ANALYSIS PROCEDURE - CAUSTIC POTASH

(1) S.Gr., Temp : Refer Chart to know the total alkalinity as KOH Concentration.

(2) Determination of KOH and K_2Co_3 : Weigh one empty dry weighing bottle with Lid. Take about 2 to 3 grams of flakes or 3 to 4 ml of lye by means of graduated pipette into the weighing bottle and weigh again. Transfer the sample into a conical flask, add about 50 ml distilled water and add 2 to 3 drops of Phenolphthalein indicator.

Weigh of sample = W grams

To know approximate volume of 1 N Hcl

(Titre value)

Percentage/5.6 = vol. of 1N Hcl for 1 gram sample.

Titrate it against standard I N Hydrochloric Acid solution upto a little before the end point. Take this reading on A. Further titrate it against 0.I N Hydrochloric Acid solution till the pink colour just disapperars. Take this reading on B. Then add 2 to 3 drops of Methyl orange indicator and continue titration against 0.1N Hydrochloric Acid to a reddish orange colour. Take this reading as C.

Total Phenolphthalein T.V. in 1 N = A + B/10 = X ml

Methyl Orange T.V.in I N = C/10 = Y ML

Phenolphthalein end point = KOH + ½ $K_2 Co_3$

Methyl Orange end point = ½ $K_2 Co_3$

KOH% = [(X-Y] X 1 N X 56 X 100]/(W X 1000)

= [(X-Y) X 5.6]/W

$K_2 Co_3$ = (2Y X 1 N x 69 x 100)/(W X 1000)

= (Y x 13.8)/W

(3) Determination of Chlorides :- Weigh accurately about 10 grams of flakes or 20 grams of the lye. Transfer it into a 250 ml conical flask, add about 50 ml distilled water, neutralize it with concentrated Nitric Acid and then add about 5 ml of the acid in excess, cool to room temperature. Pipette out 10 ml of 0.05 N $Ag No_3$ into it. Add 5 ml of Nitro benzene or Carbon Tetrachloride. Shake well. Add Ferric Ammonium Sulphate indicator. Titrate it against 0.05 N Ammonium Thiocyanate solution. The end point being appearance of permanent red-brown colour.

10 ml of Silver Nitrate = 10 ml Ammonium Thiocyanate (Blank expt.)

Silver Nitrate consumed = (10-T.V) ml

Weight of the sample = W grams

Chloride (as Kcl) percent by mass

= (AX0.05X74.5)X100/WX1000

= (A X 0.3725)/W.

(4) Determination of Iron: Weigh accurately about 10 grams of flakes or 20 grams of the lye. Transfer it into a 250 ml beaker, add about 50 ml distilled water, neutralize it with concentrated hydrochloric Acid and then add about 5 ml of the acid in excess. Add a pinch of Ammonium Persulphate and boil well for 15 to 30 mtnts (Chlorine is to be expelled), cool to room temperature. Add 10 ml of 10% Potassium Thiocyanate solution. Red colour develops due to the formation of Ferric Thiocyanate Fe(CNS). Transfer the solution into a 100 ml Nessler cylinder and make up to the mark.

This red colour is compard with red colour produced by known volume of standard Iron solution.

1 ml standard Iron solution = 0.1 mg of Fe.
Volume of standard Iron solution used = V ml
Weight of the sample = W grams

Iron (as Fe) ppm = (V x 0.1x1000)/W
= (100 x V) / W

(5) Determination of Nickel :- Weigh accurately about 10 grams of flakes,transfer to a beaker. Add about20 ml distilled water, neutralize it with Conc. Hydrochloric Acid, cool to room tempeature. Add Bromine water in drops to slight excess. Add 5 ml of 10% Sodium citrate solution. Add one spoonful of Ammonium Chloride solid (up to this stage, Bromine colour persist). Add 1 : 1 Ammonia solution in drops till Bromine colour vanishes. Add 5 ml of 0.2% Sodium dimethyl glyoxime solution. If Nickel is present, red-brown colour is developed. Transfer this solution into a 100 ml Nessler cylinder and make up to the mark.

This red brown colour is compared with red-brown colour produced by known volume of standard Nickel solution.

1 ml of standard Nickel solution = 0.1 mg of Ni
Volume of standard Nickel solution used = V ml
Weigh of sample = W grams

Nickel (as Ni) ppm = (V X 0.1 X 1000)/W
=(100xV)/W

******