DETERMINATION OF CALCIUM OXIDE CONTENT DETERMINATION OF CALCIUM OXIDE CONTENT

B.S. 1881 PART 124 SECTION 5

A COMPLEX ONE TITRATION USING E.D.T.A. Na^+ SALT SAMPLING

As for soluble Chloride analysis.

DIGESTION

Weigh out accurately about 10.0 grams to 3 d.p. of the sample into a 600ml beaker. Suspend with distilled water and swirl the contents of the beaker. Cautiously add 10ml of Conc. A.R. Hydrochloric acid, SG 1.18, 35% HCL (CORROSIVE, IRRITATING ACID VAPOUR) and allow any effervescence to subside. Add a further 150ml of boiling distilled water and reboil for 5 minutes or so. Allow to settle; a clear yellowish solution results.

When cool, add with stirring enough Sodium Hydroxide, 200g/l (CAUTION CORROSIVE) to cause the first permanent white [1] ppt. to form. The mother liquor will be now almost colourless or browny. If too much NaOH (aq) has been added, the reverse may be attained by the cautious addition of dilute Hydrochloride acid, 200g/l, until the precipitate is just discernable.

Reheat to boiling and add all at once 5 grams of disodium succinate - 2 - water. Allow to boil for a few minutes to coagulate the precipitated iron (iii) succinate-oxide.

When cool, transfer all of the contents to a 500ml volumetric flask and top up almost to the mark. Place in a cube tank at 20°C for ª hr or until the contents of the flask are also at 20°C. Top up to the mark with distilled water.

FILTRATION

Set up the filter apparatus as for the Chloride [(2)] analysis but use a Whatman No. 40 or 541 paper. Filter the contents of the flask whilst still hot but DO NOT wash with distilled water. The filtrate should be clear & colourless.

TITRATION

Measure out accurately 50.00ml aliquots of the Calcium solution into 250ml screw top conical flasks, add 20ml of Sodium Hydroxide, Triethanolamine solution [(3)] to the flasks and dilute to 150ml. Add about a level spatula to each flask of H.S.N. indicator [(4)] and swirl to mix.

Titrate the contents of each flask with E.D.T.A. di Sodium salt, 6.40 g/l solution, using magnetic stirring until the first permanent blue coloration is achieved [(5)] (NOT PURPLISH BLUE) until two results are within 0.1ml of each other [(6)] Record this volume (V).

$$\% \text{ CaO} = \frac{VE}{m}$$

V Volume of E.D.T.A. soln.
E Equivalent of E.D.T.A.
m Mass of sample taken

DETERMINATION OF E

Using a standard solution of 50.00ml of Ca^{2+} (aq) ions prepared by dissolving 1.000g of AR Calcium Carbonate in a little dilute Hydrochloric acid and making up to 1 litre at 20°C; titrate new batches of E.D.T.A. di Na^{+} salt soln. as above. The value E should be as close to 1 as possible.

$$E = \frac{t}{50.00}$$

t = Volume E.D.T.A. soln. to end point.

NOTES:-

1. Sometimes greyish & curdly.

2. The Buchner flask must be as dry as possible : wet the paper using the solution to be filtered.

3. 200g/L NaOH & 20% TRIETHANOLAMINE SOLUTION $(N/C_2H_4OH)_3$.

4. See B.S. 1881 under CaO.

5. A filter paper under the flask helps to see more clearly.

6. Usually 3 flasks - use the first as a rough guide - the other two being more accurate.

TITRATIONS TO ASSESS CONC. OF $Ag^{(*)}$(aq)

ALKALINE DICHROMATE (CHROMATE) METHOD

This method is inapplicable in acidic solutions (pH 5 or less).

METHOD

Using a pipette or burette, measure out accurately say 10ml of a solution of chloride ions of the same molarity as the silver solution is expected to be; (usually 0.1 Molar) into a conical flask. Add a spatular of "Anala-R" Calcium Carbonate and a few drops of "Alkaline Dichromate" indicator reagent. Dilute to 3 times volume. Titrate the silver from a second burette against the Chloride until the first dark red permanent ppt is formed - The $CaCO_3$ powder will show this up as a rather orangy tinge in the flask.

The volume of $Ag^{(+)}$(aq) should be of the same quantity as the Chloride added ie. 0.1M $Ag^{(+)}$: 0.1MCl^{-} in ratio of 1:1.

Calculation

$$\text{Molarity of } Ag^{(+)}(aq) = \frac{\text{Molarity of } Cl^{-} \times \text{Volume of } Cl^{-}(aq)}{\text{Volume of } Ag^{(+)}(aq)}$$

"Alkaline Dichromate reagent "

Take 0.01 Moles of N_2CrO_7 (Potassium dichromate)
and add 10ml of NaOH(aq) 200g L^{-1}
Make up to 500ml - 0.04m $CrO_{4(-)\,(aq)}$