# Titration of Calcium Ion with EDTA

**Objective**

The amount of calcium ion in a dietary calcium supplement will be determined by titration with the standard ethylenediaminetetraacetic acid solution (EDTA).

**Introduction**

The concentration of calcium ion in the blood is very important for the development and preservation of strong bones and teeth. Absorption of too much calcium in the blood may lead to the leaching of calcium from the bones and teeth. The absorption of calcium is controlled, in part, by Vitamin D.

Growing children in particular need an ample supply of calcium for the development of strong bones. Milk, which is a good natural source of calcium and which is consumed primarily by children, is usually fortified with Vitamin D to promote calcium absorption. Similarly, outdoor recess for elementary school children is mandated by law in many states because exposure to sunshine allows the body to synthesize Vitamin D.

In later adulthood, particularly among women, the level of calcium in the blood may decrease to the point where calcium is removed from the bones and teeth to replace the blood calcium, making the bones and teeth much weaker and more susceptible to fracture. This condition is known as osteoporosis and can become very serious, causing shrinking of the skeleton and severe arthritis. For this reason, calcium supplements are often prescribed in an effort to maintain the proper concentration of calcium ion in the blood. Although the results are not yet conclusive, recent reports indicate 100 mg of calcium ion, taken orally on a daily basis, may serve to prevent osteoporosis. Typically, such calcium supplements consist of calcium carbonate, $CaCO_3$.

A standard analysis for calcium ion involves *titration* with a standard solution of EDTA. In a titration experiment, a standard reagent of known concentration is added slowly to a measured volume of a sample of unknown concentration until the reaction is complete. From the concentration of the standard reagent, and from the volumes of standard and unknown taken, the concentration of the unknown sample may be calculated. In titration experiments, typically the sample f the unknown concentration is measured with a pipet, and the volume of standard solution required is measured with a buret (see Experiment 2 for a review of volumetric glassware).

EDTA is a type of molecule called a complexing agent, and is able to form stable, stoichiometric (usually 1:1) compounds with many metal ions. Reactions of EDTA with metal ions are especially sensitive to pH, and typically a concentrated buffer solution is added to the sample being titrated to maintain a relatively constant pH. A suitable indicator, which will change color when the reaction is complete, is also necessary for EDTA titrations.

**Safety Precautions**

- Safety eyewear approved by your institution must be worn at all times while you are in the laboratory, whether or not you are working on the experiment.
- The hydrochloric acid and the ammonia buffer solution used in this experiment are damaging to skin and eyes. Wash after handling. Inform the instructor if these reagents are spilled.
- Ammonia is a strong cardiac stimulant and respiratory irritant. Keep the ammonia buffer solution in the fume exhaust hood. Add the ammonia buffer to your sample in the fume exhaust hood. Do not bring the ammonia buffer into the room until it has been added to your sample (which will dilute it).
- Eriochrome T indicator is toxic and will stain skin and clothing. Wash after using. Clean up all spills. The indicator is a slurry and must be shaken prior to dispensing.
- Use a funnel when adding titrant to the buret, and place the top of the buret *below eye level* when filling.

**Apparatus/Reagents Required**

Buret and clamp, 25-mL pipet, 250-ml volumetric flask, 3 *M* HCl, standard 0.0500 *M* EDTA solution, Eriochrome Black T indicator, calcium supplement tablet.

**Procedure**

Record all data and observations directly on the report pages in ink.

**1. *Dissolving of the Calcium Supplement***

- Obtain a calcium supplement tablet. Record the calcium content of the tablet as listed on the label by the manufacturer. Place the tablet in a clean 250-mL beaker.

- Obtain a 25-mL of 3 *M* HCl solution in a graduated cylinder. Over a 5-minute period, add the HCl to the beaker containing the calcium tablet in 5-mL portions, waiting between additions until all frothing of the tablet has subsided. After the last portion of the HCl has been added, allow the calcium mixture to stand for an additional five minutes to complete the dissolving of the tablet. Since such tablets usually contain various binders, flavoring agents, and other inert material, the solution may *not* be entirely homogeneous.

- While the tablet is dissolving, clean a 250-mL volumetric flask with soap and tap water. Then rinse the flask with two 10-mL portions of distilled water. Fill your plastic wash bottle with distilled water.

- Taking care not to lose any of the mixture, transfer the tablet solution to the volumetric flask using a small funnel. To make sure that the transfer of the calcium solution has been complete, use distilled water from the plastic wash bottle to rinse the beaker that contained the calcium sample into the volumetric flask. Repeat the rinsing of the beaker twice more.

- Use a stream of distilled water from the wash bottle to thoroughly rinse any adhering calcium solution from the funnel into the volumetric flask, and then remove the funnel.

- Add distilled water to the volumetric flask until the water level is approximately 1-inch below the calibration mark on the neck of the volumetric flask. Then use a medicine dropper to add distilled water until the bottom of the solution meniscus is aligned exactly with the volumetric flask's calibration mark.

- Stopper the volumetric flask securely (hold your thumb over the stopper) and then invert and shake the flask 10-12 times to mix the contents.

***2. Preparation for Titration***

- Set up a 50-mL buret and buret clamp. Check the buret for cleanliness. If the buret is clean, water should run down the inside walls in sheets, and should not bead up anywhere. If the buret is not clean enough for use, place approximately 10-mL of soap solution in the buret and scrub with a buret brush for several minutes.

- Rinse the buret several times with tap water, and then check again for cleanliness by allowing water to run from the buret. If the buret is still not clean, repeat the scrubbing with soap solution. Once the buret is clean, rinse it with small portions of distilled water, allowing the water to drain through the stopcock.

