

# Complexometric Titration of Aluminum and Magnesium Ions in Commercial Antacids

An Experiment for General and Analytical Chemistry Laboratories



Shui-Ping Yang\* and Rwei-Ying Tsai

Department of Chemistry, National Changhua University of Education, Changhua 50058, Taiwan; \*yangsp@cc.ncue.edu.tw

The chemicals required for chemistry laboratories are obtained primarily from chemical companies. Few experiments use chemicals from consumer products. Student interest in chemistry usually suffers from the lack of relevance to their daily lives. Ultimately this leads to insufficient student chemical problem-solving ability. To address this issue a novel experiment for determining the total and individual metallic ions contents in commercial antacids using complexometric direct and back titrations is presented.

Some household chemical products have been compiled in general chemistry laboratory textbooks (such as 1, 2). Consumer products have been used as assays in the laboratory. For example, white vinegar is used for acid–base titration (3–5); bleach and vitamin tablets are applied in oxidation–reduction titration (6–8); aspirin tablets and cola drinks are employed for spectrophotometric determination (3, 4). Recently, few household products suitable for quantitative analysis have been published in chemical education literature. 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 using acid–base back titration is a popular experiment. This process is intended to teach students about acid–base chemistry and back titration. It is usually present in general chemistry laboratory textbooks (such as 3, 7, 8, 13–15) and frequently found on the Internet (e.g., 16–18). Experiments related to antacids such as rating antacids (19) and brand identification (20) have been also published.

Complexometric direct titration is usually used in general and analytical chemistry laboratories. A well-known experiment using the titration is the determination of the total hardness of water found in several textbooks (3, 5, 21–23) and Web sites (24–26). The titration has also been applied to consumer products analyses including water (27, 28), cold lozenges (29), and shower cleaners (30) in chemical education literature. Indeed, it is still used for chemical research (such as 31, 32). However, complexometric back titration has not yet been found in undergraduate chemical laboratory textbooks and chemical education literature.

This experiment, developed using three different protocols, is suitable for general chemistry and introductory quantitative analysis laboratory courses. Students can learn about various concepts and skills for sample preparation, buffer solutions preparation, complexometric direct and back titrations, metallic indicators, complex formation, masking, and blocking.

## Overview of the Protocols

According to data from Medline Plus, the majority of 80 common brands of antacids produced in the United States contain aluminum and magnesium ions, with few containing calcium ion (see the Supplemental Material<sup>W</sup>). Based on these

data, this experiment is designed for the determinations of aluminum and magnesium ions but not calcium ions in antacids.

To determine the metallic ion contents in commercial antacids, this experiments are designed using three independent protocols: total aluminum and magnesium ions, aluminum ion alone, and magnesium ion alone, respectively. Owing to the design ideas, the result obtained from one protocol can be confirmed with that from the other two protocols using dependable calculations. The aluminum ion is best determined using complexometric back titration under heating to form the Al–EDTA complex beforehand. This is because aluminum ion can form a stable complex with EDTA in a slow reaction, and an indicator used in the titration is quickly blocked by the aluminum ion.

### Protocol A

To determine the total aluminum and magnesium contents in antacids, the sample solution is kept in a pH 10.0 buffer solution followed by adding an excess known quantity of EDTA. Heating prior to adding the indicator is necessary so that the indicator is not blocked by the aluminum ion. In this buffer solution, the two metallic ions can stably form complexes with the excess EDTA. The quantity of unchelated EDTA can then be determined using complexometric back titration with a standardized zinc solution.

### Protocol B

To determine the aluminum quantity alone, the sample solution is controlled in a pH 5.0 buffer solution. An excess known quantity of EDTA is then added. At this low pH, the Al–EDTA complex can form, while the formation of Mg–EDTA complex is completely inhibited. Then heating the solution, adding the indicator, and back titrating the unchelated EDTA is similar to the Protocol A.

### Protocol C

To determine the magnesium content alone, the sample solution is kept in a pH 10.0 buffer solution followed by adding a large quantity of triethanolamine to entirely mask the Al–EDTA complexation. No heating is required for complexometric direct titration.

To help students understand the complicated concepts involved in complexometric direct and back titrations, three tables that illustrate the sequence and schematic quantities of metallic ions during the three protocols are available in the Supplemental Material.<sup>W</sup> A summary of the aluminum alone protocol is shown in Table 1.

