

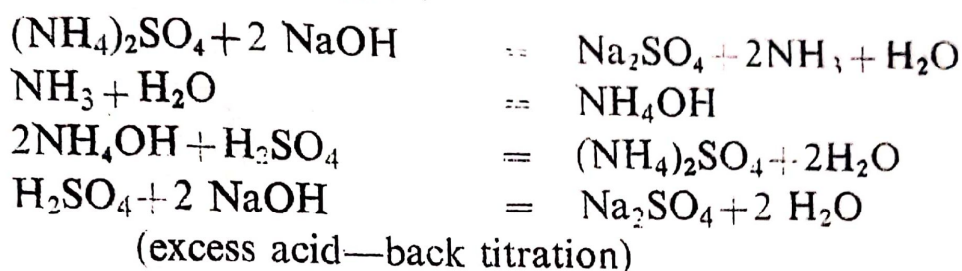
## EXPERIMENT-17

To determine the nitrogen percentage in the given sample of ammonium sulphate  $(\text{NH}_4)_2\text{SO}_4$  (Ammonical form of nitrogen).

### 17.1 PRINCIPLE:

When ammonium containing salts are heated in the presence of sodium hydroxide, ammonia\* gas is evolved which is absorbed in a known quantity of std. acid, the excess is back titrated against std. alkali.

### 17.2 CHEMICAL REACTION:



### 17.3 PROCEDURE:

#### 17.31 Preparation of the extract:

Take 1 g of given fertilizer in a beaker. Add 20 ml distilled water, shake and transfer the solution into 100 ml volumetric flask. Shake the flask and make up the volume upto the mark.

#### 17.32 Nitrogen distillation: (filtration/purification)

1/ Take 10 ml of the extract (prepared in 17.31) into a long neck Kjeldahl distillation flask and add 150 ml distilled water. 2 Convert this flask to Macro Kjeldahl distillation apparatus. 3 Prepare the receiver by taking 25 ml of N/10  $\text{H}_2\text{SO}_4$  in a conical flask. 4 Add 2-3 drops of methyl red indicator and place the flask under the *Attach*

\*Boric acid can also be used as  $\text{NH}_3$  receiving solution being a weak acid which is then titrated directly with  $\text{H}_2\text{SO}_4$  in the presence of a mixed indicator. This method needs no back titration.

condenser, adjusting the height so that the tip of the condenser is just under the liquid. 5 Now add 40 ml 50% NaOH solution to the already fixed Kjeldahl flask by opening the bung carefully, so that the upper part of the long neck does not get wet (bung will slip) or else wash with distilled water and fix the bung back to the flask. 6 Boil the flask till 2/3 portion of the contents have been distilled. 7 Remove the receiver by lowering it and washing the tip of the condenser with distilled water into the flask. 8 Titrate the excess acid in the conical flask with N/10 NaOH to a golden yellow end point (in case the colour of the indicator in the receiver, changes before removing the receiver add more known quantity of N/10 H<sub>2</sub>SO<sub>4</sub> and let the distillation go for some more time).

#### 17.4 CALCULATION:

|   |   |                                     |
|---|---|-------------------------------------|
| N/10 H <sub>2</sub> SO <sub>4</sub> taken in the receiver | = | 25 ml                               |
| N/10 NaOH used for back titration                         | = | X ml <i>10 ml</i>                   |
| Net H <sub>2</sub> SO <sub>4</sub> used                   | = | (25-X) ml                           |
| Now 1 ml of N/10 H <sub>2</sub> SO <sub>4</sub>           | = | 0.0014 g of Nitrogen*               |
| (25-X) ml of N/10 H <sub>2</sub> SO <sub>4</sub>          | = | 0.0014 (25-X) g N                   |
| 10 ml of solution contain N                               | = | 0.0014 (25-X)                       |
| 100 ml of solution contain N                              | = | 0.0014 (25-X) 100/10                |
| <i>fertilizer</i> ← 1 gm soil contains N                  | = | 1.4 (25-X) 10 <sup>-2</sup>         |
| ← 100 gm soil contains N                                  | = | 1.4 (25-X) 10 <sup>-2</sup> × 100/1 |

