行政院國家科學委員會專題研究計畫 成果報告

開發普通化學實驗課程的消費者化學實驗並評估其可行性

<u>計畫類別</u>: 個別型計畫 <u>計畫編號</u>: NSC92-2511-S-018-010-<u>執行期間</u>: 92 年 08 月 01 日至 93 年 10 月 31 日 執行單位: 國立彰化師範大學化學系暨研究所

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開發普通化學實驗課程的消費者化學實驗並評估其可行性

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Complexometric Titration of Aluminum and Magnesium Ions in Commercial Antacids: An Undergraduate Chemical Experiment

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Abstract

Chemicals required for chemistry laboratory are mostly obtained from chemical companies. Few chemical experiments use reagents from commercial sources. Nowadays, students' interest in chemistry is usually suffered from the lack of relevance of chemistry lectures and laboratories to their daily lives, which ultimately lead to students' insufficient abilities in chemical problem solving. This article presents a newly designed experiment for the determinations of metallic ions in commercial antacids using uncommon titrations, suitable for general chemistry laboratory and introductory quantitative analysis laboratory curriculums.

This experiment involves three new protocols designed for the determination of aluminum and magnesium contents in commercial antacids by complexometric titration and back titration. This newly developed experiment is very different from the quantitative analysis of acid-neutralizing power of anions in antacids described in textbooks. In the experiment, students can learn various concepts and techniques of sample preparation, complexometric titration and its back titration, metallic indicators, uses of buffer solution, complex formations, masking, and blocking.

Results indicate three protocols are no significant difference in average and precision by different analysts between students and instructor. Besides, results show there are no significant difference in average and precision of different protocols between experiment and calculation performed by instructor, and results point out the same as by students. These results verify the feasibility of three protocols. In general, student comments on the experiment designs are highly positive.

Keywords: Antacids, Consumer Chemistry, Complexometric Titration, Complexometric back Titration, Quantitative Analysis, General Chemistry Laboratory and Chemical Education Research

利用錯合滴定法測定制酸劑金屬離子的含量:一個適用於普通化學實驗課程的實驗

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中文摘要

普通化學實驗的藥品大多數取自於化學藥品公司,鮮少利用消費者產品。這個缺點是實驗的探究過程過於理想化,也無法融入真正的生活之中,因而學生學習的興趣低落,以致解決化學的能力不足。本研究的目的是開發一個制酸劑金屬離子含量的定量分析,此實驗非常有別於傳統的制酸劑之制酸力測定。

在此文章中,描述一個被分為三種分析方法的實驗設計,此是利用錯合滴定法和錯合逆 滴定法來測定制酸劑金屬離子的含量,此實驗含有一份新開發的實驗教材適用於普通化學實 驗和分析化學實驗兩課程。學生能夠學到有關樣品製備、錯合滴定法、錯合逆滴定法、金屬 指示劑、緩衝溶液、錯合物形成、遮蔽和阻塞。

藉由統計分析來評估此實驗的精確度和準確度,結果顯示:由不同的分析者利用此三種 分析方法測定制酸劑金屬離子的含量,學生與老師之間沒有顯著的差異。再者,由老師利用 此三種分析方法的不同方式測定其含量,實驗方式和計算方式的比較結果兩者之間沒有顯著 的差異。學生的結果顯示亦復如此。這些結果證實此實驗的可行性。學生對此實驗的反應也 持高度正面的評價。

關鍵詞:制酸劑、消費者化學、錯合滴定法、錯合逆滴定法、定量分析、普通化學實驗和化 學教育研究。

Complexometric Titration of Aluminum and Magnesium Ions in Commercial Antacids: An Undergraduate Chemical Experiment

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Introduction

Chemicals required for chemistry laboratory are mostly obtained from chemical companies. Few chemical experiments use reagents from commercial sources. Nowadays, students' interest in chemistry is usually suffered from the lack of relevance of chemistry lectures and laboratories to their daily lives, which ultimately lead to students' insufficient abilities in chemical problem solving. This article presents a newly designed experiment for the determinations of metallic ions in commercial antacids using uncommon titrations. This newly developed experiment is very different from the quantitative analysis of acid-neutralizing power of anions in antacids described in textbooks.

At present, a few consumer products are used in traditional analysis for general chemistry laboratory experiments. For example, white vinegar is used for acid-base titration (1-3). Vitamin tablet and bleaching powder are applied for oxidation-reduction titration (4-6), while aspirin tablet, cola drinks and margarine are employed for spectrophotometric determination (2, 7-8). Recently, little household chemical products suitable for introductory quantitative analysis have been published in literatures of chemical education. Representative examples are imposter perfumes (9), pop rocks candy (10), coffee (11), and diet tonic water (12).

The determination of acid-neutralizing power in antacids by acid-base back titration is a popular experiment for general chemistry laboratory. The experiment is intended to learn about acid-base chemistry and titration technique. It can be found easily in many textbooks of general chemistry laboratory (e.g. 2, 5-6, 13-15) and on the Internet (such as 16-18). Besides, examples of rating antacids (19) and identification the brand name of antacids (20) published in journals are suitable for the chemistry laboratory course.

Complexometric titration is a quantitative analysis used for general and analytical chemistry laboratory. The direct titration for the determination of total hardness of water is a well-known experiment, which is compiled in a number of textbooks (2, 21-23). Also, the titration is employed for the analyses of samples containing metallic ions, some of which found on the websites (such as 24-26). In the literature of chemical education, the titration is utilized for the determination of household products including water (27-28), cold lozenge (29), and shower cleaner (30). Moreover, it is still a quantitative method used for chemical research (31-32). In contrast, complexometric back titration suitable for instruction has yet been found in textbooks and literatures of chemical education.

This experiment involves three new protocols designed for the determination of aluminum and magnesium contents in commercial antacids by complexometric titration and its back titration. It is suitable for general chemistry laboratory and introductory quantitative analysis laboratory curriculums. In this experiment, students can learn about various concepts and techniques on sample preparation, complexometric titration and its back titration, metallic indicators, buffer solution, complex formations, masking, and blocking.

Ideas for Protocols

Antacids may be divided into two classes, (a) chemical antacids that work by chemical neutralization of gastric acid, most notably sodium bicarbonate; and (b) adsorptive ones that act by adsorption of the acid, including aluminum and magnesium salts, and calcium carbonate. According to data from Medline Plus related to antacids (See the Supplementary material) produced in the U. S., of the total 80 common brands, those with aluminum and magnesium ions are in majority, whereas those containing calcium represent a minor production. It is conceivable that calcium antacids may lead to constipation and renal stone formation. Therefore, the designed experiment is only intended to the determinations of aluminum and magnesium but not calcium.

Antacids containing alumina and aluminum hydroxide (or the dried gel form) are extremely insoluble in water. Hence, sample preparation prior to quantitative analysis is necessary. To dissolve aluminum ion, antacids will be mixed with dilute hydrochloric acid under heating.

