrate solution and slowly from dilute solutions . Precipitation is facilitated by addition of NH<sub>4</sub> OH.



This precipitation is soluble in mineral acids.

**5. Action with potassium ferrocyanide solution:** To a little quantity of calcium salt solution, add excess quantity of K-ferrocyanide, a yellow precipitate of calcium potassium ferrocyanide is formed. The sensitivity of the test is enhanced by the addition of an excess of a solution of NH<sub>4</sub>Cl.



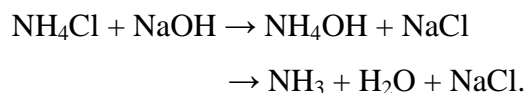
### III. AMMONIUM

The common salts of ammonium are the chloride, sulphate, nitrate and phosphate, all of which are used as fertilizers and are soluble in water. Use a solution of ammonium salt to carry out the following tests.

**1. Action of heat:** Heat some solid NH<sub>4</sub>Cl salt in a dry test tube and note the sublimation of the salt on the cooler parts of the test tube. *i.e.* ammonium gently sublimate on heating.

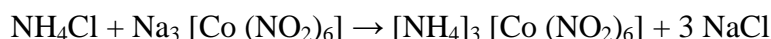
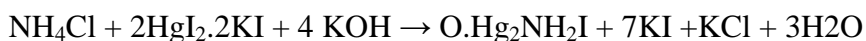
**2. Action with NaOH:** On slightly warming the solution after addition of NaOH, ammonia is evolved out, which can be identified by

- a. The characteristic smell
- b. The formation of white dense fumes when a glass rod dipped in dilute HCl is exposed to the vapour
- c. Moistened red litmus turns blue
- d. Filter paper moistened with mercurous nitrate turns black

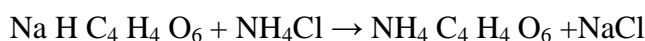


Black

**3. Test with Nessler's reagent:** To a little quantity of ammonium salt solution add a little amount of Nessler's reagent. A brown precipitate is formed even with very dilutions of ammonia. The test is extremely delicate and detects even traces of ammonia.



**4. Action with sodium hydrogen tartarate solution:-** A solution of sodium hydrogen tartarate is added to a solution of ammonium salt, then a white precipitate of ammonium acid tartarate similar to K but slightly more soluble than the corresponding K salt, from which it is by the evolution of ammonia when heated with a solution of NaOH.



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**Expt. No.:**3

**Date:**

**IDENTIFICATION OF ANION AND CATION IN UNKNOWN FERTILIZER**

**1. Tests for identification of anion or acid radicals in the given fertilizer.**

**Test 1: Action of heat:** Small quantity of fertilizer salt is taken in a test tube (dry) and gently heat on a flame.

- a) No change: May be  $\text{Cl}^-$ ,  $\text{SO}_4^-$
- b) Sublimation: May be nitrates

**Test 2: Action with conc.  $\text{H}_2\text{SO}_4$ :** A small quantity of fertilizer salt is taken in a test tube and add a little quantity of conc.  $\text{H}_2\text{SO}_4$

- a) Which gives colourless pungent smell gas, which in turn given dense fumes when glass rod dipped in ammonia solution indicates may be presence of chlorides.
- b) Reddish brown fumes indicates may be presence of nitrates

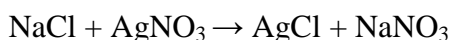
**Test 3: Action with  $\text{MnO}_2$  + Conc.  $\text{H}_2\text{SO}_4$ :** A small quantity of fertilizer salt is taken in a test tube to it add small quantity of  $\text{MnO}_2$  followed by conc.  $\text{H}_2\text{SO}_4$

- a) Greenish yellow colour less gas evolved, when exposed to sunlight indicate may be presence of chlorides
- b) Reddish brown fumes and the solution turned to green indicate may be presence of nitrates  
$$4\text{NaCl} + \text{MnO}_2 + 2\text{H}_2\text{SO}_4 \rightarrow \text{MnCl}_2 + \text{Cl}_2\uparrow + 2\text{H}_2\text{O} + 2\text{Na}_2\text{SO}_4$$

**Test 4: Action of Barium Chloride:** To fertilizer salt solution add excess of  $\text{BaCl}_2$  in a test tube .White precipitate is formed, indicates the may presence of sulphates

**Test 5: Action of  $\text{AgNO}_3$ :** To the salt solution add excess of  $\text{AgNO}_3$  solution.

- a) White precipitate : Presence of chlorides
- b) Yellow precipitate : Presence of phosphates



## II. Tests for identification of cation or basic radical in the given fertilizer

**Test 1: Action with heat:** A small quantity of fertilizer salt is taken in a test tube and heat under the flame →A white sublimation indicates the presence of ammonia ( $\text{NH}_4^+$ )

**Test 2: Action with conc.  $\text{H}_2\text{SO}_4$ :** A small quantity of fertilizer salt is taken in a test tube to it add conc. $\text{H}_2\text{SO}_4$  →A white precipitate of  $\text{CaSO}_4$  developed indicates presence of calcium (Ca)

**Test 3: Action with perchloric acid :**A small quantity of salt is taken in test tube and to it add 1 ml of perchloric acid . →**A white crystalline precipitate of potassium perchlorate indicates the presence of potassium (K)**

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**Expt. No.:**4

**Date:**

## **ESTIMATION OF NITROGEN IN FYM AND UREA**

### **4.1 Estimation of nitrogen in FYM**

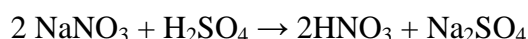
**Aim:** Estimation of total nitrogen in FYM by salicylic acid method

**Principle:**

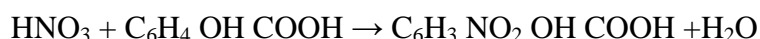
The farmyard manure contains nitrate nitrogen in addition to organic and ammoniacal form, which is digested with sulphuric acid in the presence of  $\text{CuSO}_4$ ,  $\text{K}_2\text{SO}_4$  and salicylic acid and sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3$ ). During digestion  $\text{CuSO}_4$  acts as a catalyst and  $\text{K}_2\text{SO}_4$  raises the boiling point of  $\text{H}_2\text{SO}_4$  and hastens the digestion process. By digestion the organic form of nitrogen is converted to ammoniacal form. Salicylic acid reacts with nitrates and forms nitro salicylic acid which is reduced to amino salicylic acid by sodium thiosulphate. Amino salicylic acid reacts with sulphuric acid forming ammonium sulphate. Thus by digestion all forms of nitrogen are converted to ammoniacal form and the ammoniacal form of N is distilled with Na OH and the liberated ammonia is made absorbed in boric acid solution and finally the total content of nitrogen is estimated by titrating with standard  $\text{H}_2\text{SO}_4$ .

**Reactions:**

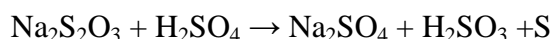
1. When FYM is treated with  $\text{H}_2\text{SO}_4$  the nitrate nitrogen is converted to  $\text{HNO}_3$



2.  $\text{HNO}_3$  so produced reacts with salicylic acid and forms nitrosalicylic acid



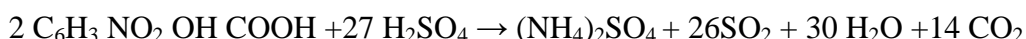
3. Sodium thiosulphate reacts with  $\text{H}_2\text{SO}_4$  and forms sulphuric acid.



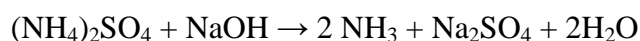
4. The sulphurous acid reacts with nitro salicylic acid and converts it to amino salicylic acid.



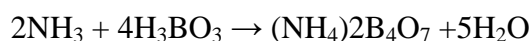
5. Amino salicylic acid on reaction with  $\text{H}_2\text{SO}_4$  gives  $(\text{NH}_4)_2\text{SO}_4$



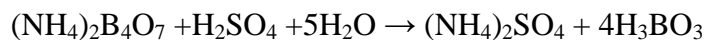
6.  $(\text{NH}_4)_2\text{SO}_4$  produced reacts with NaOH and gives ammonia



7.  $\text{NH}_3$  released is absorbed by boric acid to form ammonium tetra borate complex.



8. Ammonium tetra borate reacts with  $\text{H}_2\text{SO}_4$  and forms ammonium sulphate and boric acid.



**Reagents:**

1. **Conc.  $\text{H}_2\text{SO}_4$  of 98 per cent purity**
2. **Salicylic acid:** It fixes the nitrate nitrogen as nitro salicylic acid, thereby reduces the loss of nitrogen through volatilization.
3. **0.1N  $\text{H}_2\text{SO}_4$  :** Dissolve 2.67 mL of conc.  $\text{H}_2\text{SO}_4$  in 1000 mL of distilled water with the help of 1000 mL volumetric flask. Though it is a secondary standard solution it should not be used directly before its usage it should be standardized with same strength of primary standard solution like  $\text{Na}_2\text{CO}_3$
4. **0.1 N  $\text{Na}_2\text{CO}_3$  :** Dissolve 5.3 grams of  $\text{Na}_2\text{CO}_3$  salt in 1000 mL of distilled water with the help of 1000 mL of distilled water with the help of 1000 mL volumetric flask
5. **4 % Boric acid:** Dissolve 40 grams of boric acid powder in 1000 mL of distilled water in a reagent bottle by using measuring cylinder
6. **40% Na OH:** 40 grams of NaOH dissolved in 100 mL of distilled water.
7. **Sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3$ ):** It reduces the nitro salicylic acid to amino salicylic acid.
8. **Digestion mixture:** It contains **20 parts of  $\text{K}_2\text{SO}_4$ , 1 part of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$**  and 4 parts of  **$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$** . Copper sulphate acts as catalyst whereas  $\text{K}_2\text{SO}_4$  increases the boiling point of  $\text{H}_2\text{SO}_4$
9. **Mixed indicator:** Dissolve 0.66 mg methyl red and 0.99 mg of bromo-cresol green in 100 ml of 95 per cent ethyl alcohol with the help of 100 mL volumetric flask. Though it is an alcoholic based indicator it should be preserved under refrigeration
10. **Glass beads:** are added to prevent bumping during distillation process.
11. **Paraffin wax:** To avoid frothing during distillation process.

**Procedure:** Procedure involves 3 steps

- A. Digestion
- B. Distillation
- C. Titration

**A. Digestion:**

1. Weigh exactly 1 g of air dried FYM in a kjeldahl flask and moisten with about 20 mL of distilled water.
2. Shake well the contents of kjeldhal flask and add 25mL of conc.  $H_2SO_4$  along the sides of the flask, allow the contents for 24 hrs (overnight) for cold digestion.
3. Later add 10 g of digestion mixture, 1g of salicylic acid and 5 g sodium thiosulphate.
4. Heat the contents of the flask slowly till the carbon is oxidized. Later heat the contents of flask strongly ( $350-400^{\circ}C$ ) for 1-2 hours till a clear whitish or greenish solution is obtained.
5. The maintenance of temperature is very essential because, if the temperature is below  $350^{\circ}C$ , the distillation could not be completed .If it exceeds  $400^{\circ}C$  ,loss of nitrogen takes place .