## Experimental

The complete experimental procedure, reagent preparation, sample preparation, waste disposal, and good practices are stated in the Supplemental Material.<sup>W</sup>

## Results and Discussion

In this experiment, two antacids A and B are quantitatively analyzed by twelve students and an instructor using the three protocols stated above. Antacid A is labeled with 500 mg of an active ingredient hydrotalcite,  $\text{Mg}_6\text{Al}_2(\text{CO}_3)(\text{OH})_{16}\cdot 4(\text{H}_2\text{O})$ . Antacid B claims to have 200 mg of aluminum hydroxide dried gel,  $\text{Al}(\text{OH})_3$ , and 200 mg of magnesium hydroxide,  $\text{Mg}(\text{OH})_2$ . The hydrotalcite weight in antacid A can be determined using each of the three protocols.

To evaluate the feasibility of the proposed experiment, four comparisons—complexometric titrations versus declarations of manufacturer, complexometric titrations versus spectrophotometry, different analysts for the three protocols, and different tactics by the instructor—using the two-tailed  $t$  test and one-sided  $F$  test were conducted as shown below.

### Comparison of Titrations with Declarations

Data obtained from both complexometric titrations (CT) by the instructor and the declared values (DV) in the antacids were compared using statistical analysis. The results are shown in Table 2. The two-tailed  $t$  test indicated that there were no differences between the average weights using the complexometric titration determinations by the instructor

and the declared values in antacid A. Two significant differences related to average appeared in antacid B are  $-33.6$  mg of aluminum hydroxide dried gel and  $13.3$  mg of magnesium hydroxide ( $p < 0.001$ ).

To elucidate the disagreement in antacid B, the weights of the active ingredients obtained from both complexometric titrations and spectrophotometry and the data were compared.

### Comparison of Titrations with Spectrophotometry

Data obtained from both complexometric titrations and UV-visible spectrophotometry (VS) by the instructor were evaluated using statistical analysis. The results are displayed in Table 3. The two-tailed  $t$  test showed that there were no differences in the average weights of the active ingredients in the two antacids determined using different analytical methods (complexometric titrations versus spectrophotometry) by the instructor, except for a  $0.6$  mg small average difference for magnesium hydroxide ( $p < 0.05$ ). These results confirm that the declared active ingredient values in antacid B are not accurate.

### Different Analysts for the Protocols

Data obtained from different analysts (students and instructor) performing the three protocols were evaluated us-

**Table 1. A Summary of the Aluminum Alone Protocol**

Sequence	Metallic Ions	Schematic Quantities	Chelating Agents
1	$\text{Al}^{3+}$ (◆) and $\text{Mg}^{2+}$ (■) (in sample)	◆◆◆■ ■■■■■■	—
2	$\text{Mg}^{2+}$ (■) (masked by pH 5)	■■■■■■■	(EDTA inactive to $\text{Mg}^{2+}$ )
3	$\text{Al}^{3+}$ (◆)	◆◆◆	—
4	—	▲▲▲▲▲▲▲▲	EDTA (▲) (excess)
5	—	▲▲▲▲▲	EDTA (▲) (unchelated)
6	—	◇	Xylenol orange (◇) (free)
7	$\text{Zn}^{2+}$ (●) (used for titration)	●●●●●	—
8	—	○	Xylenol orange (○) (chelating with $\text{Zn}^{2+}$ )
9	$\text{Al}^{3+}$ (◆) (calculated)	◆◆◆	—

**Table 2. Comparison of Complexometric Titrations (CT) with Declared Values (DV)**

Antacids	Ingredients	CT Avg/ mg <sup>a</sup>	DV Avg/ mg <sup>a</sup>	Avg Dif <sup>b</sup> / mg	SD Dif <sup>c</sup> / mg	$t$ Value	$p$ Value
A	Hydrotalcite (for Al protocol)	501.2	500.0	1.2	1.2	2.390	0.139
A	Hydrotalcite (for Mg protocol)	500.4	500.0	0.4	0.5	1.512	0.270
B	$\text{Al}(\text{OH})_3$ dried gel	166.4	200.0	$-33.6$	0.3	5000000	0.000 <sup>d</sup>
B	$\text{Mg}(\text{OH})_2$	213.3	200.0	13.3	0.2	5000000	0.000 <sup>d</sup>

<sup>a</sup>The values are an average of three trials. <sup>b</sup>Difference in averages between the third and fourth columns. <sup>c</sup>Standard deviation of the differences. <sup>d</sup>This value is significant at  $p < 0.001$ .