The three protocols can be used separately to determine (a) the total aluminum and magnesium ions, (b) the alone aluminum ion, and (c) the alone magnesium ion. In tradition, an unknown metallic determination can be calculated by subtracting the total contents from a known amount of a metal. Its shortcoming is that the calculated value may be incorrect, if, for example, one of the two determinations has errors. In this experiment, student will be obtain metallic ions form the three protocols rather than calculation.

Aluminum ion is best determined by complexometric back titration with a heating condition to enhance the complexation between aluminum ion and EDTA. The back titration is used when a cation forms a stable complex with EDTA in a slow reaction or an indicator is blocked when it forms a metal ion complex whose stability constant is greater than that of the metal-EDTA complex. To clearly observe the endpoint in a low pH buffer solution, the back titration with standardized zinc sulfate solution is preferable to that with calcium solution.

To determine the total aluminum and magnesium contents in antacids, the antacid sample is kept at a pH 10.0 buffer solution followed by adding an excess known amount of EDTA. Heating the solution prior to the addition of indicator, Calmagite, is necessary to ensure that it is not blocked by EDTA. In such buffer solution, the two metallic ions can complex readily with excess EDTA. The amount of unchelated EDTA can be determined by the back titration with standardized zinc sulfate solution.

The separate aluminum amount in the antacid sample can also be quantitatively analyzed by complexometric back titration. In the analysis, the antacid sample solution is controlled at pH 5.0. An excess known amount of EDTA is then added to the solution. At this low pH, the Al-EDTA complex can format, whereas the Mg-EDTA formation is inhibited. Heating prior to the addition of indicator, xylenol orange, is imperative to prevent EDTA by blockage and to facilitate the complexation between EDTA and aluminum ion. The amount of unchelated EDTA can be determined by the back titration with standardized zinc sulfate solution.

For the magnesium ion determination, the antacid sample is placed in a pH 10.0 buffer solution followed by adding a large amount of triethanolamine to mask the Al-EDTA complexation. No heating is required for this direct titration.

To help students understanding complicated concepts on complexometric back titration, three tables are prepared to illustrate the changes of metallic ions and chelating agents during the metallic determinations as shown in the Supplementary Material. Change in the number of millimoles during alone aluminum determination is shown in Table 1.

Fable 1. A Schematic Summar	y of Alone Aluminum Protocol
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Sequence	Metallic ions	Millimoles	Chelating agents
1	Al ³⁺ and Mg ²⁺ (in sample)		



Experimental Procedure

In this experiment, detailed safety precautions, waste disposal, good practices, and reagent preparation are provided in the Supplementary Material. The reagent preparation contains two standard solutions of EDTA and zinc sulfate, two buffer solutions of acetate-acetic acid and bicarbonate-carbonate, as well as two indicators of xylenol orange and Calmagite.

Sample Preparation

- 1. Obtain an antacid tablet from your instructor and record its brand name, active ingredient and the claimed quantity of each component.
- 2. Weigh the tablet precisely to the nearest 0.0001 g (denoted as w_{tab}). Grind it in a clean and dried mortar and pestle to make a powder as fine as possible. Remove most portion of the powder on a weighing paper placed on a tared balance and precisely weigh it (denoted as w_{pow}). Transfer the powder quantitatively to a clean 250 mL of Erlenmeyer flask containing about 100 mL of deionized water and about 6 mL of 6 M. hydrochloric acid.
- 3. Boil gently the mixture for about 20 minutes on a hot plate. Place a stem funnel to the flask mouth so that the vapor can condense quickly back to water, which helps washing down the powder sticks on the flask wall. If any powder still remains on the flask wall, wash it down with a small amount of deionized water and continue heating.
- 4. Remove the flask from the hot plate and allow it to cool to room temperature or rapidly in a water bath. Filter the mixture by gravity filtration into a 250 mL of volumetric flask. Rinse the flask and solid on the filter paper with about 10 mL of deionized water twice to make sure that all metallic ions are transferred into the volumetric flask.
- 5. Dilute the solution to the calibration mark with deionized water. Stopper the flask and mix the solution well by inverting and shaking it repeatedly. Label this solution "The antacid sample solution, 250.00 mL" (denoted as V_{pow}).

Determination Procedure

Part A: Total Aluminum and Magnesium Protocol

- 1. Pipet a 10.00 mL aliquot of the antacid sample solution to a 125 mL of Erlenmeyer flask followed by about 10 mL of the bicarbonate-carbonate pH 10 buffer solution. Transfer quantitatively a 35.00 mL aliquot of the EDTA standard solution using a buret to the flask (denoted as V_{EDTA}).
- 2. Boil gently the mixture for 5 min. on a hot plate to speed up the formation of Al-EDTA complex. Add 5 drops of Calmagite indicator and mix it well. The solution should appear pure blue in color. If the EDTA is not enough to chelate completely all metallic ions, the solution should be wine red in color at this moment. Put an additional 5.00 or more mL aliquot of the EDTA solution to this wine red solution. Boil again until the color changes to

pure blue. Continue this step if necessary.

- 3. Back-titrate the solution with standardized zinc sulfate solution until the color changes to purple at the endpoint (no wine red color should persist). Record the volume used (V_{Zn}) .
- 4. Repeat the titration twice. The data of V_{Zn} should agree within 1% of the relative average deviation. Otherwise, repeat the titration and then average all results using the Q-test to reject any outliers.
- 5. Calculate the combined total number of millimoles of aluminum and magnesium ions in the antacid sample solution and in the tablet.

Part B: Alone Aluminum Protocol

- 1. Pipet a 10.00 mL aliquot of the antacid sample solution to a 125 mL of Erlenmeyer flask. Add about 10 mL of the acetate-acetic acid pH 5 buffer solution to mask the formation of Mg-EDTA complex. Transfer quantitatively a 25.00 mL aliquot of the EDTA standard solution using a buret to the flask (denoted as V_{EDTA})
- 2. Boil it gently on a hot plate for 5 min. to speed up the formation of Al-EDTA complex. Add 5 drops of xylenol orange indicator and mix well. The solution should appear lemon yellow in color at this moment. If the EDTA is not enough to completely chelate all alumnum ions, the solution should be deep red in color. Put an additional 5.00 or more mL aliquot of the EDTA solution to this deep red solution. Boil again until the color changes to lemon yellow. Continue this step if necessary.
- 3. Back-titrate the solution with standardized zinc sulfate solution until the color changes to light red at the endpoint (no deep red color should remain). Continuously titrate the solution until a light red color persists for more than 3 minutes if the light red color shortly turns back to lemon yellow. Slow titration will give good results. Record the volume used (V_{Zn}).

Note: The turning back to lemon yellow color is the consequence of small quantity of the EDTA slowly shifted to an active polydentate species in accordance with Le Chatelier's Principle because the chelating ability of EDTA with zinc ions is reduced at a low pH.

- 4. Repeat the titration twice. The data of V_{Zn} should agree within 1% of the relative average deviation. Otherwise, repeat the titration and then average all the results using the Q-test to reject any outliers.
- 5. Compute the number of millimoles and weights of aluminum present in the sample solution and the tablet.