**B. Distillation:**

1. Cool the flask to room temperature and transfer the contents of the kjeldahl flask quantitatively to distillation flask.
2. Dilute the sample by adding 200 mL of distilled water followed by addition of 2 to 3 glass beads and 10ml of paraffin wax and place it on the distillation set.
3. Keeps the beaker containing of 5 mL of 4% boric acid having mixed indicator under the delivery tube in such a way that the tip of the delivery tube should be immersed in the boric acid solution.
4. Add 100 mL of 40% NaOH solution and immediately connect the distillation flask to the condenser.
5. Heat the flask until all the ammonia is liberated (test with moist red litmus paper).
6. The completeness of distillation is checked by exposing moistened red litmus paper at the tip of the condenser. If the paper remains red, it indicates completeness of distillation or else continue the distillation.

**C. Titration :**

1. The ammonia borate is titrated against standard solution of 0.1N  $H_2SO_4$  unless blue / green colour changes to wine red colour. Note down the burette reading as titer value

**Model calculations:**

**Calculations**

Weight of FYM taken for digestion =1g  
Experimental titre value = x mL of 0.1 N H<sub>2</sub>SO<sub>4</sub>  
Blank titre value = y mL of 0.1 N H<sub>2</sub>SO<sub>4</sub>  
Actual titre value= x-y= z mL of 0.1 N H<sub>2</sub>SO<sub>4</sub>

**Model Calculations:**

**Factor derivation :**

1000mL of 1 N H<sub>2</sub>SO<sub>4</sub> =14 g of N  
1mL of 1N H<sub>2</sub>SO<sub>4</sub> = 14/1000 or 0.014 g of N  
1mL of 0.1 N H<sub>2</sub>SO<sub>4</sub> =0.014/10 = 0.0014 g of N  
1mL of 0.1N H<sub>2</sub>SO<sub>4</sub>= 0.0014 g N  
Z mL of 0.1N H<sub>2</sub>SO<sub>4</sub> =0.0014 x Z g N  
1 g of FYM contains = 0.0014 x Z  
100 g of FYM contains = 0.0014 x Z x 100/1 g N = A  
% N in FYM = A %

**Rating chart:**

	FYM	Vermicompost
Inferior	< 0.5	<1
Sufficient	0.5-0.75	1-2
Rich	>0.75	>2

**Result:** The given vermicompost contains ----- of total N. Hence it is said to be -----  
(inferior/sufficient/rich) in quality.



### Reagents:

- 1) **Conc. H<sub>2</sub>SO<sub>4</sub> of 98 per cent purity**
- 2) **Digestion mixture:** It is a mixture of two salts namely K<sub>2</sub>SO<sub>4</sub> and CuSO<sub>4</sub> mixed in the ratio of 10:1 and properly grinded by using mortar and pestle.
- 3) **4 % Boric acid:** Dissolve 40 grams of boric acid powder in 1000 mL of distilled water in a reagent bottle by using measuring cylinder.
- 4) **0.1N H<sub>2</sub>SO<sub>4</sub>:** Dissolve 2.67 ml of conc. H<sub>2</sub>SO<sub>4</sub> in 1000 ml of distilled water with the help of 1000 mL volumetric flask. Though it is a secondary standard solution it should not be used directly before its usage it should be standardized with same strength of primary standard solution like Na<sub>2</sub>CO<sub>3</sub>
- 5) **0.1 N Na<sub>2</sub>CO<sub>3</sub> :** Dissolve 5.3 grams of Na<sub>2</sub>CO<sub>3</sub> salt in 1000 mL of distilled water with the help of 1000 mL of distilled water with the help of 1000ml volumetric flask
- 6) **Mixed indicator :** Dissolve 0.66 mg methyl red and 0.99 mg of bromo- cresol green in 100 mL of 95 per cent ethyl alcohol with the help of 100 mL volumetric flask. Though it is an alcoholic based indicator it should be preserved under refrigeration

### Procedure:

The estimation of nitrogen involves 3 steps *viz.*,

#### A. Digestion ,

#### B. Distillation and

#### C. Titration.

#### A. Digestion :

- 1) Weigh exactly one gram of urea into digestion flask.
- 2) To it add 20-30 mL of concentrated H<sub>2</sub>SO<sub>4</sub>, allow the contents for cold digestion for 24 hours.
- 3) Heat the flask gently on the flame for about 5-10 minutes
- 4) Remove the flask from the flame and add 5 grams of digestion mixture.
- 5) The flask is heated continuously by using flame in the temperature ranging from 350 ± 50 °C for about 2-3 hours until clear bluish green / colourless material is retained. During digestion maintenance of temperature is important because when the temperature is above 400 °C, there is every chance of loss of nitrogen, if the temperature is below 300 °C digestion could not be completed.
- 6) Digestion mixture is transferred to 250 ml volumetric flask with the help of funnel with repeated washings and make up to volume with distilled water.

- 7) If there are any impurities, filter the solution through filter paper and filtrate collected into a beaker (digested material).

**B. Distillation:**

- 1) Pipette out 10 mL of filtrate into distillation flask, to it add 200-250 mL distilled water followed 4-5 glass beads and paraffin wax , to avoid bumping and frothing during distillation .
- 2) **Measure 30-35 mL of 4 % boric acid** mixed with 5 ml of mixed indicator ( wine red colour) in a 250 mL beaker and place the beaker at the tip of conductor and to see that tip of the condenser should immersed in boric acid solution to avoid loss of ammonia through volatilization.
- 3) Add approximate 25-30 mL of 40 % NaOH and close the top tightly.
- 4) Allow to water to circulate in the condenser.
- 5) Heat distillation flask gently and raise the temperature in the range  $350 \pm 50$  °C
- 6) Ammonia thus released during distillation and is passed through flask head and reach the condenser where gaseous ammonia (NH<sub>3</sub>) is condensed to NH<sub>4</sub>OH and is absorbed in boric acid as ammonia tetra borate.
- 7) The completeness of distillation is checked by exposing moistened red litmus paper at the tip of the condenser. If the paper remains red, it indicates completeness of distillation / else continues the distillation.

**C. Titration :**

- 1) The ammonia tetra borate is titrated against standard solution of 0.1N H<sub>2</sub>SO<sub>4</sub> until blue / green colour changes to wine red colour .Note down the burette reading as titer value

**Model calculation**

**Calculations:**

Weight of sample	= 1 gram
Volume made up	=250 mL
Aliquot taken	= 10 mL
Titer value	= Z mL

**Derivation of factor:**

1000mL of 1 N H<sub>2</sub>SO<sub>4</sub> =14 g of N

1mL of 1N H<sub>2</sub>SO<sub>4</sub> = 14/1000 or 0.014 g of N

1mL of 0.1 N H<sub>2</sub>SO<sub>4</sub> =0.014/10 = 0.0014 g of N

1mL of 0.1N H<sub>2</sub>SO<sub>4</sub>= 0.0014 g N

Z mL of 0.1N H<sub>2</sub>SO<sub>4</sub> =0.0014 x Z g N

10 mL of Urea solution contains =0.0014 x Z g N

250 mL of Urea solution contains= 0.0014 x Z x 250/10 g N

1 g of Urea sample contains = 0.0014 x Z x 250 /10 g N

100 g of Urea sample contains = 0.0014 x Z x 250 /10 x 100/1 g N = A

% N in Urea = A %

Purity % = Estimated content / Guaranteed content (46) x100

**Rating chart:**

Urea	
Inferior	< 95 %
Genuine	95-100 %
Adulterated	>100 %

**Result:** The given urea sample contains ----- of total N. Hence it is said to be -----  
(inferior/genuine / Adulterated ) in quality.

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**Expt No:5**

**Date:**

**ESTIMATION OF WATER SOLUBLE P<sub>2</sub>O<sub>5</sub> IN SINGLE SUPER PHOSPHATE  
BY PEMBERTON'S METHOD.**

**Theory:** According to specifications given by FCO, SSP contains 16% P<sub>2</sub>O<sub>5</sub>, 20%Ca and 12% S with maximum permissible water content 12%.

**Aim:** To determine water soluble phosphate content in SSP and express the result as percentage of P<sub>2</sub>O<sub>5</sub> and calculate its purity.

**Principle:** The phosphates that are present in SSP are precipitated as Ammonium phosphomolybdate (Yellow) with addition of ammonium molybdate in the presence of HNO<sub>3</sub>. The precipitate is washed with potassium nitrate solution in order to remove the acid present in it. The acid free precipitate is titrated against standard solution of NaOH unless until yellow colour disappears and run down excess of NaOH and note down the burette value. The excess NaOH not utilized for dissolving the precipitate is determined by back titrating with same strength of HNO<sub>3</sub>. The difference between alkali consumed and acid utilized during back titration gives the idea of actual amount of NaOH utilized for dissolving the precipitate.

**Reagents:**

1. **20% Ammonium molybdate solution:** Dissolve 200g of Ammonium molybdate salt in 400-500 mL of distilled water though it is insoluble in water in ordinary conditions. Heat the solution at a temperature range of 10-20°C. The solution is transferred to 1000 mL volumetric flask with the help of funnel and make up volume to mark with distilled water.
2. **Ammonia solution:**
3. **Solid ammonium nitrate salt**
4. **Red litmus paper**
5. **N/10 NaOH standard solution :**Dissolve 4g of NaOH flakes in 200-300mL of distilled water in a 500 ml beaker and transfer the solution to 1000 mL volumetric flask with the help of funnel and make up volume to mark with distilled water.
6. **N/10 standard solution of HNO<sub>3</sub>:** Mix 5.5 mL of conc. HNO<sub>3</sub> in 1000mL of distilled water with the help of 1000 mL volumetric flask.
7. **3% KNO<sub>3</sub> solution:** Dissolve 60 g of KNO<sub>3</sub> salt in 2000mL of distilled water with the help of reagent bottle by using measuring cylinder.

**Apparatus:** 250 mL borosil beaker, water bath, weighing balance, 1000 mL volumetric flasks - 2 , 100 mL volumetric flask -1, 500 mL beaker, 2500 mL capacity reagent bottle-1, 10 mL pipette-1, 25 mL burette-1, What man no. 42 filter paper and glass rod.