#### 17.5 RESULTS:

$$\% \text{ N} = 1.4 (25 - X)$$

$$\begin{aligned} \% \text{ N} &= 1.4 (25 - 10) \\ &= 1.4 \times 15 = 21 \% \end{aligned}$$

\* NH<sub>3</sub> gas is absorbed as NH<sub>4</sub> OH which neutralizes the acid (H<sub>2</sub>SO<sub>4</sub>). Nitrogen present in NH<sub>3</sub> or NH<sub>4</sub> OH is only 14 gram per formula wt. One normal sol. of NH<sub>4</sub> OH contains 14 grams of Nitrogen per liter of solution or 0.014 gm per ml of solution. Therefore 1 ml. of 1N H<sub>2</sub>SO<sub>4</sub> = 0.014 g Nitrogen per ml of NH<sub>4</sub> OH or 1 ml of N/10 H<sub>2</sub>SO<sub>4</sub> = 0.0014 g Nitrogen per ml of NH<sub>4</sub> OH.



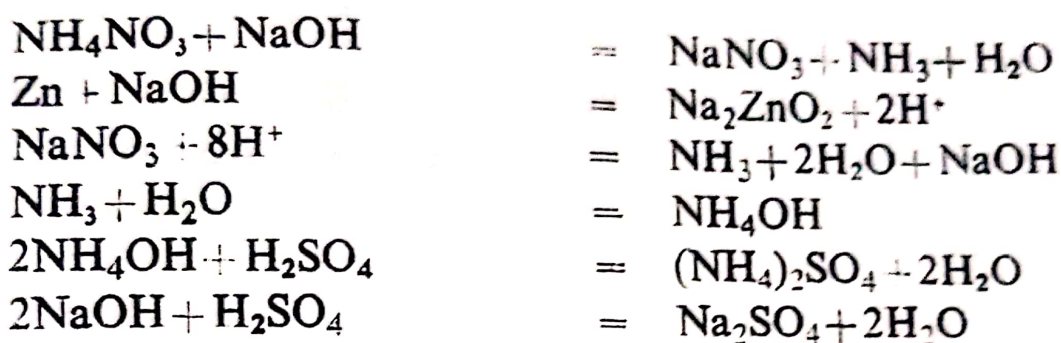
## EXPERIMENT-19

To determine the nitrogen percentage in the given sample  $\text{NH}_4\text{NO}_3$  (both ammonical and nitrate form).

### 19.1 PRINCIPLE:

Same as in nitrate nitrogen (18.1).

### 19.2 CHEMICAL REACTION:



### 19.3 PROCEDURE:

As given in 17.3.

### 19.4 CALCULATION:

As given in 17.4.

### 19.5 RESULTS:

Same unit as in 17.5.

$$\begin{aligned}\% \text{ N} &= 1.4 (25 - 3) \\ &= 1.4 (22) \\ &= 32.2 \%\end{aligned}$$

## **EXPERIMENT-20**

To determine the nitrogen percentage in the given sample of urea (organic form of nitrogen)

1. Nitrometer method
2. Digestion/distillation method

## ✓ 20.2 DIGESTION/DISTILLATION METHOD:

### 20.21 PRINCIPLE:

The material is first digested with con.  $H_2SO_4$  and the digestion mixture. The digested material is then distilled with NaOH solution. Ammonia gas is evolved and this is absorbed in std. acid solution and the excess is back titrated with std. alkali.