Part C: Alone Magnesium Protocol

- 1. Pipet a 10.00 mL aliquot of the antacid sample solution to a 125 mL of Erlenmeyer flask followed by about 10 mL of the bicarbonate-carbonate pH 10 buffer solution. Add about 3 mL of triethanolamine and swirl the mixture for 2 min. to enhance the formation of Al-triethanolamine complex such that the formation of Al-EDTA complex is masked. Stand it for a while until the turbid solution becomes mostly clear for easy observation of endpoint. Add 5 drops of Calmagite indicator and mix well. The solution should appear wine red in color.
- 2. Direct-titrate the solution with the EDTA standard solution until the color changes to pure blue at the endpoint. Record the volume used (V_{EDTA}). A trial titration first is recommended to get an ideal result. Add most portion of the EDTA solution at a time and then carefully titrate it dropwise near the endpoint.

Note: Quick titration will give a good result. If the titration is slow, the aluminum ion will be

released from the Al-triethanolamine complex and produce the Al-Calmagite complex of wine red in color. In such case, continuing titration will give a positive error.

- 3. Repeat the titration twice. The data of V_{Zn} should agree within 1% of the relative average deviation. Otherwise, repeat the titration and then average all the results using the Q-test to reject any outliers.
- 4. Compute the number of millimoles and weights of magnesium present in the sample solution and the tablet.

Results and Discussions

In the experiment, two commercial antacids A and B are quantitatively analyzed by twelve students and an instructor using the three protocols. Antacid A has a labeled 500 mg of an active ingredient hydrotalcite, $Mg_6Al_2(CO_3)(OH)_{16}\cdot4(H_2O)$, while antacid B claims to have 200 mg each of aluminum hydroxide dried gel, $Al(OH)_3$, and magnesium hydroxide, $Mg(OH)_2$.

For the protocols, stated as three parts in Experimental Procedure, the evaluation of their feasibility is divided into three aspects using two tailed t-test and one sided F-test, shown as below.

Analyst Comparison between Students and Instructor using Experimental Protocols

Data obtained from different analysts are evaluated by statistical analysis. Results are shown in Table 2.

Table 2. Results from Different Analysts between Students and Instructor

Note: This table was deleted by authors.

By the two tailed t-test and one sided F test, results show that there is no significant difference in average and precision of three protocols involving determinations of aluminum and magnesium contents in antacids by different analysts between students and instructor.

Protocol Comparison between Experiment and Calculation by instructor

Data gained from different protocols between experiment and calculation by instructor is evaluated by statistical analysis. Results are shown in Table 3.

Table 3. Results from Protocols between Experiment and Calculation by Instructor

Note: This table was deleted by authors.

By the two tailed *t*-test and one sided *F*-test, results indicate that experimental protocols in average and precision of the aluminum and magnesium contents in antacids determined by instructor are no significant difference in those that are calculated. These results confirm with the feasibility of three protocols.

Protocol Comparison between Experiment and Calculation by students

Data gained from different protocols between experiment and calculation by students is evaluated by statistical analysis. Results are shown in Table 4.

Table 4. Results from Protocols between Experiment and Calculation by Students

Note: This table was deleted by authors.

By the two tailed *t*-test and one sided *F*-test, results point out that the experimental protocols in avrage and precision are no significant difference in those that are calculated amounts of aluminum and magnesium in antacids measured by students. The feasibility of three protocols is also verified by these results.

Students' Feedbacks

In brief, students' feedbacks are described as below. I have learned that using various pH buffer solutions and appropriate indicators to determine metallic ions is important. I got the ideas of designing back-titration experiment and acquired the skill of complexometric titration. I hope I could apply all the ideas and practices of the complexometric titration and back titration for the analyses of everyday products. We could quantitatively analyze a number of consumer products containing diverse ions, if the masking concept was successfully applied. EDTA! I am eventually aware of how you grab metallic ions. I have never thought about the metallic ions can be determined by complexometric back titration. In the experiment, the idea for determining metallic ions in antacids is wonderful and the titration process is attractive due to a range of changes in color.

Conclusions

Results indicate three protocols are no significant difference in average and precision by different analysts between students and instructor. Besides, results show there are no significant difference in average and precision of different protocols between experiment and calculation performed by instructor, and the same as results carried out by students. These results verify the feasibility of three protocols.

In summary, students' comments are highly positive for the experimental design involving complexometric titration and back titration for the quantitative analysis of aluminum and magnesium ions in commercial antacids.

Acknowledgments

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Supplementary Material

Supplementary material, handouts for students including introduction, more readings, safety precautions and good practices, detailed procedures, and report sheet, is available.

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Supplementary Material: Handouts for Students

Complexometric Titration of Aluminum and Magnesium Ions in Commercial Antacids: An Undergraduate Chemical Experiment

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Introduction

Antacids

Antacids are used to relieve acid indigestion, upset and sour stomach, or heartburn. They work by neutralizing excess gastric acid in stomach. Antacids may be divided into two classes, (a) chemical antacids work by chemical neutralization of gastric acid, a notable example being sodium bicarbonate; and (b) adsorbed ones act by adsorption of the acid, including salts of aluminum and magnesium, and calcium carbonate. The former category show the most rapid onset of action, but may cause "acid rebound," a condition in which the gastric acid returns in greater concentration after the effect of drug has stopped. The latter category, whereas, by adsorption of the gastric acid, is less prone to the rebound effect.

Some antacids containing aluminum may be prescribed with a low-phosphate diet to treat hyperphosphatemia or prevent formations of phosphate urinary and kidney stones. With a larger dosage than normally required, antacids of magnesium hydroxide and oxide can produce a laxative effect. Antacids with aluminum and magnesium hydroxides, or aluminum hydroxide alone effectively prevent significant stress ulcer bleeding in postoperative patients or those with severe burns. Calcium antacids may be used as diet supplements to prevent osteoporosis, but side effects of constipation and renal stone formation may develop.

According to data from Medline Plus (see the More Readings below) related to antacid production in the U.S., there are 80 common brands in total. The common ingredients of aluminum antacids include alumina, aluminum hydroxide, and aluminum carbonate basic. Antacids of magnesium salts are generally of magnesia, trisilicate, carbonate, alginate, magaldrate, and hydroxide, while those with calcium salt are consist of calcium carbonate. Among these brands, the majority produced consist of aluminum and magnesium.

Determinative Methods

The samples analyzed for this experiment are from selected commercial antacid tablets, containing aluminum and magnesium ions as the active ingredients. Three analytical protocols based on complexometric titration with EDTA and its back-titration with zinc sulfate using suitable indicators in the appropriate buffer solutions were developed for the antacid analyses. The sample is initially dissolved in dilute hydrochloric acid with heating, which is diluted to 250.00 mL in a volumetric flask, and then 10.00 mL portions were taken for the titration.