**Procedure:**

**Sample preparation:** Dissolve 1g of oven dried SSP fertilizer in a 500 mL beaker. To it add 200-300mL

of distilled water, stir the contents thoroughly and transfer the solution to 500 ml volumetric flask and make up volume to mark with distilled water. This procedure involves 3 steps i.e

**A. Precipitation**

**B. Filtration /Separation**

**C. Titration.**

**A. Precipitation:**

- Pipette out 10 ml of fertilizer solution into 250 ml beaker.
- To it add 50 ml of distilled water followed by a piece of red litmus paper.
- Add drop by drop  $\text{NH}_3$  solution till the colour of litmus paper turn to blue (alkaline condition)
- Brought back the media towards acidic with constant addition of  $\text{HNO}_3$  drop by drop (litmus paper turn to red)
- This is done to confirm the media of solution is acidic. The reason is precipitation is carried out only under acidic condition.
- At this stage add 5g of solid ( $\text{NH}_4\text{NO}_3$ ) ammonium nitrate with constant stirring.
- Place beaker on hot water bath maintaining the temperature 60-65 °C.
- Continue heating in this range till water bubbles observed. At this stage add 20 ml of precipitation mixture (11 ml of 20 % ammonium molybdate solution + 9 ml of Conc . $\text{HNO}_3$ ) to the contents of beaker with constant stirring yellow colour observed. Further continue for 5 min and remove beaker from hot water bath.

**B. Separation/ filtration:**

- Transfer the contents of beaker (precipitate) to a funnel arranged with Whatman no. 42 filter paper with repeated washings.

- The precipitate is constantly washed with 3%  $\text{KNO}_3$  solution unless until precipitate is free from acid.

#### **Test for acidity:**

Take few ml of 0.5 % NaOH in a test tube add a drop of phenolphthalein, pink colour develops and collect the filtrate into test tube , pink colour remains as it indicates free of acids / else continue washings.

#### **C. Titration:**

- Acid free precipitate along with filter paper is transferred to 250 mL beaker.
- To it add 50 mL of distilled water and a precipitate containing filtrate gets pulped by using glass rod (Yellow)
- Fill burette with N/10 Na OH and titrate the contents of beaker unless until yellow colour disappears and run excess of 5 mL Na OH.
- Note down burette value as x mL.
- Add 2 or 3 drops of phenolphthalein indicator which gives pink colour to the solution
- Fill burette with N/10  $\text{HNO}_3$  and titrate contents till pink colour disappears.
- Note down burette values as y mL.

#### **Discussion:**

- ✚ The purpose of maintaining temperature between 60-65°C during precipitation, when the temperature is below 60 °C, the precipitation could not complete, when it exceeds 65°C , molybdate acid is followed.
- ✚ The purpose of adding solid ammonium nitrate is to get granular precipitation.
- ✚ Washing of precipitate is done with 3%  $\text{KNO}_3$  to avoid the precipitate turn to colloidal stage.

#### **Calculations:**

Volume of N/10 NaOH utilized to dissolve the precipitate= x =13 mL

Volume of N/10  $\text{HNO}_3$  utilized for neutralizing excess of NaOH = y =3mL

Actual amount of N/10 NaOH utilized for dissolving ppt = x-y = z =10mL

#### **Data:**

Weight of fertilizer solution =1g

Volume made =500ml

Aliquot taken 10ml

**Factor:**

46 L of 1N NaOH dissolves 142 g of P<sub>2</sub>O<sub>5</sub>

1 L of 1N NaOH dissolves 142/46= 3.086g of P<sub>2</sub>O<sub>5</sub>

1mL of 0.1N NaOH dissolves 3.086 x 1/1000 x 1/10= 0.0003086 g of P<sub>2</sub>O<sub>5</sub>

(Z) 10 mL of NaOH dissolves = 10 x 0.0003086 g of P<sub>2</sub>O<sub>5</sub>

10 mL of fertilizer solution contains Z x 0.0003086 g of P<sub>2</sub>O<sub>5</sub>

$$=10 \times 0.0003086 \text{ g of P}_2\text{O}_5$$

500mL of fertilizer solution contains = Z x 0.0003086 x 500/10 g of P<sub>2</sub>O<sub>5</sub>

$$=10 \times 0.0003086 \times 500/10$$

1g of fertilizer sample contains = Z x 0.0003086 x 500/10 g of P<sub>2</sub>O<sub>5</sub>

$$10 \times 0.0003086 \times 500/10$$

100g of fertilizer sample contains = Z x 0.0003086 x 500/10 x 100g

$$=0.1543 \times 100$$

% purity of P<sub>2</sub>O<sub>5</sub>= 15.43

Purity %= estimated value / guaranteed content X100

$$=15.43/16 \times 100$$

$$=96.4\%$$

**Result:**

The given SSP fertilizer contains \_\_\_\_\_ P<sub>2</sub>O<sub>5</sub> with purity % is \_\_\_\_\_

Hence, the fertilizer is said to be genuine in quality.

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**Expt No:6**

**Date:**

### **ESTIMATION OF POTASSIUM IN MOP/SOP BY FLAME PHOTOMETER METHOD**

**Theory:** Potassium chloride [KCl: Muriate of potash) and Potassium sulphate ( $K_2SO_4$ : Sulphate of potash) are the important K fertilizers. According FCO, specifications KCl contains 58-60 %  $K_2O$  while  $K_2SO_4$  contains are 48 -50 %  $K_2O$ .

**Aim:** To determine the K content in MOP fertilizer and express the result as percentage of  $K_2O$  and calculate the purity per cent.

**Principle:**

When a solution containing fertilizer (MOP) is sprayed in the form of fine mist on the flame with the help of atomizer the fertilizer salt get discharge into respective atoms by absorbing thermal energy and electrons get excited. During excitation, the electrons jump from lower orbit to higher orbit after some time they get cooled and return back to its original orbit by emitting some radiation which is in the form of light energy. The radiation which is in the form of light energy is deflected on the columnating mirror and sent to prism where light energy gets dispersed. The dispersed radiation which is in the form of light energy is passed to filter .The filter allows only a type of radiation depending on the filter used [If K filter is used ,it allows only the radiation pertains to K]. This is called monochromatic radiation .The monochromatic radiation which is in the form of light energy is send to photocell, where light energy is converted to electrical energy .The electrical energy to be amplified and the magnitude of electrical energy is read out on a galvanometer.

**Apparatus:**

Flame photometer, which comprises of a) Galvanometer b) Atomizer c) flame source 1000 mL volumetric flask,100 mL volumetric flask,10 mL pipette,50 mL pipette,500 mL volumetric flask and 25 mL volumetric flask

**Preparation of standard curve for K:**

- a) **1000 ppm stock solution of K:** Dissolve 1.9067 grams of oven dried KCl salt in 1000 mL of distilled water with the help of 1000 mL volumetric flask.
- b) **Preparation of working standards :** Pipette out 0,2,4,6,8,10, mL of 1000 ppm stock solution of K into 100 mL volumetric flask and make up to the volume to the mark with distilled water which gives 0,20,40,60,80, and 100 ppm respectively.

These concentrations are fed to flame photometer and corresponding galvanometer readings are noted down, then a standard curve is drawn by taking the concentrations of K in ppm on X axis and galvanometer readings on Y axis .By joining any 3 points, a straight line is drawn with free hand .This is called standard curve for K .The sample reading is fitted in the standard curve and the concentration of K present in the fertilizer sample is calculated.

**Sample preparation:** Dissolve one (1) gram of oven dried MOP fertilizer sample is taken in 500-600ml of distilled water in a beaker. Dissolve it thoroughly filter it through ordinary filter paper. The filtrate is transferred to 1000 ml volumetric flask with the help of funnel and volume is made up to the mark with distilled water .From this 1000 ml solution, pipette out 50 ml into 500ml volumetric flask and make up the volume up to the mark . From this 500 ml solution, pipette out 10 ml into 25 ml volumetric flask and makeup volume up to the mark with distilled water. The solution is directly fed to the flame photometer and record the sample reading.

**Observations:**

S.No.	Volume of 1000ppm stock solution of K taken in 100 mL vol.flask	Working standards [ppm or ug/mL]	Galvanometer readings	sample value
1		0		
2		20		
3		40		
4		60		
5		80		
6		100		

**Calculations:**

Galvanometer reading = A

Concentration obtained from standard curve =  $A \mu\text{g mL}^{-1}$

Wt of sample taken = 1 gram

1 mL of fertilizer solution contains =  $A \mu\text{g}$

$$\begin{aligned}
25 \text{ mL of fertilizer solution contains} &= A \times 25 \mu\text{g} \\
10 \text{ mL of fertilizer solution contains} &= A \times 25 \mu\text{g} \\
500 \text{ mL fertilizer solution contains} &= A \times 25 \times 500/10 \\
50 \text{ mL fertilizer sample contains} &= \frac{A \times 25 \times 500}{10} \\
1000 \text{ mL contains} &= \frac{A \times 25 \times 500 \times 1000}{10 \times 50} \mu\text{g} \\
1 \text{ gram fertilizer contains} &= \frac{A \times 25 \times 500 \times 1000}{10 \times 50} \mu\text{g} \\
100 \text{ grams of fertilizer sample contains} &= \frac{A \times 25 \times 500 \times 1000 \times 100}{10 \times 50 \times 10^6} = B \\
\text{Percentage of K} &= B \\
\text{Percentage of K}_2\text{O} &= b \times 1.2 \\
\text{Purity \%} &= \frac{\text{Estimated value}}{\text{Guaranteed value (60)}} \times 100 \\
&= C
\end{aligned}$$

**Result:** The % purity of MOP is \_\_\_\_\_

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**Expt. No.:7**

**Date:**

## **Pesticide Control Order**

The pesticide act was passed in Parliament in 1968. Before introduction of insecticidal bill in the Parliament our country already had experienced more than 16 years in the production, import and use of pesticides. This is essential for quality control enforcement. This act came into effect on 1<sup>st</sup> October 1971 at Central Government level. In Andhra Pradesh it was enforced from 1<sup>st</sup> December 1972.

**Objectives:** To regulate the manufacture, sale, transport, distribution and use of insecticides with a view to prevent risk to human beings and animals.

Under the insecticide act, Government of India has constituted two committees.

1. Central Insecticide Board
2. Registration Committee

They have to advice Central and State Government on technical matters arising out of administration of this act and also to carry out the various functions assigned to it under this act. Central insecticide board consists of 24 members and a secretary.

Registration committee consists of a chairman and five members nominated by Government mainly with a view to register new insecticides and formulations after scrutinizing the claims made by the manufacturer and also to specify the precautions to be taken against poisoning through handling of insecticides and to carryout incidental matters arising out of the act.

**Registration of Insecticides:** Under Insecticide act, any person desired to import or manufacture an insecticide should apply to Registration Committee for registration of insecticide. Separate form should be submitted for registration of different insecticides and committee make detailed study regarding the claims made on the efficacy / safety of particular insecticide to animals and human beings. Committee gives a registration number to the manufacturer / dealer usually within 12 months from date of application. Some times it may extend up to 6 more months. Appeal against registration or cancellation of registration has to be done within 30 days of communication to Central Government in a prescribed form. Decision of Central Government on this matter shall be final.

## **State Functionaries**

Four state functionaries were appointed by the Government under this act.

