SULPHURIC ACID – METHOD OF ANALYSIS

Total Acidity: Accurately weigh about 2 Grams of material with Weighing bottle or glass ampoule. If the glass ampoule is used, carefully mix the Test sample. Take the sample in a W.M. Bottle and slightly heat in a flame the bulb of glass ampoule previously weighed to the nearest 0.1 mg. Immerse the capillary end of the ampoule into the bottle containing the test sample and ensure that the bulb is filled up to about two-thirds of its volume during cooling (1 to 2 ml approximately). With draw the ampoule and carefully wipe the capillary end with filter paper. Seal the capillary end in the oxidizing flame without loss of glass. Remove from the flame and allow to cool. Wash the capillary and wipe carefully with filter paper. Weigh the ampoule to the nearest 0.1 mg and calculate by difference the mass of the test portion.

Carefully place the ampoule containing the test portion into the Conical Flask containing 300 ml cold water stopper the flask and shake to break the ampoule containing test portion. Cool during this operation keep cooling and shaking until the vapours are completely absorbed. Remove the stopper and rinse it with water, collecting the washings in the Conical Flask by means of a glass rod, grind the fragments of the ampoule and in particular the capillary which may have remained intact in spite of shaking, withdraw the glass rod and wash it with water collecting the washings in the Conical Flask.. Add two drops of Methyl Orange indicator solution and titrate to the end Point with standard Sodium Hydroxide solution.

Calculation:

$$\text{Total acidity (as } H_2SO_4\text{) percent by mass} = \frac{V \times N \times 4.904}{M}$$

Where

V = Volume in ml of standard Sodium Hydroxide solution used for the titration.

N = Normality of standard Sodium Hydroxide solution

M = Mass in Grams of the sample taken for the test.

..contd..2…

Residue on Ignition: Weigh to the nearest 10 Mg about 50 grams of the sample in a 100 ml capacity Silica dish previously ignited at 800° C, cooled in a desiccator and weighed. Evaporate the acid carefully on a sand bath, heating the dish containing the test portion. Heat to dryness place the dish containing the residue in an Electric furnace heated at 800° C and keep at this temperature for about 15 minutes. Remove the dish from the Furnace, cool in a desiccator and weigh. Repeat heating, cooling and weighing fill constant mass is obtained.

Calculation:

$$\text{Residue on ignition percent by mass} = \frac{M1 \times 100}{M_2}$$

M1 = Mass in grams of the residue weighed.
M2 = Mass in grams of the sample taken for test.

Determination of Iron: Dilute exactly 1 Gram of the material to 10 ml with D. water in a Nessler Cylinder Add one drop of Potassium Permanganate solution and mix thoroughly. Add 5 ml of Ammonium thiocynate (60%) solution and 10 ml of Amyl alcohol and Amyl acetate mixture (1 : 1). Make up to 50 ml. Shake vigorously and allow the layers to separate. Compare the intensity of any red colour produced in the upper layer with a control test carried out in another Nessler Cylinder in the same manner using the standard Iron solution in place of the sample.

Test for oxidisable Impurities: Take a known volume of the material (say 10 to 20 ml) in a 250 ml Conical Flask and dilute with an equal volume of water. Titrate the solution against standard Potassium Permanganate to a light permanent Pinc colour end Point.

Calculation:

$$\text{Oxidisable impurities (as } So_2\text{) percent by mass} = \frac{3.2 \times V \times N}{M}$$

V = Volume in ml of standard Permanganate solution used for the titration.

N = Normality of Potassium Permanganate colution.

M = Mass in Grams of the sample taken for the Test.

* * *