EDTA, ethylenediaminetetracetic acid, is a compound that forms strong 1:1 complexes with most metal ions. It is the most widely used as a chelating agent in analytical chemistry. Virtually every element in the periodic table can be measured using EDTA either by direct or back titration. In general, complexometric back titration is used when metallic ions forms a stable complex with EDTA in a slow reaction or when an indicator is blocked by the complex formation with the metal

ion, the stability constant of which is greater than that of the metal-EDTA complex. Since the indicator cannot release the metal ions, no color change will be observable at the endpoint of a direct titration. Both of these conditions exist in the case of aluminum ion. Therefore, the metal ion is best determined by complexometric back titration combined with a heating condition to enhance its complexation with EDTA as described below.

Total Aluminum and Magnesium Protocol

In this method, the total aluminum and magnesium ions in antacids are determined by complexometric back titration. In the determination, the antacid sample solution containing aluminum and magnesium ions are kept at a pH 10.0 with a buffer solution followed by adding an excess known amount of EDTA. Heating the solution prior to the addition of indicator, Calmagite, is necessary to ensure that the EDTA is not blocked. The heating procedure also facilitates the complexation of the two metallic ions with the EDTA. Calmagite, in the presence of unchelated EDTA, remains pure blue in color due to its free form. The amount of unchelated EDTA can then be determined by back titration with standardized zinc sulfate solution. A color change to light wine red is observed at the endpoint. The formation of Zn-Calmagite complex in small quantity is responsible for this color change. Precise results will be achieved in avoiding the formation of persisting deep red color during the endpoint detection. Table 1 is a schematic summary illustrating the sequence and role of different reagents involved in the total aluminum and magnesium protocol.

Sequence	Metallic ions	Millimoles	Chelating agents
1	Al ³⁺ and Mg ²⁺ (in sample)		
2	Mg ²⁺ (masked by pH 5)		(EDTA inactive to Mg ²⁺)
3	Al ³⁺ (determined)		
4			EDTA (excess)
5			EDTA (unreacted)
6		(yellow)	Xylenol orange (free)
7	Zn ²⁺ (used for titration)		
8		(light red)	Xylenol orange (chelating with Zn ²⁺)
9	Al ³⁺ (calculated)		

Table 1. A Schematic Summary of Total Aluminum and Magnesium Protocol

Alone Aluminum Protocol

The separate aluminum amount in the antacid sample can also be quantitatively analyzed by complexometric back titration. In the analysis, the antacid sample is controlled at pH 5.0. An excess known amount of EDTA is then added to the solution. At this low pH, the Al-EDTA complex can form, whereas the Mg-EDTA formation is inhibited. Similar to the protocol described above, heating prior to the addition of indicator, xylenol orange, is imperative to prevent EDTA is blocked by indicator and to facilitate the complexation between the EDTA and aluminum ions. The free form of Xylenol orange is lemon yellow in color. The amount of unchelated EDTA is back titrated with standardized zinc sulfate solution. The endpoint of the titration is at which the yellow color changes to light red. This change in color is due to the formation of Zn-xylenol orange complex in small quantity. Precise results will be obtained in avoiding persisting deep red color in the endpoint detection. Table 2 is a schematic summary illustrating the sequence and role of different reagents involved in the aluminum protocol.

Table 2. A Schematic Summary of Alone Aluminum Protocol

Sequence	Metallic ions	Millimoles	Chelating agents
1	Al ³⁺ and Mg ²⁺ (in sample)		
2	Mg ²⁺ (masked by pH 5)		(EDTA inactive to Mg ²⁺)
3	Al ³⁺ (determined)		
4			EDTA (excess)
5			EDTA (unreacted)
6		(yellow)	Xylenol orange (free)
7	Zn ²⁺ (used for titration)		
8		(light red)	Xylenol orange (chelating with Zn ²⁺)
9	Al ³⁺ (calculated)		

Alone Magnesium Protocol

A direct complexometric titration with EDTA is used to determine the separate magnesium quantity in the sample. The sample solution with aluminum and magnesium is kept at a pH 10.0 buffer solution followed by adding a large amount of triethanolamine to mask the Al-EDTA complex formation. No heating is required. However, swirling the mixture helps to speed up the formation of Al-triethanolamine complex. The Calmagite indicator changes to wine red in solution containing magnesium ion, a consequence of magnesium-Calmagite complex formation. The endpoint of the titration is at which the color changes to pure blue. This color change results from the complete liberation of magnesium ion from the Mg-Calmagite complex. Table 3 is a schematic summary illustrating the sequence and role of different reagents involved in the magnesium protocol.

 Table 3. A Schematic Summary of Alone Magnesium Protocol

Sequence	Metallic ions	Millimoles	Chelating agents
1	Al ³⁺ and Mg ²⁺ (in sample)		
2	Al ³⁺ (masked)		Triethanolamine
3	Mg ²⁺ (determined)		
4		(wine red)	Calmagite (chelating with
		(while red)	Mg ²⁺)
5			EDTA (used for titration)
6		(pure blue)	Calmagite (free)
7	Mg ²⁺ (calculated)		

More Readings

Books

- ✓ Greco, T. G.; Rickard, L. H.; Weiss, G. S. *Experiments in General Chemistry: Principles* and Modern Applications, 8th Ed., Prentice Hall: Upper Saddle River, 2002, pp. 67-72.
- ✓ Hall, J. F. *Experimenal Chemistry*, 6th Ed., Houghton Mifflin: Boston, 2003, pp. 375-382.
- ✓ Douglas A. Skoog, D. A.; West, D. M.; Holler, F. J.; Crouch, S. R. *Analytical Chemistry: An Introduction*, 7th Ed., Harcourt College Publishers: Fort Worth, 2000, pp. 361-382.

Journals

- ✓ Novick, S. G., Complexometric Titration of Zinc: An Analytical Chemistry Laboratory Experiment, J. Chem. Educ. 1997 74 1463.
- ✓ Yappert, M. Cecilia; DuPre, Donald B., Complexometric Titrations: Competition of Complexing Agents in the Determination of Water Hardness with EDTA, J. Chem. Educ. 1997 74 1422.
- ✓ Daniel J. Williams, Benjamin E. Huck and Angus P. Wilkinson, First-Year Undergraduate Laboratory Experiments with Zeolites, *The Chemical Educator*, **2002**, Vol. 7, Issue 1, pp 33-36.

Web Sites

- ✓ Medline Plus, antacids (Oral), <u>http://www.nlm.nih.gov/medlineplus/druginfo/uspdi/202047.html</u> (accessed July 2004).
- ✓ The Merck Manual, antacids and indigestion remedies, <u>http://www.merck.com/mrkshared/mmanual_home2/sec02/ch018/ch018g.jsp</u> (accessed July 2004).
- ✓ The Pfizer Foundation Biochemistry: Discovery Lab, How do antacids work? <u>http://www.nyhallsci.org/biochem/content/educators/antacids-educators.pdf</u> (accessed July 2004).
- ✓ The complexometric titration, <u>http://chimge.unil.ch/En/complexes/lcpx24.htm</u> (accessed July 2004).
- ✓ SUSB 017 Complexometric titration of calcium in antacids, <u>http://www.ic.sunysb.edu/Class/che134/susb/susb017.pdf</u> (accessed July 2004).