1. Licensing Officer
2. Appellate Authority
3. Insecticide Inspector
4. Insecticide Analyst

**1. Licensing Officer:** State Government appoints licensing officer for issue of manufacturing license and to look after stocking, distribution, sale, and transport and plant pest control operation in fixed jurisdictions.

### **Granting of License:**

License will be granted to those who have registered their names on payment of fee. License period is specified and has to be renewed from time to time. If license is misused, the licensing officer has the right to cancel the registration and license. Any person, against decision of licensing officer can appeal to appellate authority within 30 days. The decision of appellate authority is final.

Any person desiring to import, manufacture, sell, stock, distribute and undertake commercial pest control measure with use of pesticides may make an application to the licensing authority for the grant of license.

Under this act, all the manufacturing units have to take license from the Directory of Agriculture or Commission of Agriculture of the concerned state where as the seller, stockist, dealer or distributor has to take license from the Joint Director of Agriculture of the concerned district.

The license for manufacturing a product will be issued after the said product is registered by the registration committee of Central Insecticides Board, Faridabad (Haryana). Manufacturer has to pay a registration fee of Rs 500 per each formulation subject to maximum of Rs. 7,500 and the license is valid for a period of one year. The seller or distributor has to apply for license along with the principles certificate issued by the manufacturer or formulator. The seller or distributor should also mention the place of selling or the place of distribution or place of stock in the application form. He has to pay a fee of Rs. 100 per formulation subject to a maximum of Rs. 1500 and license is valid for a period of one year.

**2. Appellate Authority:** State Government appoints appellate authority to consider appeals against the decisions of License Officer.

**3. Insecticide Inspector:** He is appointed by State / Central Government.

**Powers of Insecticide Inspector:**

- He has power to search any place if he has doubt about manufacturing and sale of insecticide against insecticide act.
- He has power to inspect, examine and make copies of registers and documents maintained by manufacturer or dealer or any other person concerned with the act.
- He can assume power of police officer under section 42 criminal procedure act 1973 for the purpose of enquiry of correct name and address of person from whom sample was collected.

**Duties of Insecticide Inspector:**

- Should inspect all the establishments pertaining to manufacture, sale and distribution not less than 3 times in a year.
- He should satisfy himself that the license is not mis utilised.
- He has to draw the sample and send for analysis.
- He has to investigate on any complaint received by him.
- He has to initiate prosecution in respect of person violating act.
- He should maintain proof of record of all visits made by him

**Procedure to be followed by Insecticide Inspector:** When an Insecticide Inspector seizes any record, register, document or stock under the provision of act, he shall inform the magistrate and take his orders regarding the custody of the stock. After obtaining the orders of magistrate, he can release the documents. The sample collected at random is divided into 3 equal parts, labeled with full details and sealed. One part is sent to analyst for analysis, one part is given to the person from whom the sample was collected and 3<sup>rd</sup> sample is retained by inspector to produce in the court in case of any legal problem.

**4. Insecticide Analyst:** He is appointed by Central / State Government by giving notification.

- Insecticide analyst has power to call for information and can take necessary action for examination of samples sent to him either from Insecticide Inspector or from persons from whom sample was obtained.
- The samples will be analyzed by analyst by following ISI approved methods and the report is signed and sent to concerned officers for intimation and action.

**Report of Insecticide Analyst:** The report is given within 2 months to insecticide inspector in duplicate in prescribed form duly signed. The inspector gives a copy of report to concerned person and other copy is kept in file for use in any prosecution in respect of sample. The state government may prohibit the sale, distribution and use of an insecticide which is likely to involve risk to human beings and animals for a period of 2 months. If the investigation is not completed the period may further extended to one more month.

**Prohibition of import and manufacture of certain pesticides:**

- A person should not import or manufacture a misbranded insecticide whose sale, distribution or use of which is prohibited.
- An insecticide which is not in accordance with insecticide act and violates the rules should not be imported or manufactured.

**Transport and storage of insecticide:** No insecticide should be transported or stored in contact with food stuffs and animal feed. If any insecticide is leaked during transport or storage, the concerned agency should take measures immediately to prevent poisoning or pollution of that insecticide with soil, water or air.

**Violations of the act:**

1. For the first time if the offence is made imprisonment for a period of 2 years or with a fine, which shall not be less than Rs 10,000 but which may extend to Rs 50,000 or with both
2. For the second and subsequent offence , imprisonment for a time which may extend for 3 years or with fine which should not be less than Rs 50,000 but which may extend to Rs 75,000 or with both .

**Insecticide Analysts and insecticide testing labs in A.P.:**

The Assistant Director of Agriculture is in-charge pesticide testing laboratory is designated as Insecticide Analyst who analyzes the pesticide samples and give the report in form No.IX within 30 days after receiving the sample. In Andhra Pradesh there are 4 such labs located in Rajendranagar, Guntur, Tadepalligudem and Ananthpur .

**Expt. No. :8**

**Date:**

## **STUDY OF PESTICIDE FORMULATIONS AND PHYSICAL TESTS FOR DIFFERENT FORMULATIONS**

### **8.1. Study of pesticide formulations**

Formulation is the treatment given to an active ingredient with the aim of improving its handling, storage, distribution, application and effectiveness. Pesticide formulation is the process of transforming pesticide chemical (ai) into a product, which can be applied effectively, safely and economically.

#### **Why formulations are prepared**

- Ease of application and handling
- To increase the effectiveness
- For uniform distribution
- To avoid phytotoxicity through overdosing
- To ensure safety to human being, animals, etc., while application and after use

**Types of pesticide formulations:** There are two types of pesticide formulations, they are,

#### **I. Solid formulations**

#### **II. Liquid formulations**

#### **III. Gaseous formulations**

#### **I. SOLID FORMULATIONS :**

##### **a) Dusts:**

These are solid formulations, which can be used without dilution. These are advantageous as these eliminate the use of water. Dusts are prepared by mixing the active ingredient with carriers / diluents and pulverized to a particle size of 1-40 microns. The best diluents are silica, talc, kaolin, attapulgate. These contain 0.1 to 25 % toxicant. These should be applied in the early hours of morning.

- Some dusts are prepared directly without any diluent Eg. Sodium fluoride
- Some dusts are prepared by mixing with active diluents Eg. Pyrethrum is mixed with rotenone or DDT to increase the toxicity of pyrethrum. Similarly rotenone is mixed with sulphur.

- Some dust are prepared by mixing with inert diluents like talc in order to cover more area or to reduce phytotoxicity or to improve the physical and chemical properties.  
Eg. BHC 50 % dust

#### **Advantages**

- Dusts are easy to apply
- Less phytotoxic
- They can be used where there is scarcity of water

#### **Disadvantages**

- Drifting is a problem
- Dermal toxicity and inhalation hazards are more
- Needs repeated application to control pests.

#### **b) Wettable Dispersible Powders (WDP):**

Wettable Powders are also fine particle size formulations. The size of the particles ranges from 5-50 $\mu$ . Wettable powders when dissolved in water should form uniform and stable suspension. These are used to prepare pastes. When these powders are used for spraying purpose, they are also known as water dispersible powders and are prepared by blending with a wetting agent and dispersible agent. Wettable powders should satisfy the following conditions.

- They should form uniform and stable suspension when mixed with water.
- They should not form cakes or lumps during storage.
- It should stick on the object where it is applied for a longer period.
- The particles should not be too heavy, so that they will not settle down. Also, the particles should not be too light, so that they will not float on the surface of water.

Wettable powders are subjected to physical tests like bulk density test, particle density test, sieve test, wettability test, suspensibility test etc.

Ex: Atrazine, Acephate, Carbendazim, Blitox

For eg. DDT 50 % WDP contains 50 % active ingredient, 30-40 % diluents, 4-5 % dispersing agent and 1-2 % wetting agent.

**c) Granules (G):** These are used widely for the control of soil inhabiting pests. These make the plants poisonous for sucking insects. Granular formulations are those formulations, in which the pesticide is impregnated in suitable carrier and then converted into granules. They are marketed in polythene or gunny bags. The granules

are smooth and spherical. The size of granules ranges from 1-2 mm in diameter. These formulations are generally very toxic. Most of the systemic insecticides are formulated as granules. Granules are of different sizes such as 8/12, 20/30, 30/60 etc., Granules are subjected to different physical tests like bulk density, particle density, sieve test etc. Granules can be subdivided into low volatile matter (LVM) and regular volatile materials (RVM) depending on the volatile matter content. They have the following physical properties.

<b>Physical property</b>	<b>LVM</b>	<b>RVM</b>
Moisture	1%	6%
Volatile matter	5%	15%
Water retention	180%	135%
Bulk Density	30-35 lbs	40-45 lbs
Specific heat	0.25	0.25
pH	7-9	5-6.5

The carrier materials for granules are kaolin, attapulgite and bentonite etc. Granules can be directly applied to soil. No costly equipment is required for application. There is no drift problem.

**Advantages:**

1. These release toxicant with control
2. Drifting is not possible during application
3. Reduce the toxic hazard to the farmer
4. Water is not required for application
5. Do not affect beneficial organisms
6. Can be handled and applied very easily.

Ex: Phorate, Carbofuran

**II. LIQUID FORMULATIONS:**

**a) Oil Concentrates :**

These contain a high percentage of active ingredient (a.i.) in an organic solvent like xylene, cyclohexane etc., they are to be diluted before use.

**b) Oil solutions:**

These are ready made liquid formulations which need not be diluted . The active ingredient is less than 5 per cent. Most of the house holds formulations are nothing but oil solutions .Eg. Baygon, Flit, Finit , Hit etc.,

**c) Aqueous concentrates:**

These contain high per cent active ingredient dissolved in water and only those chemicals which are highly soluble in water are formulated as aqueous concentrates.

Eg. Phosphamidan (Dimecron)

**c) Emulsifiable concentrates(EC):**

These are oil solutions of the technical grade insecticides with enough emulsifiers and adjuvant. An emulsion is a colloid of 'Liquid in liquid'. Emulsifiable concentrates are prepared by dissolving the active ingredient in desired quantities in suitable proportions of emulsifiers and sticking agents. Emulsifiers made it possible for the break up and suspension of oil droplets into microscopically small particles in water to form a fairly stable milky emulsion . On addition to water the emulsifier causes the oil to disperse immediately and uniformly throughout the aqueous emulsion stable for at least a day. Emulsified concentrates separates into two layers on storage. Hence, it has to be shaken properly before use. Emulsified concentrates are subjected to physical tests like emulsion stability test, cold test, heat test, flash point test, accelerated storage test etc.

**Ex:** Monocrotophos, Endosulfan, Chlorpyriphos, Malathion.

**Ultra Low Volume Formulations:** These formulations are concentrated solutions of toxicant dissolved in a solvent. An ideal ULV solvent is that which has good dissolving power, low volatility, low viscosity, non phytotoxic and compatible with most of the chemicals. The solvents used are a mixture of heavy aromatisation naphtha, dutrex, vegetable oils, glycols and mineral oils. These low volume sprays proved better in areas where insect control has to be done in vast areas.