**Table 3. Comparison of Complexometric Titrations (CT) with UV-Vis Spectrophotometries (VS)**

Antacids	Ingredients	CT Avg/ mg <sup>a</sup>	VS Avg/ mg <sup>a,b</sup>	Avg Dif <sup>c</sup> / mg	SD Dif <sup>d</sup> / mg	$t$ Value	$p$ Value
A	Hydrotalcite (for Al protocol)	501.2	499.7	1.5	$-0.4$	1.297	0.251
A	Hydrotalcite (for Mg protocol)	500.4	500.0	0.4	$-0.3$	0.627	0.558
B	$\text{Al}(\text{OH})_3$ dried gel	166.4	166.2	0.2	0.1	0.904	0.408
B	$\text{Mg}(\text{OH})_2$	213.3	212.6	0.6	0.1	3.062	0.028 <sup>e</sup>

<sup>a</sup>Each entry of titrations and spectrophotometry are the average of three values. <sup>b</sup>The SHIMADZD UV-120 Spectrophotometer is used for the determinations. <sup>c</sup>Difference in averages between the third and fourth columns. <sup>d</sup>Standard deviation of the differences. <sup>e</sup>This value is significant at  $p < 0.05$ .

**Table 4. Different Analysts Performing the Three Protocols**

Antacids	Protocols	Students Avg/ mg <sup>a</sup>	Instructor Avg/ mg <sup>a</sup>	Avg Dif <sup>b</sup> / mg	SD Dif <sup>c</sup> / mg	<i>t</i> Value	<i>p</i> Value	<i>F</i> Value	<i>p</i> Value
A	Total Al and Mg	500.1	501.1	-1.0	1.6	0.842	0.405	12.433	0.077
A	Aluminum Alone	499.4	501.2	-1.8	1.2	1.288	0.206	1.831	0.416
A	Magnesium Alone	500.8	500.4	0.4	1.5	0.333	0.741	7.073	0.131
B	Total Al and Mg	5.805 <sup>d</sup>	5.799 <sup>d</sup>	0.006 <sup>d</sup>	0.044 <sup>d</sup>	0.227	0.822	77.537	0.013 <sup>e</sup>
B	Aluminum Alone	169.1	166.4	2.7	2.2	1.850	0.072	35.212	0.028 <sup>e</sup>
B	Magnesium Alone	210.7	213.3	-2.6	2.6	1.557	0.128	78.479	0.013 <sup>e</sup>

<sup>a</sup>Thirty-six and three values separately obtained by students and instructor, respectively, are averaged. <sup>b</sup>Difference in averages between the third and fourth columns. <sup>c</sup>Standard deviation of the differences. <sup>d</sup>These values are presented in millimoles. <sup>e</sup>This value is significant at  $p < 0.05$ .

**Table 5. Different Tactics by Instructor**

Antacids	Tactics	Former Avg/ mg <sup>a</sup>	Latter Avg/ mg <sup>a</sup>	Avg Dif <sup>b</sup> / mg	SD Dif <sup>c</sup> / mg	<i>t</i> Value	<i>p</i> Value	<i>F</i> Value	<i>p</i> Value
A	Total vs Al	501.1	501.2	-0.1	-0.8	0.190	0.867	3.808	0.208
A	Total vs Mg	501.1	500.4	0.7	-0.1	2.468	0.132	0.389	0.720
A	Al vs Mg	501.2	500.4	0.8	0.6	1.329	0.315	1.923	0.342
B	Total vs Al Plus Mg	5.799 <sup>d</sup>	5.790 <sup>d</sup>	0.009 <sup>d</sup>	-0.001 <sup>d</sup>	3.725	0.065	0.357	0.737
B	Al vs Total Minus Mg	166.4	167.1	-0.7	-0.3	2.576	0.123	1.326	0.430
B	Mg vs Total Minus Al	213.3	213.8	-0.5	-0.2	4.065	0.056	3.000	0.250

<sup>a</sup>Three values of each tactic by instructor are averaged. The average obtained from former tactics and latter tactics separately, for example, Total vs Al, former is Total (Total Al and Mg protocol) and latter is Al (Alone Al protocol). <sup>b</sup>Difference in averages between the third and fourth columns. <sup>c</sup>Standard deviation of the differences. <sup>d</sup>These values are presented in millimoles.

ing statistical analysis. The results are shown in Table 4. The two-tailed *t* test showed that no difference was presented in the average total aluminum and magnesium ion contents and individual ion content in the two antacids determined by students and the instructor using the three protocols.