### 20.22 PROCEDURE:

Weigh 0.5 g of Urea, transfer it to 100 ml volumetric flask and make up the volume. Take 10 ml of the solution in a digestion flask. Add 30 ml. con.  $H_2SO_4$  and 10 g digestion mixture\* to the

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(\*) = Digestion mixture contains 10 parts  $K_2SO_4$  or anhydrous  $Na_2SO_4$ , 1 part  $FeSO_4$  and 0.5 parts  $CuSO_4$

$K_2SO_4$  or  $Na_2SO_4$  are needed to raise the temperature of the digestion process and  $FeSO_4$  and  $CuSO_4$  acts as catalyst to promote oxidation of organic matter in soil and acts as catalyst respectively. For plant samples selenium should be used in place of  $FeSO_4$ . The maximum shall be (10g  $K_2SO_4$  + 1g  $CuSO_4 \cdot 5H_2O$  + 0.1 g Se).

flask. Digest the contents of the flask till the solution is colourless or nearly so. After cooling transfer the contents of the flask to a 250 ml volumetric flask and make up the volume.

Take 10 ml of this solution in a Kjeldahl distillation flask and run the distillation test as described in 17.3.

(Ammonical nitrogen distillation).

20.23 CALCULATION:

|   |                     |                      |
|---|---------------------|----------------------|
| N/10 H <sub>2</sub> SO <sub>4</sub> taken in the receiver | = X ml              | <sup>25</sup>        |
| N/10 NaOH used in titration                               | = Y ml              | (25 - 18.5) ml = 6.5 |
| Net acid used   | = (Y - X) = T ml    |                      |
| 1 ml of N/10 H <sub>2</sub> SO <sub>4</sub>               | = 0.0014 g N        |                      |
| T ml N/10 H <sub>2</sub> SO <sub>4</sub>                  | = 0.0014 T g of N   |                      |
| 10 ml of solution contains nitrogen                       | = 0.0014 Tg         |                      |
| 250 " " " "   | = 0.0014 T × 250/10 |                      |
|   | = 0.035 T g         |                      |
| Now 0.5 g Urea contain nitrogen                           | = 0.035 T g         |                      |
| 100 " " " "   | = 0.035 T × 100/05  |                      |
|   | = 7 T               |                      |

20.24 RESULTS:

Nitrogen in Urea = 7 T %



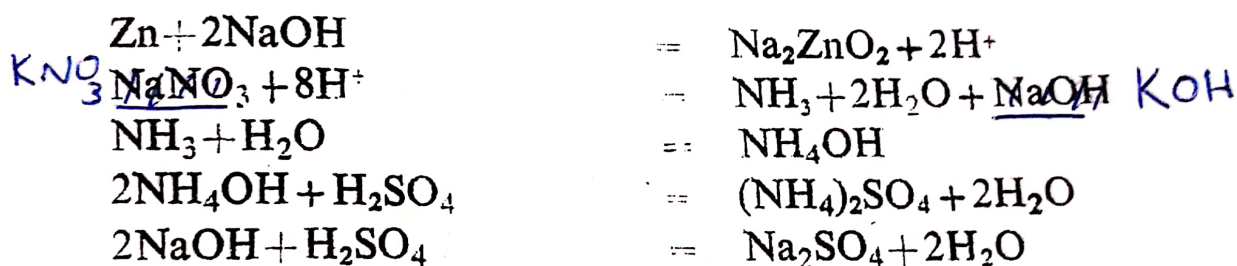
## EXPERIMENT-18

To determine nitrogen percentage in the given sample of potassium nitrate ( $\text{KNO}_3$ ) (nitrate form of nitrogen).

### 18.1 PRINCIPLE:

Nitrate salts are heated with sodium hydroxide in the presence of zinc dust and ferrous sulphate (as catalyst). Hydrogen gas is formed which combines with nitrate nitrogen to form ammonia which is then absorbed in std.  $\text{H}_2\text{SO}_4$ , excess of which is back titrated against std. sodium hydroxide.

### 18.2 CHEMICAL REACTION:



### 18.3 PROCEDURE:

As given in 17.3.

### 18.4 CALCULATIONS:

As given in 17.4.

### 18.5 RESULTS:

Units same as in 17.5.

$$\% \text{N} = 1.4 (25 - x)$$

$$\% \text{N} = 1.4 \times 12$$

$$= 16\%$$

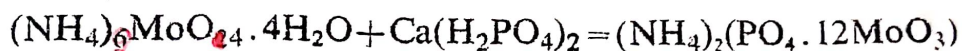
## EXPERIMENT-22

To determine the percentage of available  $P_2O_5$  in the given sample of phosphorus fertilizer.