**III.GASEOUS FORMULATIONS:****A) Aerosols:**

These are solid or liquid particles suspended in air with the help of propellants. Aerosols discharge a fine spray of liquid from self-dispersing disposable or refillable container . The toxicants are present in oil solvent together with a non-toxic propellant

gas (Freon or CO<sub>2</sub>). As the petroleum solvent is forced out of the container with the propellant, it is atomized. The size of these minute insecticide particles released from the container may range from 1-50 μm. Aerosols conveniently used for the control of in-door household pests.

Aerosol formulations released from thermal aerosol generators or foggers are also used outdoors in temperate climates when the wind velocity does not exceed 8 kmph. Orchard and plantation areas with bushes and trees not covered thoroughly with conventional sprayings are best treated by fog or mist generators.

#### **b) Fumigants:**

To prepare these formulations, the toxicant must be sufficiently volatile to produce a toxic concentration of vapour in a closed space. Insect control by fumigation is practiced in a number of fields like

1. Building fumigation
2. Product fumigation
3. Crop fumigation
4. Soil fumigation

Fumigants have the ability to penetrate as gas into inaccessible and hidden infestations unlike other types of formulations

#### **Definitions related to pesticide formulations**

1. **Adjuvants:** These are the materials that are added to a pesticide mixture to improve mixing and application.

Ex: Improves the wetting ability of a spray solution, control evaporation of spray droplets etc.

2. **Stickers:** These are the substances used to prevent the runoff of spray fluids when applied to crops. Many protective colloids, gelling agents like polyethylene polysulfide are used as stickers.
3. **Surfactants:** These are the chemicals which are used to reduce the internal tension between two liquids or between liquid and solids.
4. **Dry lubricants:** These are the materials used to increase the free flowing nature of the formulations (dusts). Materials like powdered graphite, talc, gypsum etc. are used.
5. **Emulsion:** An emulsion is a heterogeneous system consisting of at least one immiscible liquid intimately dispersed in another, in the form of droplets.
6. **Dispersibility:** The ability of the powdered concentrate to be suspended in water as a fine solid particle is known as dispersibility.

## 8.2 Physical tests for different pesticide formulations

Physical tests are those tests that are conducted for different formulations to know whether they are having all specifications as per ISI standards.

Different types of physical tests are as follows.

1. Sieve test: a) Dry sieving  
b) Wet sieving
2. Bulk Density test
3. Particle Density / Specific gravity test
4. Suspensibility test
5. Wettability test
6. Emulsion stability test
7. Cold test
8. Heat stability test
9. Flash point test
10. Accelerated storage test

Above 1-5 tests are normally conducted for solid formulations. 6-8 tests are conducted for liquid formulations and 9<sup>th</sup> & 10<sup>th</sup> tests are conducted both for solid and liquid formulations.

### 1. Sieve test:

**a) Dry sieving:** It is carried out for granular formulations. Normally 1 mm sieve is used. A known quantity of granular formulations is weighed and transferred to sieve and sample is sieved for 5 minutes. Amount of sample retained on the sieve is weighed and expressed in terms of percentage.

For X gm of sample sieved → Y gm is retained

For 100 gm of sample sieved → 100 x Y

----- gm

X

(More the retentivity on sieve, better the quality)

**b). Wet sieving:** This test is carried out for dusts and wettable powders. 0.1 mm sieve is used for this test. A known quantity of sample is weighed and transferred to 0.1 mm sieve and water is allowed to pass through the sieve for 10-15 minutes. At the end, the

amount of pesticide retained on the sieve is dried and weighed and expressed in percentage.

For X gm of sample sieved → Y gm is retained

For 100 gm of sample sieved →  $\frac{100 \times Y}{X}$  gm

(Lesser the retentivity on sieve, better the quality)

**2. Bulk density test:** A 100 mL measuring cylinder is taken & its empty weight is recorded ( $W_1$  g). Then the cylinder is filled with given solid formulation and its weight is recorded ( $W_2$  g). Bulk density of a given solid formulation is calculated as

$$\frac{W_2 - W_1}{100} \text{ g / cc}$$

**3. Particle Density / Specific gravity test:** Specific gravity is measured by using specific gravity bottle. It is the weight of given volume of pesticide to the weight of an equal volume of water. Empty weight of the specific gravity bottle is recorded ( $W_1$  g) and the bottle is filled up to the rim with pesticide (Solid formulation) and the weight of specific gravity bottle + pesticide has to be recorded ( $W_2$  g). Then the pesticide is removed and the bottle is washed with water. Then the bottle is filled with water and weight is recorded ( $W_3$  g).

Empty weight of specific gravity bottle	=	$W_1$ g
Weight of bottle + pesticide	=	$W_2$ g
Weight of bottle + water	=	$W_3$ g
Weight of pesticide	=	$W_2 - W_1$ g
Weight of water	=	$W_3 - W_1$ g

$$\text{Specific gravity of pesticide} = \frac{W_2 - W_1}{W_3 - W_1}$$

**4. Wettability test:** Usually wettability test is done for wettable powder formulations. Five grams of wettable powder is transferred to a 500 mL beaker containing 400 mL of distilled water and stir the contents with a glass rod. If the formulation is of good quality uniform suspension is obtained within 5 minutes.

**5. Suspensibility test:** Five grams of pesticide sample is taken and slurry is made with 40 mL of distilled water. This slurry is transferred to 500 mL measuring cylinder and

the volume is made up to 500 mL with distilled water. The suspension is shaken end over end. The measuring cylinder is kept aside for half an hour and the sample is drawn with a pipette from top 5 cm layer and is transferred to a 25 mL beaker (10 mL aliquot from pipette is transferred) whose empty weight is already recorded ( $W_1$  g). Then oven dry the sample and record the weight again ( $W_2$  g) and express in percentage. (More the percentage of retention of pesticide in a suspension, better the suspensibility and better the quality of pesticide).

**6. Emulsion stability test:** For the detection of emulsion stability about 10-15 beakers (500 mL capacity) are taken and 250 mL of distilled water is added to each of these beakers. Artificial hardening is created in water by adding different concentrations of salts by doubling the quantities. Then, to each of these beakers, add 2 mL of pesticide formulation and stir the contents with a glass rod. There is a particular salt concentration at which pesticide emulsion forms a precipitate at the bottom of the beaker. This point is considered as emulsion stability point, beyond which, the water with high salt concentration is not suitable for spraying purpose.

**7. Cold test:** Pesticide is subjected to varying ranges of temperatures ranging from 0 to 80°C. Then the pesticide is analyzed for its active ingredient. The temperature at which pesticide loses its active ingredient is taken as an index for cold test.

**8. Heat stability test:** The pesticide is subjected to increase in temperature ranging from 30 to 100°C for 2 days. Then the pesticide is analyzed for its active ingredient. The temperature at which the pesticide loses its active ingredient is taken as an index for heat stability test.

**9. Accelerated storage test:** This test is conducted both for liquid and solid formulations. The formulation is subjected to a pressure of 25 kg / sq. cm for half an hour. Then formulation is analyzed for its active ingredient. The optimum pressure at which the pesticide can be stored is known through this test.

**10. Flash point test:** Flash point test is carried out by using an instrument known as “Rotary Vacuum Flash Evaporator”. In this method, a known quantity of formulation is taken into a 250 mL conical flask and is connected to a suction pump. Vacuum is created by using suction (air is removed from the flask). Under vacuum conditions, sample is heated to a temperature at which the sample begins to boil or melt. This point is taken as an index for this test.

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**Expt. No.:**9

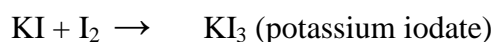
**Date:**

### **IODOMETRIC TITRATIONS -DETERMINATION OF IODINE PRESENT IN THE GIVEN SAMPLE SOLUTION.**

**Theory:** Titrations involving iodine are called Iodometric titrations. These are of two types.

1. **Iodometry:** This is the titration concerned with determination of iodine liberated during a chemical reaction and the liberated iodine is titrated against standard hypo solution which is a reducing agent.
2. **Iodimetry:** This is a titration concerned with using iodine solution directly, which is titrated against standard hypo solution

Iodine is a sublimable substance. The iodine vapours have a tendency to corrode the pans of balance. Iodine is sparingly soluble in water. To increase its solubility, potassium iodide is used, which combines with iodine and forms potassium iodate. This is highly unstable and it liberates I<sub>2</sub> into solution.



Starch is used as an indicator in iodometric titrations. Starch is insoluble in cold water and forms unstable suspension in cold water. Starch is hydrolyzed under alkaline and strongly acidic conditions. Therefore the solutions where starch has to be used as an indicator should be slightly acidic or neutral in reaction. Starch solution has to be prepared fresh just before use. Starch indicator is always added in the middle of titration (Iodometry) to avoid the formation of starch – iodine complex.

**Aim:** To find out the amount of iodine present in the given iodine solution or to find out the strength of given iodine solution.

**Principle:** A known volume of the given iodine solution is titrated against standard hypo solution till a pale yellow colour is noticed. At this stage a few drops of starch is added and titration is continued with hypo solution till the bluish violet colour disappears (in the presence of starch as an indicator). So, that the hypo solution is oxidized to sodium tetra thionate (Na<sub>2</sub>S<sub>4</sub>O<sub>6</sub>) and the iodine solution is reduced to sodium iodide (NaI). From the titer value using the standard factor the amount of iodine is calculated or the strength of iodine is determined by using the formula

$$V_1N_1 = V_2N_2$$

(1000 m L of 1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> reacts with 126.91 grams of I<sub>2</sub> or 127.00 grams (Approx.)



- 6) Continue the titration with hypo solution till the bluish violet colour disappears.
- 7) Note down the titer value and repeat the titration till two consecutive concurrent readings are obtained.
- 8) From the titer value using the standard factor 1mL of 0.1 N hypo= 0.0127 grams iodine, the amount of iodine and its strength is calculated.

**OBSERVATIONS:**

S. No.	Indicator used	Aliquo taken(mL)	Volume of 0.1N hypo consumed (mL)		
			Initial A	Final B	Actual (B- A)

**Calculations for determination of amount of iodine:**

Titer value : ZmL of 0.1 N hypo

**Factor:**

1 mL of 0.1 N Hypo = 0.0127 grams of iodine

Z mL of hypo contains = 0.0127 x Z grams of iodine

10 mL of iodine solution contains = 0.0127 x Z grams of iodine

1000 mL of iodine solution contains = 0.0127 x Z x 1000 grams of iodine= Y grams

10

= Y grams of iodine

**Calculations for determining strength of iodine**

$$N_1V_1 = N_2V_2$$

Normality iodine is calculated by using formula

$$N_1V_1 = N_2V_2$$

$N_1$  = Normality of iodine

$V_1$  = Volume of iodine

$N_2$  = Normality of hypo

$V_2$  = Volume of hypo

**Result:** The amount of iodine present in 1000 mL of iodine solution is----- g

and it's concentration is -----.