By applying the one-sided *F* test, the results pointed out that the precision for metallic ion contents in antacid A determined using the three protocols by the students was not different from that by the instructor. However, the data for antacid B obtained by the instructor were significantly more precise than those obtained by the students ( $p < 0.05$ ).

#### Different Tactics by Instructor

Data gained from the hydrotalcite determination using each protocol for antacid A and the dependent calculations of the active ingredients for antacid B were evaluated using statistical analysis. The results are shown in Table 5.

Using the two-tailed *t* test and one-sided *F* test, the results indicated that there were no differences in the average and precision for the total aluminum and magnesium ion determinations in the two antacids using different protocols by the instructor.

#### Student Feedback

The feedback received from the students was positive and reflects the students' enthusiasm for the experiment. Some typical comments are:

- I have learned that determining the metallic ion content using various pH buffer solutions and appropriate indica-

tors is important.

- I learned how to design a back-titration experiment and perform complexometric titration.
- I hope that I could apply all of the ideas and practices that I learned from complexometric titration and back titration to analysis everyday products.
- We could quantitatively analyze a number of consumer products containing diverse ions if the masking concept was successfully applied.
- EDTA! I am aware of how metallic ions can be determined. I never thought about how metallic ions can be determined by complexometric back titration.
- In this 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.

#### Hazards

Disodium dihydrogen ethylenediaminetetraacetate dihydrate, zinc sulfate heptahydrate, sodium acetate trihydrate, and sodium carbonate may cause eye, skin, and respiratory tract irritation. Hydrochloric acid is corrosive and damageable to the skin, eyes, and respiratory. Triethanolamine causes skin irritation and severe eye irritation. Ethanol is a highly flammable liquid. There must be no open flames in the laboratory. Adequate ventilation must be used. Xylenol orange and calmagite may cause irritation to the eyes and skin. Magnesium hydroxide, aluminum hydroxide, and hydrotalcite are

the active ingredients in commercial antacids, thus there is no hazard in normal handling. Protective gloves and goggles must be worn at all times. [Editor's Note: CLIPs are available in *J. Chem. Educ.* for sodium acetate (2002, 79, 1314); sodium carbonate (2002, 79, 1315); HCl(aq) (2001, 78, 873).]

## Conclusions

The results showed that the average and precision for the determinations of active ingredients in the two antacids by complexometric titrations using the three protocols were not different from those obtained using spectrophotometry. There were no differences found in the total aluminum and magnesium ion contents and individual ion content in the two antacids using the three protocols determined by both students and instructor. Differences in the precision appeared in one of the two antacid experiments. Moreover, no difference was found in the averages and precisions for these determinations using different tactics by the instructor.

The students gave positive comments on complexometric direct and back titrations. Because of the good results from the students and instructor and the positive comments made by students, the proposed experimental design is highly feasible for use in general chemistry and introductory analytical chemistry laboratories.

## Acknowledgments

The authors thank the National Science Council of Taiwan for financially supporting this research by grant 92-2511-S-018-010 and Hon Man Lee for helpful suggestions.

## <sup>W</sup>Supplemental Material

Handouts for students and notes for the instructor are available in this issue of *JCE Online*.