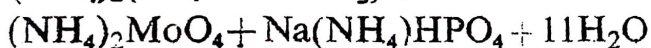
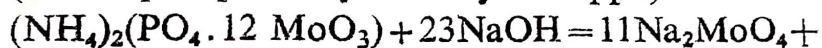
### 22.1 PRINCIPLE:

Phosphorus (present as orthophosphate) can be determined by precipitating it as ammonium phosphomolybdate by adding ammonium molybdate in acidic medium. The ppt. is filtered, washed and dissolved in std. sodium hydroxide. The excess of which is back titrated against std.  $H_2SO_4$  solution.

### 22.2 CHEMICAL REACTION:



(Amm. phosphomolybdate-yellow ppt.)



### 22.3 PROCEDURE:

Weigh 0.5 g of the given fertilizer. Transfer into a beaker and add 30 ml water to it. Boil and transfer the contents to 250 ml volumetric flask giving washing to the beaker and make up the volume to the mark with distilled water. Take 10 ml solution into a conical flask. Add a few drops of ammonium hydroxide until turbidity appears and add 5 ml of con. nitric acid. To the flask also add 15 ml of the 50% solution of ammonium nitrate. Heat the contents of the flask upto  $70^\circ C$  and add 50 ml of the 3% solution of ammonium molybdate from the burette, first drop by drop and then in the form of a stream shaking the flask vigorously for 5 minutes. Allow the ppt to settle. Filter through Whatman No. 40 filter paper. Give washing with dilute sodium nitrate solution or ice cold distilled water until the filtrate is acid free

... that should be composted only in limited amounts include wood ashes (a source of lime) and sawdust (requires extra nitrogen).



(Does not turn blue litmus red). Transfer the ppt. alongwith the filter paper to the same conical flask. Dissolve the ppt. in N/10 NaOH by adding 10 ml at a time by means of a pipette into the flask and titrate the excess of alkali against N/10  $H_2SO_4$  to a colourless and point.

#### 22.4 CALCULATION:

Wt. of fertilizer taken = 0.5 g

Total amount of N/10 NaOH used in dissolving the ppt. = X ml

Amount of N/10  $H_2SO_4$  used for back titration = Y ml

Net amount of N/10 NaOH used = (X - Y) = T ml

1 ml of N/10 NaOH solution = 0.000309 g of  $P_2O_5$

T ml of N/10 NaOH solution = 0.000309 T g  $P_2O_5$ \*

10 ml of fertilizer solution contains  $P_2O_5$  = 0.000309 Tg

250 " " " " " " = 0.000309 T  $\times$  250/10 g

0.5 of fertilizer contains  $P_2O_5$  = (0.000309 T)250/10 g

100 g " " " " = (0.000309 T)250/10  $\times$  100/0.5

= 1.545 T

#### 22.5 RESULTS:

%  $P_2O_5$  = 1.545 T.

(\*) In the chemical reaction out of 23 molecules of NaOH on LHS only one molecule of Na is used to form  $Na(NH_4)HPO_4$  which also contains only one molecule of P. In simple words the ammonium phosphomolybdate ppt contains Na and P in the ratio of 1:1. Therefore one can write that: 1 N Sol. of NaOH (23g Na/l) = 1N Sol. of P (31 g of P/l) or 1N NaOH = 31/23 g of P/l = 0.001347g of P/ml = 0.000309 g of  $P_2O_5$  /ml. hence.  
1 ml N/10 NaOH = 0.000309 g of  $P_2O_5$