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**Expt. No.:10**

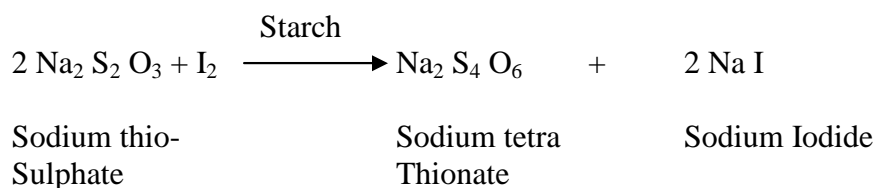
**Date:**

**STANDARDIZATION OF SODIUM THIOSULPHATE SOLUTION BY  
IODIMETRY**

**Theory:** The solubility of iodine is 0.335 grams per liter of water at 25 °C. In addition to the little solubility of iodine, aqueous solution of iodine has an appreciable vapor pressure and therefore concentration decreases slightly on account of volatilization. These two difficulties are overcome by dissolving iodine in aqueous solution of potassium iodide. The more is the concentration of potassium iodide, the greater is the solubility of iodine due to formation of triiodide ion. The resulting solution has much lower vapour pressure than the solution of iodine in pure water. Consequently the loss of iodine by volatilization is decreased. Never place the vessel containing iodine solution open as vapour pressure is still appreciable so keep it closed always except during the titration. When the iodine solution is treated with a reducing agent the free iodine reacts with the reducing agent and eventually the entire triiodide ion is decomposed. Therefore the solution behaves like free solution. AR grade (Analytical Reagent grade) resublimed iodine and iodate free potassium iodide should be used. The solution should be standardized against pure arsenious oxide or barium thiosulphate monohydrate or sodium thiosulphate solution, which has been iodine solution.

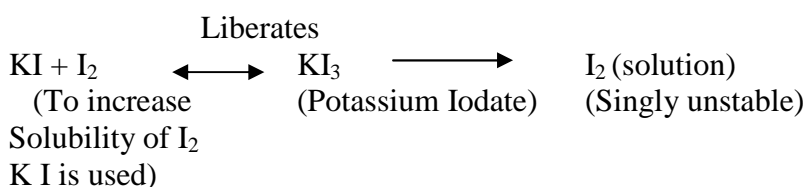
**Aim:** To standardize the sodium thiosulphate solution prepared in the laboratory by iodimetry.

**Principle:** The strength of given sodium thiosulphate solution is determined by titration with standard iodine solution using starch as indicator. During titration sodium thiosulphate reacts with iodine and produce sodium tetrathionate and sodium iodide.



**Reagents:**

1. **Standard 0.1N iodine solution:** Dissolve 20 grams of potassium iodide salt in 30-40 mL distilled water taken in 1 liter of volumetric flask. Weigh accurately 12.7 grams of AR grade resublimed iodine on a watch glass and transfer it by means of a funnel into the concentrated solution of potassium iodide. Insert the glass stopper and shake the contents well until all the iodine is dissolved. Make the volume to 1 liter by addition of distilled water which gives exactly 0.1 N iodine.



- 2. Standard Sodium thiosulphate solution (0.1N):** Dissolve 24.82 grams of sodium thiosulphate crystals in distilled water taken in 1 liter volumetric flask and make the volume to 1 liter with distilled water which gives approximately 0.1 N solution, the strength of which is to be determined by titrating against standard iodine solution
- 3. 5% starch solution:** Weigh exactly 5 grams of starch and make slurry with 30 mL distilled water. Boil 70 mL of distilled water and add this starch slurry in boiling water with stirring. See the starch is completely dissolved. This solution has to be prepared fresh.

**Procedure:** Transfer 10 mL of iodine solution to a 250 mL conical flask with the help of a pipette and dilute the contents by adding 20-30 mL distilled water. Titrate the contents of conical flask against sodium thiosulphate solution whose normality is to be standardized till a pale yellow colour appears. At this stage add few drops of starch as an indicator. Continue the titration against hypo till the bluish violet colour disappears. Note down the titer value and find out the strength of sodium thiosulphate solution using the formula

$$N_1 V_1 = N_2 V_2$$

While calculation if the strength of the solution is more, the solution should be diluted with distilled water. If the strength is less more hypo salt is to be added and bring the strength of sodium thiosulphate solution to the required normality.

**Result:** As the Normality of hypo is more than 0.1 N, it is diluted by adding 9.5 mL of distilled water and finally the normality of hypo is corrected to 0.1N

**Observations:** Prepared hypo solution has approximately 0.1 N then 0.1 N iodine solutions is used for titration with hypo solution.

Normality of hypo is calculated by using formula

$$N_1 V_1 = N_2 V_2$$

$$N_1 = 0.1 \times 10 / 9.9$$

$$N_1 = 0.1N$$

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Expt No:11

Date:

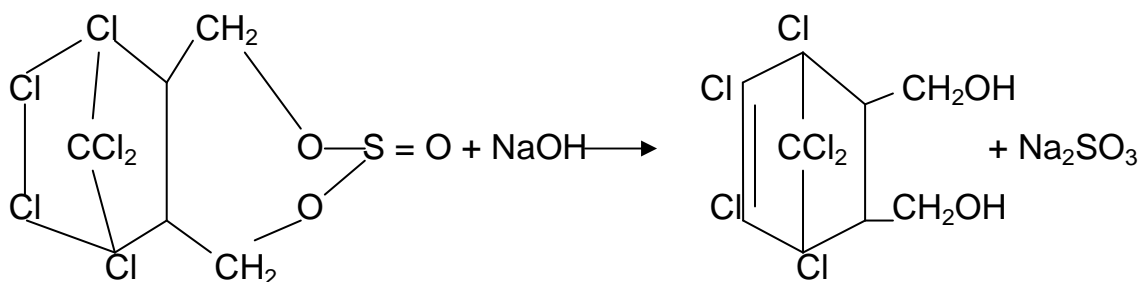
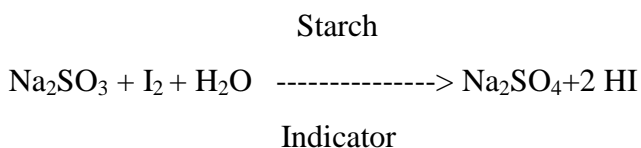
### DETERMINATION OF PURITY OF ENDOSULFAN BY IODIMETRY

**Theory:** Endosulfan is a widely used broad spectrum insecticide, which comes under cyclodiene compounds of organo chlorine insecticides. Technical grade endosulfan is a mixture of two isomers viz.,  $\alpha$  and  $\beta$  in 4:1 ratio. This chemical is stable under acidic conditions and compatible with other pesticides. This is rapidly hydrolyzed under alkaline conditions. It is formulated as 1% dust, 17.5 % and 35 % EC. Molecular weight of endosulfan is 407 grams [Equivalent weight: 203.5 g].

**Aim:** To find out the per cent purity of the given endosulfan commercial formulation by iodimetry.

**Principle:** A known quantity of Endosulfan is refluxed with methanolic-sodium hydroxide to form endosulfan alcohol and sodium sulphite [ $\text{Na}_2\text{SO}_3$ ]. The sodium sulphite, so formed is acidified and titrated against standard iodine solution using freshly prepared starch as an indicator till bluish violet colour is appeared. From the titer value using standard factor, the purity of endosulfan is calculated.

#### Reaction:



**Factor:** 1 mL of 0.1N iodine = 0.02035 grams of endosulfan

#### Reagents:

1. Methanol

2. NaOH Pellets
3. 1: 6 H<sub>2</sub>SO<sub>4</sub>: Dilute 14.70 mL of Conc. H<sub>2</sub>SO<sub>4</sub> to 100 mL with distilled water.
4. Phenolphthalein indicator: One gram of phenolphthalein in 100 mL of 75 % ethyl alcohol.
5. 0.1 N iodine solution: Weigh 20 grams of potassium iodide and dissolve in 40-50 mL of distilled water. Weigh 12.7 grams of I<sub>2</sub> and transfer to potassium iodide solution and stir the sample till completely dissolved. Transfer the solution to liter volumetric flask and make up the volume to 1 liter with distilled water . Keep the solution over night and store in amber coloured bottle [Photo sensitive ]
6. 5 % starch indicator: Weigh exactly 5 grams of starch and make slurry with 30 mL distilled water. Boil 70 mL of distilled water and add this starch slurry in boiling water with stirring. See the starch is completely dissolved .This solution has to be prepared fresh.

**Procedure:**

1. Weigh accurately 1 gram commercial formulation of given endosulfan 35 % EC by differential weighing and transfer it to a 250ml conical flask with the help of 25 ml of methanol in 2(or)3 installments
2. Add 20 pellets of Na OH to the flask and reflux the contents on a hot water bath for two hours for hydrolysis to takes place.
3. After refluxing cool the contents to room temperature and transfer the contents into 500 mL conical flasks with 50-60 mL distilled water.
4. Put one or two drops of phenolphthalein indicator when the contents turn to pink.
5. Neutralize the basicity of the contents by adding drop by drop 1:6 H<sub>2</sub> SO<sub>4</sub> until the pink colour disappears.
6. Add 0.5 mL more of 1:6 H<sub>2</sub> SO<sub>4</sub> to make the contents acidic.
7. Now put a few drops of starch as an indicator and titrate the contents against 0.1N iodine solution till a bluish violet colour appears.
8. Note down the titer value and calculate the purity of endosulfan using the standard factor i.e., 1 mL of 0.1N mL of iodine = 0.02035 grams of endosulfan.

**Observations:**

S. No.	Aliquot taken(mL)	Indicator used	Volume of 0.1N Iodine consumed (mL)		
			Initial A	Final B	Actual B- A

**Calculations:**

Weight of endosulfan taken = W grams

Titer value = Z ml of 0.1 N iodine solution

**Factor:**

1 mL of 0.1 N iodine = 0.02035 grams of a.i of Endosulfan

Z mL of 0.1 N iodine = 0.02035 x Z grams of a.i of Endosulfan

W grams of Endosulfan contains = 0.02035 x Z grams of a.i of Endosulfan

100 grams of Endosulfan contains =  $0.02035 \times Z \times \frac{100}{W}$  grams of a.i of Endosulfan

= Y

Percent purity = Y

**Result:** The a.i. present in 100 grams of Endosulfan of 35 % EC is -----

**Remarks:** As per IS I specifications, Endosulfan 35 % EC contains active ingredient in the range of 32.5 to 37.5 % to be acceptable for marketing.