Safety Precautions

- Protective eyewear approved by your institution must be worn at all times while you are in the laboratory.
- Triethanolamine is harmful if swallowed. Causes skin irritation and severe eye irritation.
- Solutions will spatter if heated too strongly. To avoid this, heat solutions and allow boiling gently. Placing a funnel to the flask mouth also prevent spatter.

Disposal

- Place any mixture containing zinc ion and triethanolamine in the collection bottles provided.
- Unused solutions containing EDTA, acetate-acetic acid buffer, bicarbonate-carbonate buffer, and the samples may be disposed by neutralization, dilution with water, and then flushing down the drain.

Good Practices

- It is important to use deionized rather than distilled water for solutions preparation and rinsing all glassware.
- To read the scale accurately, allow a waiting time of 30 seconds for the solution to adsorb onto the buret wall.
- In complexometric back titration, the order for the additions of solutions and indicators, heating and back-titration must be carried out as described in the procedure below.
- In any titration involving masking, the detection of endpoint must be followed as described

in the procedure below. Note: Titration in Part B should is slow but in Part C quick.

Reagent Preparation

- 1. **EDTA standard solution:** Dry about 4.0 g of reagent grade EDTA dihydrate, Na₂H₂EDTA·2H₂O (molecular weight = 372.25), in an oven at 80°C for one hour. Then accurately weigh out approximately 3.723 g (± 0.0001 g) of it. Quantitatively transfer the compound into a 1000 mL of volumetric flask, add a half full of deionized water, allow mixing to dissolve it, and then dilute to the mark. Stopper the flask and mix it well by inverting and shaking. If 3.7225 g of dried EDTA is used, the exact concentration of the solution should be 0.01000 M.
- 2. **Standardized Zinc sulfate solution:** Accurately weigh out approximately 2.875 g (to the nearest 0.0001 g) of reagent grade zinc sulfate heptahydrate, $ZnSO_4 \cdot 7H_2O$ (formula weight = 287.53). Transfer it quantitatively into a 1000-mL volumetric flask, add a half full of deionized water with mixing to dissolve it, and then dilute to the mark. Stopper the flask and mix it thoroughly by inverting and shaking. If 2.8753 g of the zinc salt is used, the exact concentration of the solution should be 0.01000 M.
- 3. Acetate-acetic acid buffer solution: Dissolve 54.6 g of sodium acetate trihydrate, CH₃COONa·3H₂O (molecular weight = 136.08), in a 1000-mL beaker containing about 500 mL of deionized water. Add 20 mL of 6 M. hydrochloric acid and then dilute it with deionized water to the 1000-mL mark with stirring. This buffer solution should have a pH of 5.0 ± 0.1 .
- 4. **Bicarbonate-carbonate buffer solution:** Mix 23.63 g of sodium bicarbonate, NaHCO₃ (formula weight = 84.01), with 72.88 g of sodium carbonate, Na₂CO₃ (formula weight 106.00) in a 1000-mL beaker. Dissolve the mixture with a half full of deionized water and then dilute the solution to the 1000-mL mark. This buffer should have a pH = 10.0 ± 0.1 .
- 5. **Xylenol orange indicator:** Dissolve 0.10 g of the acid or sodium salt form of xylenol orange in 50 mL of absolute ethanol. This prepared lemon-yellow-colored indicator is suitable for analyzing solutions at pH = 5.0. The solution prepared from the acid form is indefinitely stable, whereas that from the salt form may be used only for several months.
- 6. **Calmagite indicator:** Dissolve 0.25 g of Calmagite with 50 mL of absolute ethanol. The blue indicator is suitable for analyzing solutions at pH = 10.0. If the indicator appears purple in color, dropwise a pH 10 buffer solution until it changes back to blue in color. Calmagite gives a sharper endpoint for this experiment than Eriochrome Black T.

Sample Preparation

- 6. Obtain an antacid tablet from your instructor and record its brand name, active ingredient and the claimed quantity of each component.
- 7. Weigh the tablet precisely to the nearest 0.0001 g (denoted as w_{tab}). Grind it in a clean and dried mortar and pestle to make a powder as fine as possible. Remove most portion of the powder on a weighing paper placed on a tared balance and precisely weigh it (denoted as w_{pow}). Transfer the powder quantitatively to a clean 250 mL of Erlenmeyer flask containing about 100 mL of deionized water and about 6 mL of 6 M. hydrochloric acid.
- 8. Boil gently the mixture for about 20 minutes on a hot plate. Place a stem funnel to the flask mouth so that the vapor can condense quickly back to water, which helps washing down the powder sticks on the flask wall. If any powder still remains on the flask wall, wash it down with a small amount of deionized water and continue heating.

- 9. Remove the flask from the hot plate and allow it to cool to room temperature or rapidly in a water bath. Filter the mixture by gravity filtration into a 250 mL of volumetric flask. Rinse the flask and solid on the filter paper with about 10 mL of deionized water twice to make sure that all metallic ions are transferred into the volumetric flask.
- 10. Dilute the solution to the calibration mark with deionized water. Stopper the flask and mix the solution well by inverting and shaking it repeatedly. Label this solution "The antacid sample solution, 250.00 mL" (denoted as V_{pow}).

Procedure

Part A: Total Aluminum and Magnesium Protocol

- 6. Pipet a 10.00 mL aliquot of the antacid sample solution to a 125 mL of Erlenmeyer flask followed by about 10 mL of the bicarbonate-carbonate pH 10 buffer solution. Transfer quantitatively a 35.00 mL aliquot of the EDTA standard solution using a buret to the flask (denoted as V_{EDTA}).
- 7. Boil gently the mixture for 5 min. on a hot plate to speed up the formation of Al-EDTA complex. Add 5 drops of Calmagite indicator and mix it well. The solution should appear pure blue in color. If the EDTA is not enough to chelate completely all metallic ions, the solution should be wine red in color at this moment. Put an additional 5.00 or more mL aliquot of the EDTA solution to this wine red solution. Boil again until the color changes to pure blue. Continue this step if necessary.
- 8. Back-titrate the solution with standardized zinc sulfate solution until the color changes to purple at the endpoint (no wine red color should persist). Record the volume used (V_{Zn}) .
- 9. Repeat the titration twice. The data of V_{Zn} should agree within 1% of the relative average deviation. Otherwise, repeat the titration and then average all results using the Q-test to reject any outliers.
- 10. Calculate the combined total number of millimoles of aluminum and magnesium ions in the antacid sample solution and in the tablet.