## Literature Cited

- Stratton, W. J.; Steehler, G. A.; Pienta, N. J.; Middlecamp, C. H. *Chemistry in Context Laboratory Manual: Applying Chemistry to Society*, 4th ed.; McGraw Hill: Boston, 2003.
- Richardson, B. C.; Chasteen, T. G. *Experience the Extraordinary Chemistry of Ordinary Things*, 3rd ed.; John Wiley & Sons: New York, 1998.
- Greco, T. G.; Rickard, L. H.; Weiss, G. S. *Experiments in General Chemistry: Principles and Modern Applications*, 8th ed.; Prentice Hall: Upper Saddle River, NJ, 2002; (vinegar) pp 65–70; (aspirin tablet) pp 107–118; (antacid) pp 187–192; (water hardness) pp 227–234.
- Hunt, H. R.; Block, T. F.; McKelvy, G. M. *Laboratory Experiments for General Chemistry*, 4th ed.; Brooks/Cole: Monterey, CA, 2002; (vinegar) pp 97–104; (cola drink) pp 257–264.
- Wentworth, R. A. D. *Experiments in General Chemistry*, 7th ed.; Houghton Mifflin Company: Boston, 2003; (vinegar) pp 97–104; (water hardness) pp 225–236.
- Heasley, V. L.; Christensen, V. J.; Heasley, G. E. *Chemistry and Life in the Laboratory*, 5th ed.; Prentice Hall: Upper Saddle River, NJ, 2000; (bleach) pp 79–84.
- Bettelheim, F. A.; Landesberg, J. M. *Laboratory Experiments for General, Organic, and Biochemistry*, 4th ed. Harcourt College Publishers: Fort Worth, TX, 2001; (antacid) pp 239–246; (vitamin) pp 507–514.
- Zvi, S.; Pike, R. M.; Foster, J. C. *Microscale General Chemistry Laboratory with Selected Macroscale Experiments*, 2nd ed.; John Wiley: New York, 2003; (vitamin) pp 195–204; (antacid) pp 249–258.
- Mowery, K. A.; Blanchard, D. E.; Smith, S.; Betts, T. A. *J. Chem. Educ.* **2004**, *81*, 87.
- Davis, C. M.; Mauck, M. C. *J. Chem. Educ.* **2003**, *80*, 552.
- Mabrouk, P. A.; Castriotta, K. *J. Chem. Educ.* **2001**, *78*, 1385.
- Herman, H. B.; Jezorek, John R.; Tang, Z. *J. Chem. Educ.* **2000**, *77*, 743.
- Hall, J. F. *Experimental Chemistry*, 6th ed.; Houghton Mifflin Company: Boston, 2003; (antacid) pp 361–374.
- Wink, D. J.; Gislason, S. F.; Kuehn, J. E. *Working with Chemistry: A Laboratory Inquiry Program*; W. H. Freeman and Company: New York, 2000; (antacid) pp 103–108.
- Bishop, C. B.; Bishop, M. B.; Whitten, K. W. *Standard and Microscale Experiments in General Chemistry*, 4th ed.; Saunders College Publishing: Fort Worth, TX, 2000; (antacid) pp 309–320.
- Acid–Base Back Titration: Analysis of a Commercial Antacid. <http://www.academic.marist.edu/~jffp/chem351E1.htm> (accessed Mar 2006).
- Analysis of Commercial Antacids. <http://www.owl.net.rice.edu/~chem151/lab151/antacidB/> (accessed Mar 2006).
- Antacid Analysis: A Back-Titration. <http://icn2.umeche.maine.edu/genchemlabs/Antacid/antacid2.htm> (accessed Mar 2006).
- Martin, G. *J. Chem. Educ.* **1988**, *65*, 214.
- Burden, Stanley L.; Petzold, Christopher J. *J. Chem. Educ.* **1999**, *76*, 1544.
- Postma, J. M.; Roberts, J. L., Jr.; Hollenberg, J. L.; Postma, J. M. *Chemistry in the Laboratory*, 5th ed.; W. H. Freeman: New York, 2000; (water hardness) pp 367–376.
- Murov, S.; Stedjee, B. *Experiments and Exercises in Basic Chemistry*, 6th ed.; John Wiley: New York, 2004; (water hardness) pp 175–186.
- Slowinski, E. J.; Wolsey, W. C.; Masterton, W. L. *Chemical Principles in the Laboratory with Qualitative Analysis*, 6th ed.; Saunders College Publishing: Fort Worth, TX, 1997; (water hardness) pp 197–204.
- Complexometric Ca Determination. <http://www.csudh.edu/oliver/che230/labmanual/calcium.htm> (accessed Mar 2006).
- Complexometric Titration of Calcium in Antacids. <http://www.ic.sunysb.edu/Class/che134/susb/susb017.pdf> (accessed Mar 2006).
- Determination of Copper and Nickel by Complexometric Titration. <http://www.ipfw.edu/chem/321/Edta.pdf> (accessed Mar 2006).
- Ceretti, H.; Hughes, E. A.; Zalts, A. *J. Chem. Educ.* **1999**, *76*, 1420.
- Yappert, M. Cecilia; DuPre, Donald B. *J. Chem. Educ.* **1997**, *74*, 1422.
- Novick, S. G. *J. Chem. Educ.* **1997**, *74*, 1463.
- Weigand, W. A. *J. Chem. Educ.* **2000**, *77*, 1334.
- Hudson, R. J. M.; Rue, E. L.; Bruland, K. W. *Environ. Sci. Technol.* **2003**, *37*, 1553–1562.
- Jo, K. D.; Dasgupta, P. K. *Talanta* **2003**, *60*, 131–137.