**Conclusion:** The sample is Accepted / Rejected for marketing

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**Expt. No.:**12

**Date:**

**DETERMINATION OF PURITY OF METASYSTOX BY ACID –BASE  
NEUTRALIZATION METHOD**

**Theory:** **Metasystox** is a widely used broad spectrum systemic insecticide .It belongs to thiophosphoric acid derivative. It is also called as oxy-demeton methyl / Methyl demEton . Its molecular weight is 246. It is stable under acidic conditions. Hence to improve its shelf life, manufacturers add small amount of acid. This compound is hydrolyzed under alkaline conditions. It is marketed as 25% EC.

**Aim:** To determine per cent purity in given sample of metasystox 25% EC by acid base neutralization method.

**Principle:** Metasystox when reacted with NaOH (alkali) at room temperature, gets hydrolyzed to form sodium salt of dimethyl thiophosphoric acid and sulfoxide of ethyl thio ethanol. Amount of hydrolysis that took place is determined by back titrating the excess unused NaOH against standard 0.1N HCl using methyl red as indicator. After correcting for free acidity, per cent purity is determined by using the factor 1ml of 0.1 N NaOH reacts with 0.0246 g of Metasystox.

**Reagents:**

**1. Standard 0.1 N NaOH:** Weigh 4 g of NaOH, Dissolve in distilled water and make up the volume to 1 litre in volumetric flask.

**2. 0.1 N HCl:** Dissolve 8.6 mL of concentrated HCl in distilled water and make up the volume to one litre with distilled water.

Specific gravity =1.14

% purity = 37

Mol. Wt. = 36.5

Mass (Equivalent weight)

Volume = -----  
Sp. Gravity

$$V = \frac{36.5}{1.14} = 32 \text{ mL}$$

37 mL of pure acid is present in 100 mL of conc. HCl

32 mL of pure acid is present in

$$= \frac{32 \times 100}{37} = 86.5 \text{ mL of HCl}$$

86.5 mL of conc. HCl/ litre of distilled water = 1 N

For 0.1 N 8.6 ml HCl/litre of water

**3. One per cent methyl red indicator:** 1 g of methyl red is dissolved in 100 mL of 95% ethyl alcohol.

**Apparatus:** Burette, conical flask, pipette, beakers, volumetric flasks.

**Procedure:**

**A. Titration after hydrolysis:**

Weight accurately approximately 1 gram of metasytox by differential weighting and transfer it to a 250 mL conical flask with the help of 10 ml of 0.1N NaOH solution. Shake the contents vigorously and leave it like that for about 15 minutes for hydrolysis to take place. After 15 minutes dilute the contents by adding 15-20 mL distilled water. Put drop by drop methyl red indicator till the contents turn to light yellow. Titrate the contents of the flask against 0.1N HCl till the light yellow colour changes to light pink. Note down the titre value and call it as experimental titer value.

**B. Titration for free acidity:**

Take the same quantity of Metasytox as in the case of above titration and transfer it to a 250 ml conical flask. Dilute the contents by adding distilled water. Put methyl red drop by drop till a light pink colour appears .Titrate the contents against 0.1 N NaOH till a pale yellow colour appears .Note down the Titer value and call it as titer value for free acidity.

**C. Blank titration:**

Pipette out 10 ml of 0.1 N NaOH solution into a 250 ml conical flask, dilute the contents by adding distilled water and add methyl red till a pale yellow colour appears. Titrate the contents against 0.1 N HCl till a light pink colour appears. Note down the titer value and call it as blank titer value.

Deduct experimental titer value + titer value for free acidity from the blank titer value to get the volume of 0.1N NaOH solution utilized for hydrolysis of metasytox

.From this volume using the standard factor 1 ml of 0.1N NaOH is equivalent to 0.02463 grams of metasystox , the purity is calculated .

**Observations:**

Weight of metasystox taken : W grams  
Experimental titer value : x ml of N/10 HCl  
Free acidity titer value : y ml N/10 HCl  
Blank titer value : Z ml N/10 HCl  
Volume of 0.1 N NaOH utilized : Z-Y-X =A ml

For hydrolysis of metasystox

**Factor:**

1 mL of 0.1 N NaOH = 0.02463 grams of metasystox  
A mL of 0.1 N NaOH = 0.02463 x A grams of metasystox  
W grams of metasystox contained = 0.02463 x A grams of metasystox  
100 grams of metasystox contained = 0.02463 x A x 100/ W grams of a.i. metasystox

**Result:** The per cent active ingredient in metasystox 25 % is

**Remarks:** As per ISI specifications, metasystox 25 % EC contains a.i. in the range of 22.5 to 27.5 per cent to be acceptable for marketability.

**Conclusion:** The given sample is Accepted / Rejected for marketing.

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**Expt. No.:13**

**Date:**

### **DETERMINATION OF PURITY OF MALATHION BY IODOMETRY**

**Theory:** Malathion is an insecticide belonging to dithiophosphoric acid group. It is soluble in water. It is stable between pH 5.2 to 7.0. It is hydrolyzed both under acidic and alkaline conditions. Under acidic conditions malathion is hydrolyzed to form dimethyl thiophosphoric acid and mercapto ester of succinic acid. Under alkaline conditions it is hydrolyzed to form sodium / potassium salt of dimethyl dithio phosphoric acid and ethyl ester of fumaric Acid.

It is one of the safest insecticides used on field crops, vegetables, house hold pests *etc.* It should not be stored in containers made of iron and aluminum, since the insecticide corrodes the metal and undergoes hydrolysis. The technical compound is colourless liquid, but the commercial formulation is violet in colour due to the presence of organic dyes. Its molecular wt is 330.6 g.

**Aim:** To determine the per cent purity in commercial formulation of malathion by iodometry.

**Principle:** Malathion is refluxed with 1 *N* sodium hydroxide. During hydrolysis malathion reacts with NaOH to form sodium salt of dimethyl dithio phosphoric acid and ethyl ester of fumaric acid. The amount of hydrolysis that has taken place is determined by allowing the sodium salt of dimethyl dithio phosphoric acid to react with standard Iodine solution. The unused iodine is back titrated against 0.1 *N* sodium thiosulphate using starch as indicator. The per cent purity is determined using the factor 1ml of 0.1*N* iodine will react with 0.002065 g of malathion.

#### **Reagents:**

- 1) **1*N* NaOH:** Weigh 40 gm of NaOH and dissolve in distilled water and make up the volume to 1 liter in a volumetric flask.
- 2) **2 *N* HCl:** Dissolve 167.4 mL of concentrated HCl in distilled water and make up the volume to 1 litre with distilled water in a volumetric flask (1*N* = 83.7 mL /L).
- 3) **0.1 *N* Iodine:** Weigh 20 g of potassium iodide (KI) and dissolve in 40-50 ml of distilled water. Weigh 12.7 gm of iodine on electronic balance and transfer to KI solution. Stir the sample till the iodine is completely dissolved. Transfer the solution to 1 liter volumetric flask and make up the volume to 1 litre with distilled water. Allow the solution to stand over night and store the solution in amber coloured bottle as the solution is photosensitive.

- 4) **0.1 N Sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ):** Weigh 24.82 g of sodium thiosulphate and dissolve in distilled water and make up the volume to 1 liter.
- 5) **5% starch solution:** Weigh 5 gm of starch. Prepare slurry, by mixing with 30 ml distilled water. Boil 70 ml water and added this slurry to water and dissolve completely.

**Apparatus:** 500 ml Round bottom flask (Refluxing flask), refluxing apparatus, 500 ml conical flask, 1 litre volumetric flasks, burette, measuring cylinders, rubber tubing, burners *etc.*

**Procedure:**

1. Weigh accurately 0.1 gram of Malathion formulation by differential weighing and transfer it to 250 mL conical flask with the help of small installments of 25 mL 1 N Na OH and reflux the contents on a hot water bath for 1 hour
2. After the reflux condensation, cool the contents of the flask to the room temperature and transfer the contents to a 500 mL conical flask with 50 to 60 mL of distilled water.
3. Put one or two drops of phenolphthelain indicator when the contents turn to pink colour .Then, add drop by drop 2 N HCl to neutralize the basicity until the pink colour disappears.
4. Add 0.5 mL more of 2 N HCl to make the contents acidic.
5. After the contents are acidified, add 20 ml of standard iodine solution with the help of pipette.
6. Then, reaction take place between iodine and sodium salt of dithio phosphoric acid. Later, add few drops of freshly prepared starch indicator.
7. Titrate the contents against 0.1 N hypo solutions until the bluish violet colour disappears .This called experimental titer value.
8. A blank experiment also conducted without malathion and the titer value so obtained is designated as blank titer value. Deduct experimental titer value from the blank titer value, which gives the amount of 0.1 N iodine utilized for the reaction. From this value using the standard factor *i.e.*, 1 ml of 0.1 N iodine = 0.002065 grams of malathion .The Malathion purity is calculated.

**Observations:**

S. No.	Aliquot taken	Indicator used	Volume of 0.1N hypo consume (ml)		
			Initial A ml	Final B ml	Actual B- A

**Calculations:**

Weight of empty beaker =  $W_1$  grams

Weight of Empty beaker + Malathion =  $W_2$  grams

Weight of Malathion taken (W) =  $W_2 - W_1$

Experimental titer value = X mL of 0.1 N hypo

Blank titer value = Y mL of 0.1 N hypo

Actual titer value =  $Y - X = Z$  mL of 0.1 N hypo

(Z ml of iodine utilized for the reaction )

Titer value = Z ml of 0.1 N iodine solution

**Factor:**

1 ml of 0.1 N iodine = 0.002065 grams of malathion

Z ml of 0.1 N iodine =  $0.002065 \times Z$  grams of malathion

W grams of Malathion contains =  $0.002065 \times Z$  grams of malathion

100 grams of Malathion contains =  $0.002065 \times Z \times \frac{100}{W}$  grams of malathion

W

= Y

Percent purity = Y

**Result:** Per cent purity of malathion 50% EC =

**Remarks:** As per ISI specifications malathion 50% EC should contain active ingredient in the range of 45-55% to be acceptable for marketing.

**Conclusion:** The sample is accepted / rejected for marketing.

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**Expt. No.:**14

**Date:**

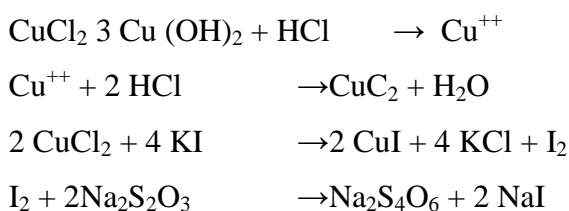
### **DETERMINATION OF COPPER CONTENT IN COPPER OXYCHLORIDE**

**Theory:** Copper oxychloride is a fungicide (copper fungicide) marketed as 80% WP. It contains 50% copper. This fungicide is marketed as Fytolon, Blitox. Its molecular formula is  $\text{CuCl}_2 \cdot 3 \text{Cu}(\text{OH})_2$  which is formed due to the reaction of cupric chloride and copper hydroxide in 1:3 ratio.