Part B: Alone Aluminum Protocol

- 6. Pipet a 10.00 mL aliquot of the antacid sample solution to a 125 mL of Erlenmeyer flask. Add about 10 mL of the acetate-acetic acid pH 5 buffer solution to mask the formation of Mg-EDTA complex. Transfer quantitatively a 25.00 mL aliquot of the EDTA standard solution using a buret to the flask (denoted as V_{EDTA})
- 7. Boil it gently on a hot plate for 5 min. to speed up the formation of Al-EDTA complex. Add 5 drops of xylenol orange indicator and mix well. The solution should appear lemon yellow in color at this moment. If the EDTA is not enough to completely chelate all alumnum ions, the solution should be deep red in color. Put an additional 5.00 or more mL aliquot of the EDTA solution to this deep red solution. Boil again until the color changes to lemon yellow. Continue this step if necessary.
- 8. Back-titrate the solution with standardized zinc sulfate solution until the color changes to light red at the endpoint (no deep red color should remain). Continuously titrate the solution until a light red color persists for more than 3 minutes if the light red color shortly turns back to lemon yellow. Slow titration will give good results. Record the volume used (V_{Zn}).

Note: The turning back to lemon yellow color is the consequence of small quantity of the EDTA slowly shifted to an active polydentate species in accordance with Le Chatelier's Principle because the chelating ability of EDTA with zinc ions is reduced at a low pH.

9. Repeat the titration twice. The data of V_{Zn} should agree within 1% of the relative average

deviation. Otherwise, repeat the titration and then average all the results using the Q-test to reject any outliers.

10. Compute the number of millimoles and weights of aluminum present in the sample solution and the tablet.

Part C: Alone Magnesium Protocol

- 5. Pipet a 10.00 mL aliquot of the antacid sample solution to a 125 mL of Erlenmeyer flask followed by about 10 mL of the bicarbonate-carbonate pH 10 buffer solution. Add about 3 mL of triethanolamine and swirl the mixture for 2 min. to enhance the formation of Al-triethanolamine complex such that the formation of Al-EDTA complex is masked. Stand it for a while until the turbid solution becomes mostly clear for easy observation of endpoint. Add 5 drops of Calmagite indicator and mix well. The solution should appear wine red in color.
- 6. Direct-titrate the solution with the EDTA standard solution until the color changes to pure blue at the endpoint. Record the volume used (V_{EDTA}). A trial titration first is recommended to get an ideal result. Add most portion of the EDTA solution at a time and then carefully titrate it dropwise near the endpoint.

Note: Quick titration will give a good result. If the titration is slow, the aluminum ion will be released from the Al-triethanolamine complex and produce the Al-Calmagite complex of wine red in color. In such case, continuing titration will give a positive error.

- 7. Repeat the titration twice. The data of V_{Zn} should agree within 1% of the relative average deviation. Otherwise, repeat the titration and then average all the results using the Q-test to reject any outliers.
- 8. Compute the number of millimoles and weights of magnesium present in the sample solution and the tablet.

Report A (This sheet is suitable for antacids containing Al and Mg in identical form)

 Date:
 2004/06/17
 Student name:
 Shu-Ling Zoa

 Course:
 General Chemistry Laboratory
 Team members:
 Team members:

 Instructor:
 Shui-Ping Yang
 Student name:
 Student name:

Data and Results

Brand name: <u>N-brand Tablets (Used as antacid A)</u> Manufacturer: <u>Taiwan Y-Y-Y Pharmacy Company</u>

Weight of Aluminum content: <u>500</u> mg / tablet (ingredient labeled as <u>*Hydrotalcite*</u> form). Weight of Magnesium content: <u>500</u> mg / tablet (ingredient labeled as <u>*Hydrotalcite*</u> form).

Weight of the antacid tablet (w_{tab}) : <u>0.9388</u> g Weight of the powder sample (w_{pow}) : <u>0.9380</u> g Volume of the sample solution prepared (V_{pow}) : <u>250.00</u> mL

Useful Information

Hydrotalcite: molecular formula: Mg₆Al₂(CO₃)(OH)₁₆·4(H₂O); molecular weight: 603.98

Part A: Hydrotalcite Determination by Total Aluminum and Magnesium Protocol

	Trial 1	Trial 2	Trial 3
Sample determined Volume (V _{used}), mL	10.00	10.00	10.00
EDTA used Volume (V_{EDTA}), mL Molarity (M_{EDTA}), M Millimole (mm_{EDTA} ' = M_{EDTA} * V_{EDTA}), mmol Average millimole (mm_{EDTA}), mmol	<u>40.00</u> <u>0.01000</u> <u>0.4000</u>	$\begin{array}{r} 40.00\\ \hline 0.01000\\ \hline 0.4000\\ \hline 0.4000 \end{array}$	<u>40.00</u> <u>0.01000</u> <u>0.4000</u>
Standardized zince sulfate solution titrated Initial volume (V_{iZn}) , M Final volume (V_{fZn}) , mL Volume used $(V_{Zn} = V_{fZn} - V_{iZn})$, mL Molarity (M_{Zn}) , M Millimole $(mm_{Zn}, M_{Zn}, M_{Zn})$, mmol Average of millimole (mm_{Zn}) , mmol	$\begin{array}{r} 0.00 \\ 13.56 \\ 13.56 \\ 0.01000 \\ 0.1356 \end{array}$	$\begin{array}{r} 0.00\\ \hline 13.53\\ \hline 13.53\\ 0.01000\\ \hline 0.1353\\ \hline 0.1355 \end{array}$	$\begin{array}{r} 0.00 \\ \hline 13.55 \\ \hline 13.55 \\ 0.01000 \\ \hline 0.1355 \end{array}$
10.00 mL of the sample solution Total millimole $(mm_{Al+Mg(10)} = mm_{EDTA} - mm_{Zn})$), mmol	0.2645	
250.00 mL of the sample solution Total millimole $[mm_{Al+Mg(250)} = mm_{Al+Mg(10)} *(2)]$	250/10)], mmol	6.613	
Al and Mg in a tablet Total millimole $[mm_{Al+Mg} = mm_{Al+Mg(250)} *(w_{tab})$,/ w _{pow})], mmol	6.619	

Hydrotalcite weight $[w_{hydrotalcite} = mm_{Al+Mg} * (60)]$	3.98 / 8)], mg	499.7	
Compare with value claimed ($w_{claimed}$) Weight difference ($\Delta w_{hydrotalcite} = w_{hydrotalcite} - w_{cla}$ Weight difference % [$\Delta w\% = (\Delta w_{hydrotalcite} / w_{cla})$	_{aimed}), mg _{aimed})*100], %	-0.3 -0.06	
Precision (by zinc volume used) Deviation of each trial (D), mmol Average deviation (AD), mmol Relative average deviation (RMD), %	0.01	0.01 0.01 0.08	0.01

