



# Utilization of Used-Up Stationery Product Chalk for Developing New Experiment

Sudesh Bhaskar Ghoderao

Associate Professor

RNC Arts, JDB Commerce and NSC Science College, Nashik Road, Nashik, India

## Abstract

The compounds of calcium are widely used in various types of materials like construction materials, agricultural and medicinal products, stationery materials etc. It is, however, present along with other metal ions. It is very important to select such a material for setting an experiment in quantitative analysis of calcium only. The analysis of chalk is ideal in this respect. The piece of chalk smaller than length of 1 cm finds no use for writing is a cheap, easily and commonly available used-up consumer product which is relatively pure and suitable for this experiment. This new experiment requires one laboratory period, if any of the two methods, complexometry or permanganatometry is used. The students can compare their results with the expected value as well as amongst themselves, therefore; it will create interest in the minds of students for the study of consumer products.

**Key words:** Chemistry Education, Pedagogy, Low Cost Experiment, Determination of Calcium

## Introduction

Calcium is widely distributed and exceedingly important element occurring in nature and forms about 3 - 5 % of the earth's crust<sup>1</sup>. The precipitated calcium sulphate, which is generally  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ , is employed in the preparation of chalk pencils<sup>2</sup>. A piece of chalk in general measures about 7 to 8 cm. But being brittle it breaks into pieces during its use for writing purpose. The smaller piece finds no use for writing, as one cannot hold such a small piece in hand comfortably. The material being soft also produces powder during the use. The pieces and powder are considered as waste and can be used for quantitative determination of calcium. In an experiment of qualitative analysis using simple chemical and physical tests, eleven household compounds are mentioned of which one is white chalk<sup>3</sup>. There is no experiment for quantitative determination of calcium, the major constituent of chalk in the university syllabus. So in the present work is given such an exercise in titrimetry. It is done by complexometry (method I) as well as by permanganatometry i.e. redox titrations (method II)<sup>4</sup>.

The chalk material is soluble in acids<sup>5</sup> and so is dissolved in hydrochloric acid (1:1) by heating the solution. The water insoluble calcium sulphate is converted, here, into soluble calcium chloride. The insoluble material, if remains, is the binder material which is removed by filtration and the filtrate is diluted to known volume. The standardization

of EDTA is done using standard metal ion solution as of  $Zn^{2+}$  using Eriochrome Black T indicator and the pH is maintained using a buffer of pH=10 during determination of calcium.

In permanaganatometry, the calcium is precipitated from an aliquot as calcium oxalate using ammonium oxalate. Satisfactory results are obtained by adding ammonium oxalate to hot acidic solution of calcium ions and finally neutralizing with aqueous ammonia solution. The precipitate formed, after digesting for about half an hour, consists of relatively coarse crystals which are readily filtered and washed with dilute solution of ammonium oxalate owing to common ion effect. The precipitate then washed with dilute water and then dissolved in hot dilute sulphuric acid. The liberated oxalic acid is titrated against previously standardized potassium permanganate solution. This is done to find the amount of calcium ions equivalent to oxalate ions liberated in dissolution of the precipitate of calcium oxalate. In the titration with previously standardized potassium permanganate the oxidation of oxalate is slow at the beginning, but becomes faster once reaction has started. This is done through heating at  $60^{\circ}C$  or by the catalytic activation due to  $Mn(II)$  ions<sup>6</sup>.

### Chemicals required

The chemicals used are of AR Grade and standard procedures are used for the preparation of required reagent solutions.

### Disintegration of sample

Hydrochloric acid (1:1)

### Method I (Complexometry)

EDTA (0.01 M approx.), Eriochrome Black T (1%), Ammonia-ammonium chloride buffer solution (pH - 10), EDTA (0.2 M),  $Zn^{2+}$  solution (0.01 M)

### Method II (Permanganatometry)

Hydrochloric acid (1:1), Ammonium oxalate (6 %), Dilute ammonia (1:1), Methyl red indicator (0.1 %), Sulphuric acid (1:8), Sulphuric acid (3 M),  $KMnO_4$  solution (~0.025 N),  $Na_2C_2O_4$ , (std. 0.025 N), Ammonium oxalate (1 %), Manganese sulphate (0.2 M)

### Experimental Procedure

#### Disintegration of sample

Accurately weighed sample (- 0.4 g) is transferred to a 100 mL-capacity beaker. To it 10 mL of water and 10 mL of hydrochloric acid (1:1) is added. The contents of beaker are heated to dissolve. Any insoluble material, if remains, is filtered. The residue is given the washings till free from acid. The filtrate and washings are diluted to 250 mL in a volumetric flask.

## Method I (Complexometry)

### Standardization of EDTA

An aliquot of 10 mL of standard zinc ion solution (0.01 M) is taken in a conical flask with pipette and neutralized with dilute ammonia (1:1). To it is added 10 mL distilled water, 2 mL buffer (pH 10) and Eriochrome Black-T indicator. The solution is titrated versus EDTA (approx. 0.01 M) till the end point as wine red to blue is obtained.

