C 142 (Expt. No. 7)

NAME : ____________________________________ BATCH : _______

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<u>ESTIMATION OF CALCIUM IN MILK POWDER THROUGH EDTA COMPLEXOMETRY</u>

AIM

To estimate the amount of Calcium in milk powder by complexometric titration using Ethylenediaminetetraacetc acid (EDTA).

THEORY

Calcium comprises 1.5 - 2.0 % of our body weight. It is required by the body to produce strong bones and teeth, and 99% of the calcium of our body is present in bones and teeth.

Milk is a heterogeneous mixture of proteins, sugar, fat, vitamins and minerals. Milk and milk products are some of the natural sources of calcium.

In this experiment, we will estimate the amount of calcium present in milk by complexometric titration using the well-known chelating ligand ethylenediaminetetraacetc acid (EDTA). EDTA reacts with calcium and with many other metal ions to form very stable complexes. Milk will be analyzed for its calcium content using EDTA, which reacts to form CaEDTA. The calcium in the milk reacts quantitatively with EDTA at pH 10 to form a very stable complex. EDTA titrations involve the use of an indicator such as eriochrome black-T. This is a metal ion indicator whose color changes when it binds to a metal ion. For an indicator to be useful, it must bind metal less strongly than does EDTA. A small amount of Mg^{2+} is added to the indicator (In) to form a magenta-colored complex. As EDTA is added, it reacts first with free calcium, and then with the small amount of the MgIn complex. As the final traces of calcium in the milk is being complexed, the slight excess of EDTA converts the magenta-colored magnesium complexed indicator into the free blue anion.

The titration is carried out at a pH of 10. This is because it results in increased $EDTA^{-4}$ concentration which favors complex formation.
The indicator used in this titration is eriochrome black-T. The color change of the indicator from magenta to blue occurs at pH above 7. At this pH, precipitation of hydroxide does ***not*** occur. A pH of 10 is maintained by adding a buffer composed of ammonia and ammonium chloride. Being a weak base, ammonia raises the pH by forming hydroxide ions. It also neutralizes added acids, preventing the lowering of pH, while the ammonium ion can react with any added hydroxide to form ammonia and water, thus preventing variations in pH.

$$Ca^{2+} + EDTA^{-4} \longrightarrow CaEDTA^{-2}$$

$$MgIn\ (magenta) + EDTA^{-4} \longrightarrow MgEDTA^{-2} + In^{-2}\ (blue)$$

The color change occurs over a range of 2 drops of EDTA, from magenta to purple, and to sky-blue. The color change is slow, with changes taking 2 to 3 seconds at a pH of 10.

MATERIALS REQUIRED

Milk powder, EDTA solution (0.01 M), $ZnSO_4$ solution (0.01 M), NH_4Cl, KNO_3, dilute H_2SO_4, NaOH (0.1 M), Eriochrome black-T (EBT), burette, volumetric flasks, conical flasks, beakers, pipettes.

PROCEDURE

(A) The following solutions are prepared :

1. **<u>0.01 M EDTA solution</u>**

EDTA disodium dehydrate (0.9306 g) is weighed out into a 250 ml volumetric flask, dissolved in distilled water, and the volume is made up to the mark with distilled water, by shaking.

2. **0.01 M $ZnSO_4$ solution as primary standard**

0.1614 g of zinc sulfate is weighed out into a beaker, dissolved in dilute H_2SO_4, neutralized with NaOH solution, and transferred to a 100 ml volumetric flask. The volume is made up to the mark with distilled water, by shaking.

3. **Ammonia – Ammonium Chloride solution as buffer (pH = 10)**

17.5 g of ammonium chloride is mixed with 142 ml of concentrated ammonia (sp. gr. = 0.88–0.90), and the mixture made up to 250 ml with distilled water.

4. **Indicator**

0.030g of EBT is mixed with KNO_3 Solid mixture containing 1% of indicator.

(B) **Determination of strength of EDTA solution**

10 ml of standard $ZnSO_4$ solution is pipetted out into a 250 ml conical flask. To this solution, 5 ml of $(NH_4Cl + NH_4OH)$ buffer solution is added, and the solution diluted to 100 ml with distilled water. This solution is warmed to about $40^{o}C$. The indicator solution (5 drops) is added, and the mixture shaken to obtain a wine-red color. This solution is titrated with EDTA solution until the wine-red color changes to blue. The titration is repeated to get concordant values.

Table 1 **Determination of strength of EDTA solution**

Volume of $ZnSO_4$ used = 10 ml

Sl. No.	Initial Reading (ml)	Final Reading (ml)	Titre Value (ml)
1.			
2.			
3.			

$$\textbf{Strength of EDTA } (S_1) = \frac{V_{ZnSO4} \times S_{ZnSO4}}{V_{EDTA}}$$

(C) <u>Estimation of Calcium in Milk Powder</u>

3.0 g of dry milk powder is weighed out into a 100 ml beaker. Approximately 100 ml of warm distilled water is added, and the solution is stirred so as to ensure complete dissolution.

This solution is transferred, ***quantitatively***, **by repeated washings with distilled water**, into a 100 ml volumetric flask. The solution is made up to the mark with distilled water.

10 ml of the milk solution is pipetted out into a 250 ml conical flask. To this solution, 5 ml of ($NH_4Cl + NH_4OH$) buffer solution is added, and the solution diluted to 100 ml with distilled water. This solution is warmed to about $40^{o}C$. The indicator solution (5 drops) is added, and the mixture shaken to obtain a wine-red color. This solution is titrated with EDTA solution until the wine-red color changes to blue. The titration is repeated to get concordant values.

Table 2 <u>Determination of strength of Calcium in Milk solution</u>

Volume of milk solution used = 10 ml

Sl. No.	Initial Reading (ml)	Final Reading (ml)	Titre Value (ml)
1.			
2.			
3.			

$$\textbf{Strength of } Ca^{2+} \ (S_2) = \frac{V_{EDTA} \times S_{EDTA}}{V_{Ca^{2+}}}$$

[<u>NOTE</u> : Strength of EDTA (from Table 1) = S_1]

CALCULATIONS

$$Ca^{2+} \text{ (in grams)} = \frac{0.04 \times V_{EDTA} \times S_{EDTA} \times 1000}{f}$$

f = number of grams of milk powder in 100 ml of solution.

RESULT

Ca^{2+} present = ………………….