ANALYSIS PROCEDURE – CAUSTIC SODA

(1) Sp. Gr., Temp : Refer chart to know the total alkalinity of NaOH. Concentration.

(2) Determination of NaOH and $Na_2 Co_3$:

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.

Weight of the sample = W grams

To know the approximate volume of 1 N Hcl
(Titre value)

Percentage/4 = Volume of 1 N Hcl for 1 gram sample.

Titrate it against standard 1 N Hydrochloric acid solution upto a little before the end point. Take this reading as A. Further titrate it against 0.1 N Hydrochloric Acid solution till the pink colour just disappears. Take this reading as B. Then add 2 to 3 drops of Methyl Orange Indicator and continue titration against 0.1N Hydrochloric Acid to 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 1N = C/10 = Y ml

Phenolphthalein end point = Na OH + ½ $Na_2 Co_3$

Methyl Orange end point = ½ $Na_2 Co_3$

NaOH% = (x-y) x 1 N x 40 x 100/W x 1000
= (x-y)4/W

Na_2Co_3% =2Yx 1 Nx53X100/(Wx1000)
= Y x 10.6/W

(3) Determination of Chlorides:- Weigh accurately about 10 grams of flakes or 20 grams of 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 out10 ml of 0.05N Silver Nitrate solution into it. Add 5 ml of Nitrobenzene. or Carbon Tetrachloride. Sheke well. Add Ferric ammonium sulphate indicator solution.Titrate it against 0.05 N Potassium thiocyanate solution. The end point being appearance of permanent red-brown colour.

10 ml of Silver Nitrate = 10 ml of Potassium thiocyanate
(Blank expt.)

Silver Nitrate consumed = (10 – T.V.) Ml
= A ml

Weight of the sample = W grams

Chloride (asNaCl) percent by mass
= Ax0.05x58.5x100/(W x 1000)
= Ax0.2925/W

(4) Determination of Chlorates and Perchlorates (as Sodium Chlorate):
Weigh accurately about 50 grams of sample in a beaker and neutralize it with Sulphuric Acid (1:1) carefully after placing the beaker in cold water. Add 5 ml of the acid in excess and dilute to 250 ml. Transfer the contents of the beaker to a 500 ml conical flask. Add 25 ml of Ferrous Ammonium Sulphate solution. Close the flask with abunsen valve and boil the contents gently for 15 minutes. Allow the flask to cool to room temperature. After cooling, titrate the contents of the flask with 0.02 N Potassium Permoangonate solution slowly with stirring till a pink colour persists.note the volume as V2.

Run a blank by titrating 25 ml of Ferrous Ammonium Sulphate containing 5 ml of Sulphuric Acid and 200 ml water against 0.02N Potassium Permanganate, proceeding in the same way as that of test. Note the Volume as VI.

Weight of the sample = W grams
Chlorates (as NaClo3) ppm = {(VI-V2) X N X 17.75)/M} X 1000
= {(VI-V2) X N X 17750}/M

(5) Determination of Iron: Weigh accurately about 10 grams of flakes or 20 grams of lye, transfer it into a 250 ml beaker, add about 50 ml distilled water. Neutralise 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 mnts (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 Ferricthiocyanate $Fe(CNS)_3$. transfer the solution into a 100 ml Nessler Cylinder and make up to the mark.
This red colour is compared with red colour produced by known volume of standard Iron solution.

1 ml of 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 0.1 x 1000)/W
= (100 x V)/W.

(6) Determination of Nickel: Weigh accurately about 10 grams of flakes, transfer to beaker, add about 20 ml distilled water, neutralize it with concentrated Hydrochloric Acid, cool to room temperature. Add Bromine water in drops to slight excess. Add 5 ml of 10% Sodium Citrate solution. Add one spoonful of solid Ammonium Chloride (up to this stage, Bromine colour should 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 upto 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
Weight of sample = W grams
Nickel (as Ni) ppm = (V x 0.1 x 1000)/W
= (100 x V)/W

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