Part B: Hydrotalcite Determination by Alone Aluminum Protocol

	Trial 1	Trial 2	Trial 3
Sample determined Volume (V _{used}), mL	10.00	10.00	10.00
EDTA used in sample Volume (V_{EDTA}), mL Molarity (M_{EDTA}), M Millimole ($mm_{EDTA'} = M_{EDTA} * V_{EDTA}$), mmol Average of millimole (mm_{EDTA}), mmol	25.00 0.01000 0.2500	25.00 0.01000 0.2500 0.2500	25.00 0.01000 0.2500
Standardized zince sulfate solution titrated Initial volume (V_{iZn}) , M Final volume (V_{fZn}) , mL Volume used $(V_{Zn} = V_{fZn} - V_{iZn})$, mL Molarity (M_{Zn}) , M Millimole $(mm_{Zn}) = M_{Zn} * V_{Zn}$, mmol Average of millimole (mm_{Zn}) , mmol	$\begin{array}{r} 0.00\\ 18.41\\ 18.41\\ 0.01000\\ 0.1841 \end{array}$	0.00 18.39 18.39 0.01000 0.1839 0.1840	$\begin{array}{r} 0.00 \\ 18.41 \\ 18.41 \\ 0.01000 \\ 0.1841 \end{array}$
10.00 mL of the sample solution Al millimole $(mm_{Al(10)} = mm_{EDTA} - mm_{Zn})$, mmc	bl	0.0660	
250.00 mL of the sample solution Al millimole $[mm_{Al(250)} = mm_{Al(10)} * (250/10)]$, n	mmol	1.649	
Al and Mg in a tablet Al millimole $[mm_{Al} = mm_{Al(250)} *(w_{tab} / w_{pow})],$ Hydrotalcite weight $[w_{hydrotalcite} = mm_{Al} * (603)]$	mmol 3.98 / 2)], mg	<u> </u>	
Compare with value claimed ($w_{claimed}$) Weight difference ($\Delta w_{hydrotalcite} = w_{hydrotalcite} - w_{hydrotalcite}$) Weight difference % [$\Delta w \% = (\Delta w_{hydrotalcite} / w_{hydrotalcite})$]	v _{claimed}), mg v _{claimed})*100], %	<u>-1.5</u> -0.30	
Precision (by zinc volume used) Deviation of each trial (D), mmol Average deviation (AD), mmol Relative average deviation (RMD), %	0.01	0.01 0.01 0.05	0.01

Part C: Hydrotalcite Determination by Alone Magnesium Protocol

	Trial 1	Trial 2	Trial 3
Sample determined Volume (V _{used}), mL	10.00	10.00	10.00
EDTA used in sample Initial volume (V_{iZn}) , M Final volume $(V_{fZn} = V_{fZn} - V_{iZn})$, mL Volume used $(V_{EDTA} = V_{fEDTA} - V_{iEDTA})$, mL Molarity (M_{EDTA}) , M Millimole $(mm_{EDTA}) = M_{EDTA} * V_{EDTA})$, mmol Average millimole (mm_{EDTA}) , mmol	$\begin{array}{r} 0.00 \\ \hline 19.85 \\ \hline 19.85 \\ 0.01000 \\ \hline 0.1985 \end{array}$	0.00 19.81 19.81 0.01000 0.1981 0.1983	$\begin{array}{r} 0.00 \\ \hline 19.83 \\ \hline 19.83 \\ 0.01000 \\ \hline 0.1983 \end{array}$
10.00 mL of the sample solution Mg millimole ($mm_{Mg(10)} = mm_{EDTA}$), mmol		0.1983	
250.00 mL of the sample solution Mg millimole $[mm_{Mg(250)} = mm_{Mg(10)} * (250/10)]$, mmol	4.958	
Mg in a tablet Mg millimole $[mm_{Mg} = mm_{Mg(250)} *(w_{tab} / w_{pow})]$ Hydrotalcite weight $[w_{hydrotalcite} = mm_{Mg} * (603)]$	l, mmol 8.98 / 6)], mg	<u>4.962</u> <u>499.5</u>	
Compare with value claimed $(w_{claimed})$ Weight difference $(\Delta w_{hydrotalcite} = w_{hydrotalcite} - w$ Weight difference % $[\Delta w\% = (\Delta w_{hydrotalcite} / w)$	c _{laimed}), mg c _{laimed})*100], %	<u>-0.5</u> 0.1	
Precision (by EDTA volume used) Deviation of each trial (D), mmol Average deviation (AD), mmol Relative average deviation (RMD), %	0.02	0.02 0.01 0.07	0.00

Report B (This sheet is suitable for antacids containing Al and Mg in different form)

Date:	2004/06/17	Student name: <u>Shu-Ling Zoa</u>	
Course:	General Chemistry Laboratory	Team members:	
Instruct	or: Shui-Ping Yang		

Data and Results

Brand name: <u>Gp-brand Tablets (Used as antacid B)</u> Manufacturer: <u>Taiwan P-F Pharmaceuticals Limited</u> Aluminum weight: <u>200</u> mg / tablet (ingredient labeled as <u>Al(OH)₃</u> form). Magnesium weight: <u>200</u> mg / tablet (ingredient labeled as <u>Mg(OH)₂</u> form).

Weight of the antacid tablet (w_{tab}): <u>1.2072</u> g Weight of the powder sample (w_{pow}): <u>1.2020</u> g Volume of the sample prepared (V_{pow}): <u>250.00</u> mL

Useful Information

Alumina: molecular formula: Al₂O₃; molecular weight: 101.96 Aluminum hydroxide: molecular formula: Al(OH)₃; molecular weight: 77.89 Aluminum carbonate: molecular formula: Al₂(CO₃)₃; molecular weight: 234.00 Magnesia: molecular formula: MgO; molecular weight: 40.31 Magnesium hydroxide: molecular formula: Mg(OH)₂; molecular weight: 58.32 Magnesium carbonate: molecular formula: MgCO₃; molecular weight: 84.31

Trial 1 Trial 2 Trial 3 Sample determined Volume (Vused), mL 10.00 10.00 10.00 EDTA used in sample Volume (V_{EDTA}), mL 35.00 35.00 35.00 0.01000 0.01000 0.01000 Molarity (M_{EDTA}), M Millimole (mm_{EDTA} ' = M_{EDTA} * V_{EDTA}), mmol 0.3500 0.3500 0.3500 Average millimole (mm_{EDTA}), mmol 0.3500 Standardized zince sulfate solution titrated Initial volume (V_{iZn}), M 0.00 0.00 0.00 Final volume (V_{fZn}), mL 12.10 12.08 12.12 Volume used ($V_{Zn} = V_{fZn} - V_{iZn}$), mL 12.10 12.08 12.12 0.01000 0.01000 0.01000 Molarity (M_{Zn}), M Millimole (mm_{Zn} ^{*} = M_{Zn} ^{*} V_{Zn}), mmol 0.1210 0.1208 0.1212 Average of millimole (mm_{Zn}), mmol 0.1210 10.00 mL of the sample solution Total millimole $(mm_{Al+Mg(10)} = mm_{EDTA} - mm_{Zn})$, mmol 0.2290 250.00 mL of the sample solution Total millimole $[mm_{Al+Mg(250)} = mm_{Al+Mg(10)} * (250/10)]$, mmol 5.725