- Obtain a 25-mL volumetric pipet and rubber safety bulb. Using the bulb to provide suction, fill the pipet with tap water and allow the water to drain out. During the draining, check the pipet for cleanliness. If the pipet is clean, water will not bead up anywhere on the interior during draining.

- If the pipet is not clean, use the rubber bulb to pipet 10-15 mL of soap solution. Holding your fingers over both ends of the pipet, rotate and tilt the pipet to rinse the interior with soap for 2-3 minutes. Allow the soap to drain from the pipet, rinse several times with tap water, and check again for cleanliness. Repeat the cleaning with soap if needed. Finally, rinse the pipet with several 5-10 mL portions of distilled water.

- Obtain 200mL of standard 0.0500 *M* EDTA solution in a 400 mL beaker. Keep the EDTA solution covered with a watch glass when not in use.

- Transfer 5-10 mL of the EDTA solution to the buret. Rotate and tilt the buret to rinse and coat the walls of the buret with the EDTA solution. Allow the solution to drain through the stopcock to rinse the tip of the buret.

- Rinse the buret twice more with 5-10 mL portions of the standard EDTA solution.

- After the buret has been thoroughly rinsed with EDTA, fill the buret to slightly above the zero mark with the standard EDTA.

- Allow the buret to drain until the solution level is slightly below the zero mark. Read the volume of the buret (to two decimal places), estimating between the smallest scale divisions. Record this volume as the initial volume for the first titration to be performed.

- Rinse four clean 250-mL Erlenmeyer flasks with distilled water. Label the flasks as samples 1, 2, 3, and 4.

- Transfer approximately 50 mL of your calcium solution from the volumetric flask to a clean, *dry* 150-mL beaker. Using the rubber bulb for suction, rinse the pipet with several 5-10 mL portions of the calcium solution to remove any distilled water still in the pipet.

- Pipet exactly 25 mL of the calcium solution into each of the four Erlenmeyer flasks.

**3. Titration of the Calcium Samples**

- Each sample in this titration must be treated one at a time. Do not add the necessary reagents to a particular sample until you are ready to titrate. Because the buffer solution used to control the pH of the calcium samples during the titration contains concentrated ammonia, it will be kept stored in the exhaust hood. Take calcium sample 1 to the exhaust hood and add 10 mL of the ammonia buffer.

- At your bench, add 5 drops of Eriochrome T indicator solution to calcium sample 1. The sample should be wine-red at this point; if the sample is blue, consult the instructor.

- Add EDTA from the buret to the sample a few milliliters at a time; with swirling after each addition, and carefully watch the color of the sample. The color change Eriochrome T is from red to blue, but the sample will pass through a gray transitional color as the endpoint is neared.

- As the sample becomes gray in color, begin adding EDTA *dropwise* until the pure blue color of the endpoint is reached. Record the final volume used to titrate sample 1 t two decimal places, estimating between the smallest scale divisions on the buret.

- Refill the buret, and repeat the titration procedure for the three remaining calcium samples. Remember not to add ammonia buffer or indicator until you are actually ready to titrate a particular sample.

**4. Calculations**

- For each of the four titrations, use the volume of EDTA required to reach the endpoint and the concentration of the standard EDTA solution to calculate how many moles of EDTA were used. Record.

- Moles EDTA = (volume used to titrate in liters) x (molarity)

- Based on the fact that the calcium/EDTA reaction is 1:1 stoichiometry, calculate how many moles of calcium ion were present in each sample titrated. Record.

- Using the atomic mass of calcium, calculate the mass of calcium ion present in each of the four samples titrated, and the average mass of calcium present.

- Based on the fact that the average mass of calcium calculated above represents a 25-mL sample, taken from a total volume of 250 mL used to dissolve the tablet, calculate the mass of calcium present in the original tablet.

- Compare the mass of calcium present in the tablet calculated from the titration results with the nominal mass reported on the label by the manufacturer. Calculate the percent difference between these values.

**Questions**

1. Most calcium supplements consist of *calcium carbonate*, $CaCO_3$, since this substance is readily available and relatively cheap. Based on your experimentally determined average mass of calcium ion, calculate the mass of calcium carbonate that would be equivalent to this amount of calcium ion.

2. Suggest at least two reasons why your experimentally determined amount of calcium might differ from that listed by the manufacturer of the calcium supplement you used.

3. Use your textbook or an encyclopedia of chemistry to write at least three *natural* sources of calcium in that should be included in the diet.

**Pre-Laboratory Questions**

1. Using you textbook or an encyclopedia of chemistry, discuss some uses of calcium I the human body.
2. The reagent used as titrant in this experiment, EDTA, is a complexing agent for calcium and for many other metal ions. Use your textbook or an encyclopedia of chemistry to write a definition of this term.
3. The pH of the samples in this experiment will be adjusted by the use of a concentrated **buffered solution**. Use your textbook or an encyclopedia of chemistry to write what is meant y a buffered solution and give an example of such a solution.

**Results/ Observations**

Dissolving of the Calcium Supplement
Observation:

Did the tablet dissolve *completely*?
Was the solution of the tablet *homogeneous*?

Titrations
Concentrations of *standard* EDTA solution used, *M*
Volume of calcium solution taken for titration, mL

| ***Sample*** | ***1*** | ***2*** | ***3*** | ***4*** |
|---|---|---|---|---|
| Final volume EDTA, mL | | | | |
| Initial volume EDTA, mL | | | | |
| Volume EDTA used, mL | | | | |
| Moles EDTA used | | | | |
| Moles $Ca^{2+}$ | | | | |
| Mass of $Ca^{2+}$ present, g | | | | |

Average mass of $Ca^{2+}$ present, g
Average mass of $Ca^{2+}$ present in original tablet, g
Listed mass of $Ca^{2+}$ present in tablet (from label)
Percent difference between experimental mass and listed mass