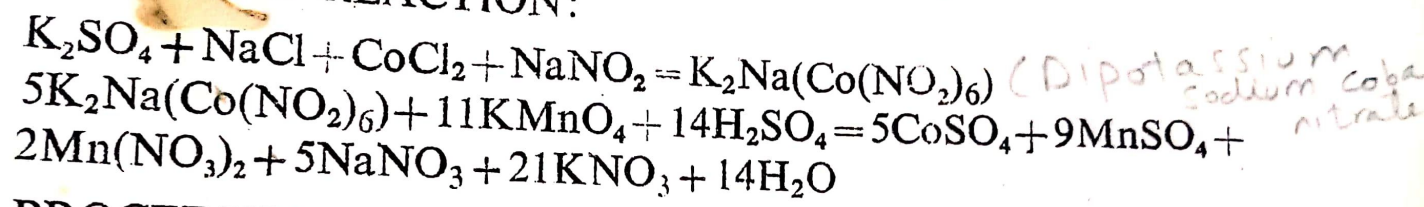
## EXPERIMENT-25

To determine the amount of  $K_2O$  in the given sample of potassium fertilizer.

### 25.1 PRINCIPLE:

Potassium can be determined by precipitating it as dipotassium sodium cobaltinitrate. Ppt is dissolved in oxalic acid and back titrated with  $KMnO_4$ . (Potassium per manganate)

### 25.2 CHEMICAL REACTION:



### 25.3 PROCEDURE:

1 Weigh 0.5 g of fertilizer. 2 Transfer into a beaker and add 30 ml of water to the beaker. 3 Boil and transfer the contents to 250 ml volumetric flask giving washing to the beaker and make up the volume. 4 Take 10 ml solution in china dish and evaporate it over water bath. 5 Add to it 10 ml saturated solution of NaCl, 10 ml of 10%  $CoCl_2$  and 10 ml of 10%  $NaNO_2$  solution in order. 6 Stir the contents with a glass rod and dry these over a water bath with occasional shaking to a thick paste. 7 Dissolve this paste in 10 ml of 10%  $CH_3COOH$  and allow it to stand for 15 minutes. 8 Then add 10 ml of water and filter the contents through a geuche crucible using suction pump (A layer of asbestos should be used in the filtration). 9 Give washings with 2.5%  $H_2SO_4$  solution until the filtrate is colourless. 10 Take 50 ml of N/20  $KMnO_4$  solution in a beaker. 11 Add about the same quantity of dil.  $H_2SO_4$  to it and boil. 12 When the solution is just about to boil, add crucible along with ppt to the beaker and boil it for 2-3 minutes. 13 Add 50 ml



N/20 oxalic acid and stir it for few minutes. Then back titrate the excess of acid against N/20  $KMnO_4$ . Calculate the amount of  $KMnO_4$  used. From this calculate the %  $K_2O$  in the sample.

25.4 CALCULATION:

|   |                              |
|---|------------------------------|
| N/20 $KMnO_4$ used for oxidizing the ppt = A ml |                              |
| 1 ml of N/20 $KMnO_4$                           | = 0.000425 g of $K_2O$       |
| A " " " "                                       | = 0.000425 A g of $K_2O$     |
| 10 ml sol. contain $K_2O$                       | = 0.000425 A                 |
| 250 " " " "                                     | = 0.000425 A $\times$ 250/10 |
| 0.5 g fertilizer contain $K_2O$                 | = 0.000425 A $\times$ 250/10 |
| 100 " " " "                                     | = 0.000425 A $\times$ 250/10 |
|   | $\times$ 100/0.5             |
|   | = 2.125 A                    |

25.5 RESULTS:

%  $K_2O$  = 2.125 A.

(\*) It is an empirical factor and is calculated as under. The chemical reaction shows that on molecular weight basis 11 parts of  $KMnO_4$  reacts with 5 parts of dipotassium sodium cobaltinitrate or 11  $KMnO_4 = 5K_2O = 10K$ , now  $KMnO_4$  contributes 5 units of released oxygen (Eq. of  $KMnO_4 = 1/5$  M. Wt.). Therefore,  $KMnO_4$  contributes oxygen as (11  $\times$  5) and = 10 of K or 1  $KMnO_4$  combines with 10/55 parts of K which means 1 ml of 1N  $KMnO_4 = 10/55$  (39 g/l of K)