**Aim:** To determine per cent purity in given sample of copper oxychloride by iodometry.

#### **Principle:**

A known volume of blitox extract is treated with potassium iodide solution to liberate Iodine. The liberated Iodine is titrated against standard hypo solution in the presence of freshly prepared starch as an indicator till a white precipitate of cuprous iodide is formed. From the titre value using the standard factor the amount of copper is calculated.



#### **Reagents:**

- 1. Acetic acid**
- 2. Saturated disodium hydrogen phosphate**
- 3. 10 % Potassium iodide:** 10 grams of KI is weighed and dissolved in 100 ml of distilled water.
- 4. 5 % starch :** 5 grams of starch is mixed with 30 ml of distilled water and this is transferred to 70 ml of boiling water and dissolved completely (This has to be prepared fresh ).
- 5. Standard hypo solution (0.1N):** Weigh 24.83 grams of sodium thiosulphate and dissolve in distilled water and make up the volume to 1 liter in volumetric flask.

**Apparatus:** 250 ml conical flask, 1000 ml, vol. flask, 100 ml flask, Beakers, Measuring cylinders (5 ml, 10 ml, 25 ml), water bath, burette, Dropper, balance.

**Procedure:****A) Preparation of Blitox extract:**

1. Transfer 20 grams of blitox powder (80 % WP) to a 1 liter beaker and add 50 ml of distilled water followed by 10 mL of acetic acid.
2. Stir the contents vigorously and heat the contents on a flame till the contents just boiled.
3. At this stage remove the beaker from the flame and cool the contents to room temperature.
4. After cooling add 10 mL of saturated di-sodium hydrogen phosphate solution to remove iron if at all present as an impurity in the blitox powder.
5. Transfer the contents of the beaker to a 1liter volumetric flask fitted with a funnel with repeated washings with distilled water.
6. Transfer the contents of volumetric flask to another container through a funnel fitted with filter paper.
7. The filtrate is green in colour, which is nothing but the Blitax extract.

**B). Determination of copper in the Blitax extract:**

1. Transfer 10 mL of the blitox extract to a 250 mL conical flask with the help of a pipette
2. Dilute the contents of the flask by adding 20-30 ml distilled water.
3. Now ,add 10 mL of 10% Potassium iodide solution with a measuring cylinder when the contents turn to brown due to liberation of Iodine
4. Titrate the contents against standard hypo solution (0.1N) till a pale yellow colour suspension appears.
5. At this stage put a few drops of starch indicator and continue the titration till a white precipitate of cuprous iodide is formed.
6. Note down the titer value and repeat the experiment till two consecutive concurrent readings are obtained.
7. From the titer value using the factor ( Equivalent wt. of copper is 63.56)

1ml of 0.1N hypo is = 0.006456 grams of copper, the amount of copper is calculated.

**Observations:**

S. No.	Aliquot taken	Indicator used	Volume of 0.1N hypo consumed (mL)		
			Initial A mL	Final B mL	Actual B- A

**Calculations:**

Titer value = Z mL of hypo solution

**Factor:**

1mL of 0.1N hypo = 0.006356 grams of copper

Z mL of 0.1N hypo contains = 0.006356 x Z gram of copper

10 mL of blitox extract contains = 0.006356 x Z gram of copper

1000 mL of blitox extract contains =  $\frac{0.006356 \times Z \text{ gram of copper} \times 1000}{10}$

1000 mL of blitox extract was prepared from 20 grams of blitox powder

Hence,

20 grams of Blitax contains =  $\frac{0.006356 \times Z \text{ grams of copper} \times 1000}{10}$

100 grams of Blitax contains =  $\frac{0.006356 \times Z \text{ g. of copper} \times 1000 \times 100}{10 \times 20}$  = Y g. of Cu

**Result:** The amount of copper present in 80% WP of copper oxychloride is

**Remarks:** As per ISI specification percent copper present in 80% WP of copper oxychloride should be in the range of 48-52 per cent.

**Conclusion:** The sample is accepted / rejected.

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**Expt. No.:15**

**Date:**

**DETERMINATION OF SULPHUR CONTENT IN SULPHUR FUNGICIDE  
(ELEMENTAL SULPHUR)**

**Theory:** Sulphur is formulated as dust and wettable powder and is used as fungicide as well as insecticide. To increase the free flowing nature of dust formulation, sulphur, is mixed with inert materials like gypsum, talc, kaolinite to an extent of 1 to 5 % of its weight. Sulphur is formulated as 1-5 % dust and 80 % wettable powder.

Ex: Elemental sulphur

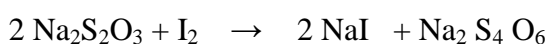
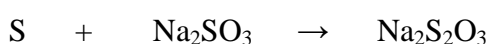
**Aim:** To determine per cent purity of sulphur WP (80%) (Element sulphur) by iodimetry.

**Principle:**

A known quantity of the sulphur compound is refluxed with sodium sulphite in the presence of ethanol to form sodium thiosulphate (Hypo) .The hypo so formed is acidified and titrated against standard iodine solution using freshly prepared starch as an indicator till a bluish violet colour is appeared .After correcting for free sodium thiosulphate in the compound the per cent purity is calculated using standard factor.

1 mL of 0.1 N iodine = 0.0032 gram of sulphur

Ethanol



Starch

**Reagents:**

- 1. 99 % Ethanol :**
- 2. Sodium sulphite crystals :**
- 3. 20 % acetic acid :** 20 ml in 100 ml distilled water
- 4. 10 % formaldehyde:**
- 5. 5 % starch indicator :** Five grams of starch is mixed with 30 mL of distilled water and this is transferred to 70 ml of boiling water and dissolved completely (This has to be prepared fresh ).
- 6. Standard 0.1 N Iodine solution:** Weigh 20 g of Potassium iodide and dissolve in 40-50 ml of distilled water. Weigh 12.7 gm of iodine on electronic balance and transfer to KI solution. Stir the sample till the iodine is completely dissolved. Transfer the solution to 1 liter volumetric flask and make up the volume to 1 litre

with distilled water. Allow the solution to stand over night and store the solution in amber coloured bottle as the solution is photosensitive.

**Apparatus:** 500 ml round bottom flask, refluxing apparatus, 250 ml conical flask, 100 ml volumetric flask, filter paper, funnel, 20 ml pipette, 10, 20 and 50 ml measuring cylinder, beakers, burette, dropper, balance etc.

**Procedure:**

1. Weigh accurately 0.2 grams of the given sulphur formulation and transfer it to a 250 ml conical flask.
2. Wet the sample by adding a few drops of distilled water.
3. Add 30 ml of 99 % ethanol followed by 5 gram of sodium sulphite crystals to the conical flask and reflux the contents on a hot water bath for 1 hour
4. After reflux condensation cool the contents to room temperature
5. Transfer the contents of conical flask to a 100 mL volumetric flask fitted with a funnel with repeated washings with distilled water .Make the volume to 100 mL with distilled water
6. Shake the contents of the volumetric flask thoroughly and transfer the contents to another container provided with a funnel and filter paper.
7. After the complete filtration, pipette out 10 or 20 mL of the filtrate to a 250 mL conical flask.
8. Now add 10 mL of 20 % acetic acid solution followed by 10 mL of 10 % formaldehyde solution and add a few drops of freshly prepared starch
9. Titrate the contents of the flask with 0.1 N Iodine solution till a **bluish / bluish violet** colour appears.
10. Note down the titer value and call it as experimental titer value
11. Weigh the same quantity of sulphur formulation as in the case of above titration and reflux the contents as above without sodium sulphite crystals following the normal procedure ,and find out the free hypo present in the sulphur formulation .The titer value so obtained is designated as titer value for free sodium thiosulphate .
12. Deduct the free sodium thiosulphate titer value from the experimental titer value which gives the volume of standard iodine solution i.e., required for the neutralization of sodium thiosulphate liberated from the sulphur compound.
13. Using this titer value and using the standard factor i.e.,  
**1 ml of 0.1N Iodine solution = 0.0032** grams of sulphur, the purity of sulphur is Calculated.

**Observations:**

S. No.	Aliquot taken	Indicator used	Volume of 0.1N Iodine consume (ml)		
			Initial A ml	Final B ml	Actual B- A

**Calculations:**

The experimental titer value : x mL of 0.1N I<sub>2</sub>

Free hypo titer value : y ml of 0.1N I<sub>2</sub>

Volume of 0.1 N iodine utilized : x- y = z ml of 0.1N I<sub>2</sub>

**Factor:**

Aliquot taken = 10 ml

**1 mL of 0.1 N iodine = 0.0032 gram of sulphur**

Z mL of 0.1 N iodine = 0.0032 x z grams of sulphur

10 mL o filtrate contains = 0.0032 x z grams of sulphur

100 mL of filtrate contains = 0.0032 x z x 100/10 grams of sulphur

0.2 grams of sulphur formulation contains =  $\frac{0.0032 \times z \times 100}{10}$  grams of S

100 grams of sulphur formulation contains

$$= \frac{0.0032 \times z \times 100 \times 100}{10 \times 0.3} \text{ grams of sulphur}$$

$$= Y$$

$$\% \text{ sulphur} = Y$$

**Result:** Per cent sulphur present in given sample is \_\_\_\_\_

**Remarks:** As per ISI standards; 80% WP of sulphur should contain a.i. within the range of 75-85% to be acceptable for marketing.

**Conclusion:** The sample is accepted / rejected.

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**Expt. No.:16**

**Date:**

**COMPATIBILITY OF FERTILIZERS WITH INSECTICIDES AND  
FUNGICIDES**

To plant protection as well as plant production schedule very often it becomes necessary to combine application of different agricultural chemicals due to several reasons. It is very economical to mix various agrochemicals together to save cost of material application, to save water in water scarcity areas and time. Time in the sense ,little time may be available for separate sprays because of emergency in pest or disease situation ,because of short intervals between rains particularly in the monsoon season .Though combining application is advantageous , it may create problem if one is not careful .

Pesticides and fertilizers being agricultural chemicals , tend to react with each other through various chemical reactions such as oxidation ,reduction , hydration ,hydrolysis ,neutralization ,double decomposition etc., .The reaction products so formed may have no pesticide value .For example **CAPTAN** , a fungicide can be mixed with agricultural materials that are alkaline in reaction because under alkaline condition **CAPTAN** gets decomposed there by loosening its fungicidal value . Some times materials can be mixed for immediate application but not for using after certain time interval.

It is therefore essential to know the interactions between agriculture chemicals that are to be mixed .Sometimes mixing agriculture chemicals may result in an increase or decrease in the activity of materials to be mixed .Sometimes the reaction products may be phytotoxic or may effect the spraying equipment like clogging of nozzles corroding of spraying equipment .Hence, the farmers should have a sound knowledge about the compatibility of various agro-chemicals.

**Note:** For compatibility of different chemicals refer the latest **Vyavasaya Panchangam** published by ANGRAU.

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