Part A: Total Aluminum and Magnesium Content

Al and Mg in a tablet

Total millimole $[mm_{Al+Mg} = mm_{Al+Mg(250)} *(w_{tab} / w_{tab})]$	w _{pow})], mmol	5.750	
Compare with value claimed Difference ($\Delta mm = mm_{Al+Mg} - mm_{claimed}$), mmo Difference % [$\Delta mm\% = (\Delta mm / mm_{claimed})$ *10	ol 00], %	-0.244 -4.07	
Precision (by zinc volume used) Deviation of each trial (D), mmol Average deviation (AD), mmol Relative average deviation (RMD), %	0.00		0.00

Part B: Determination of Aluminum Content

	Trial 1	Trial 2	Trial 3
Sample determined Volume (V _{used}), mL			10.00
EDTA used in sample Volume (V_{EDTA}), mL Molarity (M_{EDTA}), M Millimole (mm_{EDTA} ' = M_{EDTA} * V_{EDTA}), mmol Average of millimole (mm_{EDTA}), mmol	25.00 0.01000 0.2500	25.00 0.01000 0.2500 0.2500	<u>25.00</u> <u>0.01000</u> <u>0.2500</u>
Standardized zince sulfate solution titrated Initial volume (V_{iZn}) , M Final volume (V_{fZn}) , mL Volume used $(V_{Zn} = V_{fZn} - V_{iZn})$, mL Molarity (M_{Zn}) , M Millimole $(mm_{Zn}, = M_{Zn}, V_{Zn})$, mmol Average of millimole (mm_{Zn}) , mmol	$\begin{array}{r} 0.00 \\ \hline 16.52 \\ \hline 16.52 \\ 0.01000 \\ \hline 0.1652 \end{array}$	$\begin{array}{r} 0.00\\ \hline 16.50\\ \hline 0.01000\\ \hline 0.1650\\ \hline 0.1652\\ \end{array}$	$\begin{array}{r} 0.00 \\ \hline 16.54 \\ \hline 16.54 \\ \hline 0.01000 \\ \hline 0.1654 \end{array}$
10.00 mL of the sample solution Al millimole $(mm_{Al(10)} = mm_{EDTA} - mm_{Zn})$, mmol	0.08480	
250.00 mL of the sample solution Al millimole $[mm_{Al(250)} = mm_{Al(10)} * (250/10)]$, n	mmol	2.120	
Alone aluminum in a tablet Al millimole $[mm_{Al} = mm_{Al(250)} *(w_{tab} / w_{pow})],$ Al ₂ O ₃ weight $[w_{Al2O3} = mm_{Al} * 101.96],$ r Al(OH) ₃ weight $[w_{Al(OH)3} = mm_{Al} *77.89]$ Al ₂ (CO ₃) ₃ weight $[w_{Al2(CO3)3} = mm_{Al} *234.0]$	mmol ng], mg 0], mg	<u>2.129</u> <u>166.1</u>	
Compare with value claimed ($w_{claimed}$) Al ₂ O ₃ weight difference [$\Delta w_{Al2O3} = w_{Al2O3} - w_{claimed}$], mg Al(OH) ₃ weight difference [$\Delta w_{Al(OH)3} = w_{Al(OH)3} - w_{claimed}$], mg Al ₂ (CO ₃) ₃ weight difference [$\Delta w_{Al2(CO3)3} = w_{Al2(CO3)3} - w_{claimed}$], mg Al ₂ O ₃ weight difference % [($\Delta w_{Al2O3} / w_{claimed}$)*100], mg Al(OH) ₃ weight difference % [($\Delta w_{Al2O3} / w_{claimed}$)*100], mg Al ₂ (CO ₃) ₃ weight difference % [($\Delta w_{Al2(CO3)3} / w_{claimed}$)*100], mg Al ₂ (CO ₃) ₃ weight difference % [($\Delta w_{Al2(CO3)3} / w_{claimed}$)*100], mg		-33.9	

Precision (by zinc volume used)

Deviation of each trial (D), mmol	0.00	0.02	0.02
Average deviation (AD), mmol		0.01	
Relative average deviation (RMD), %		0.01	

Part C: Determination of Magnesium Content

	Trial 1	Trial 2	Trial 3
Sample determined Volume (V _{used}), mL			10.00
EDTA used in sample Initial volume (V_{iEDTA}), mL Final volume (V_{fEDTA}), mL Volume used ($V_{EDTA} = V_{fEDTA} - V_{iEDTA}$), mL Molarity (M_{EDTA}), M Millimole ($mm_{EDTA}^{-} = M_{EDTA} * V_{EDTA}$), mmol Average millimole (mm_{EDTA}), mmol	$\begin{array}{r} 0.00 \\ 14.28 \\ 14.28 \\ 0.01000 \\ 0.1428 \end{array}$	$\begin{array}{r} 0.00\\ 14.19\\ 14.19\\ 0.01000\\ 0.1419\\ 0.1423 \end{array}$	$\begin{array}{r} 0.00 \\ 14.22 \\ 14.22 \\ 0.01000 \\ 0.1422 \end{array}$
10.00 mL of the sample solution Mg millimole ($mm_{Mg(10)} = mm_{EDTA}$), mme	ol	0.1423	
250.00 mL of the sample solution Al millimole $[mm_{Mg(250)} = mm_{Al(10)} * (250/10)],$	mmol	3.558	
Alone aluminum in a tablet Mg millimole $[mm_{Mg} = mm_{Mg(250)} *(w_{tab} / w_{pow})]$, mmol MgO weight $[w_{MgO} = mm_{Mg} * 40.31]$, mg Mg(OH) ₂ weight $[w_{Mg(OH)2} = mm_{Mg} * 58.32]$, mg MgCO ₃ weight $[w_{MgCO3} = mm_{Mg} * 84.31]$, mg		<u>3.573</u> 208.4	
$\begin{array}{l} Compare \ with \ value \ claimed \ (w_{claimed}) \\ MgO \ weight \ difference \ [\Delta w_{MgO} = w_{MgO} - w_{claimed}], \ mg \\ Mg(OH)_2 \ weight \ difference \ [\Delta w_{Mg(OH)2} = w_{Mg(OH)2} - w_{claimed}], \ mg \\ MgCO_3 \ weight \ difference \ [\Delta w_{MgCO3} = w_{MgCO3} - w_{claimed}], \ mg \\ MgO \ weight \ difference \ \% \ [(\Delta w_{MgO} / w_{claimed})^*100], \ mg \\ Mg(OH)_2 \ weight \ difference \ \% \ [(\Delta w_{Mg(OH)2} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ [(\Delta w_{MgCO3} / w_{claimed})^*100], \ mg \\ MgCO_3 \ weight \ difference \ \% \ (MgCO_3 + MgCO_3 + MgCO$		+8.4	
Precision (by EDTA volume used) Deviation of each trial (D), mmol Average deviation (AD), mmol Relative average deviation (RMD), %	0.05	$\begin{array}{r} 0.04 \\ \hline 0.03 \\ \hline 0.02 \end{array}$	