### Determination of calcium

An aliquot of 25 mL diluted solution of chalk is taken in a conical flask and neutralized with dilute ammonia. To it 10 mL water, 2 mL ammonia-ammonium chloride buffer solution (pH=10), 1 mL Mg-EDTA complex and Eriochrome Black-T indicator are added. The solution is titrated against previously standardized EDTA solution taken in a burette till the end point as blue to wine red. The amount of  $\text{Ca}^{2+}$  is determined by using equation, 1 mL of 1M EDTA = 40.08 mg of  $\text{Ca}^{2+}$ .

## Method II (Permanganatometry)

### Standardization of $\text{KMnO}_4$ solution:

An aliquot of 10 mL of standard sodium oxalate solution (0.025N) is taken in the titration flask. After adding 10 mL of sulphuric acid (3M) it is warmed and titrated against  $\text{KMnO}_4$  solution until the permanent pink colour is obtained. From the titration reading exact normality of  $\text{KMnO}_4$  solution is calculated.

### Determination of calcium

An aliquot of 100 mL of the chalk solution is taken in a 100 mL beaker. To it 3-4 drops of methyl red Indicator is added. The solution is heated to boiling and then 30 mL of ammonium oxalate (6 %) solution is added slowly and with constant stirring. To this hot solution (70°-80 °C), ammonia (1:1) is added drop wise from a burette and with constant stirring until solution is neutral or faintly alkaline (red to yellow). This solution is allowed to stand for 30 minutes in a warm place. The precipitate is filtered through Whatman filter paper No. 41. It is washed initially with ammonium oxalate (1 %) followed by water till the washings are free from chloride. The filtrate is rejected. The precipitate is dissolved in about 40 mL of hot sulphuric acid (1:8) and the paper is washed by hot water. The solution and washings are diluted to 100 mL in a volumetric flask.

An aliquot of 10 mL of this diluted solution (containing liberated oxalic acid) is taken in a conical flask and 25 mL of sulphuric acid (3 M) is added. The contents of flask are heated to 60-70°C, or are added 5 mL of manganese sulphate (0.2 M) before start of titration and then titrated it against previously standardized  $\text{KMnO}_4$  solution (0.025 N) till a permanent pink colour appears. The amount of  $\text{Ca}^{2+}$  is determined by using equation, 1 mL of 1N  $\text{KMnO}_4$  = 20.04 mg of  $\text{Ca}^{2+}$ .

**Result Table:** Determination of  $\text{Ca}^{2+}$  in chalk sticks

Sample Code	Manufacturer	Amount of Sample, g	Method I Complexometry		Method II Permanganatometry	
			Titer mL EDTA (0.01M)	Calculated % Ca	Titer mL $\text{KMnO}_4$ (0.02525N)	Calculated % Ca
Chalk C1 White	Ravi Sanket, Katraj, Pune	0.42535	24.2	22.80	8.0	23.79
Chalk C2 White	Udygdham, Talegaon Dabhade	0.38513	23.8	24.76	7.5	24.63
Chalk C3 Yellow	Udygdham, Talegaon Dabhade	0.41891	23.8	22.77	7.7	23.25

**Conclusion**

This experiment can be given to undergraduate students. It will help to familiarize the students with techniques such as precipitation and volumetric analysis. They can also learn the theoretical background and comparative aspects of two titrimetric methods of estimation of  $\text{Ca}^{2+}$ . The composition of chalk is not given by any manufacturer. So the students can be asked to calculate the percentage of Ca in the chalk.

The study indicates that all the chalks have almost identical composition. The results obtained by two different methods are also comparable. The samples C2 and C3 are from the same manufacturer; but of white and yellow colour respectively. The percentage of  $\text{Ca}^{2+}$  in white coloured chalk is more than yellow coloured chalk, which may be due to the colouring matter added to later. This matter being insoluble in the disintegration step is separated in the filtration process.

For the disintegration of sample less time is required. So the total experiment requires, only one laboratory period, if any of two methods given is used. The students can compare their results with the expected value as well as amongst themselves and can extend this study to 'error analysis' too. The experiment, therefore; will create interest in the minds of students for the study of consumer products.

**Acknowledgements:** Author is thankful to Dr. V.D.Kelkar, Ex faculty of SPPU, Pune for his guidance and The Principal, SMRK AK Mahila Mahavidyalaya, Nashik for providing necessary facilities during this work.

**References:**

1. Hazela, K.C. and Pandey, O.P. 'Advanced Study of Metals', Kishore Publishing House, Knapur (1974), p.105
2. De, A.K., 'A textbook of Inorganic Chemistry', 6<sup>th</sup> ed., Wiley Eastern Ltd., New Delhi (1983), pp. 260-261
3. Solomone, S., Fiilep, A. – Poszmik and Lee, A., J. Chem. Educ., 68, 328, (1991)
4. Vogel, A.I., 'A Textbook of Quantitative Inorganic Analysis', ELBS, 3<sup>rd</sup> ed., (1973), (a) p.296
5. Dean, J. A., Ed. 'Lange's Handbook of Chemistry', 13<sup>th</sup> ed., McGraw Hill International ed., Chemistry Series (1987), p.4.37 (Table 4.1)
6. Cullis, C.F., Ladburg, J.W., J. Chem. Educ., 32, 555, (1955)