are placed on a Petri dish and gradually heated on a hot plate. The ASA melts entirely before the SA begins to melt.

Formation of Colored Complexes

SA readily forms complexes with iron, but ASA does not (*6*). A pea-sized amount of each solid is placed in two different test tubes. To each is added 5 mL of tapwater and 5 mL of isopropyl rubbing alcohol (70%). A few staples (check to be sure they are magnetic) are put into each tube. A violet color will appear in the tube containing SA. Gentle heating by running hot water over the tube may be needed.

Acknowledgment

We want to thank Jia Wei for providing the NMR and IR on our final product and Michael Pence for supplying information about the Philadelphia water supply. We are also grateful for helpful conversations with Anthony Addison, Robert Hutchins, and Peter Wade. Finally, we wish to thank the Drexel University general-chemistry students who helped us to test and refine our procedure.

Literature Cited

1. Ault, A. *Techniques and Experiments for Organic Chemistry*, 5th ed.; Allyn and Bacon: Boston, 1987; p 325.
2. Lowrance, W. W. *Tetrahedron Letters* **1971**, *37*, 3453–3454.
3. Doyle, M.; Plummer, B. F. *J. Chem. Educ.* **1993**, *70*, 493.
4. Brewster, R. Q.; VanderWerf, C. A.; McEwen, W. E. *Unitized Experiments in Organic Chemistry*; Van Nostrand: New York, 1977; p 28.
5. Most, Jr., F. *Experimental Organic Chemistry*; Wiley: New York, 1988; p 376.
6. *Comprehensive Organometallic Chemistry*; Pergamon, 1982; Chapter 15.7, p 461.

Determinations of Carbon Dioxide by Titration

New Experiments for General, Physical, and Quantitative Analysis Courses

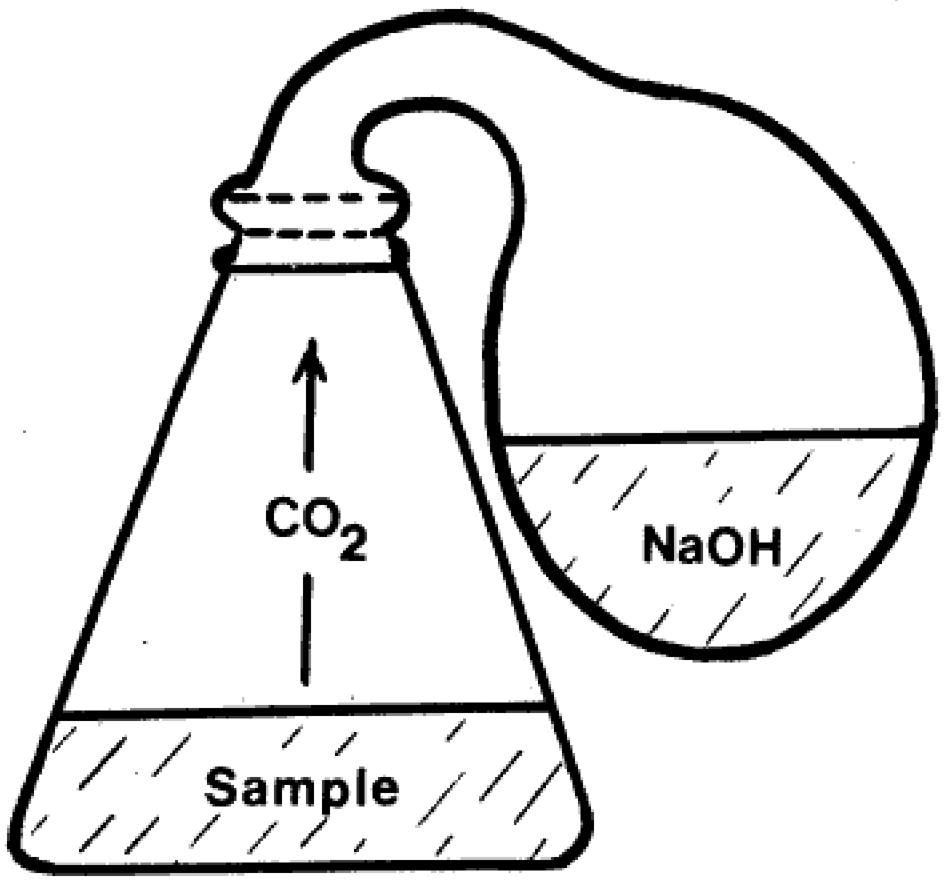
S. K. Crossno and L. H. Kalbus
California State University, San Bernardino, CA 92407

G. E. Kalbus
California State University, Long Beach, CA 90840

An important laboratory experiment often included in a quantitative analysis course is the titration of mixtures containing various combinations of sodium hydroxide, sodium carbonate, and sodium bicarbonate (*1, 2*). Such an experiment illustrates the principles of acid–base reactions and presents an interesting problem in stoichiometry for students. A recently developed experiment also provides for a variety of new experiments involving the analysis of commercial products, such as carbonated beverages and antacids, and makes use of rubber balloons in the laboratory apparatus (*3*).

Any sample capable of releasing carbon dioxide, either by physical agitation or by acid treatment, can be analyzed by this method. Because many real life commercial samples contain dissolved carbon dioxide gas or some type of carbonate, the experimental procedure can be modified to accommodate the analysis of all such samples. The evolved carbon dioxide gas is passed into a balloon containing excess sodium hydroxide solution where it is absorbed and converted into an equivalent amount of sodium carbonate. Containment of the carbon dioxide in such a closed, expandable system provides the necessary contact time with the sodium hydroxide for complete absorption, which is not possible if passed through an open container of excess sodium hydroxide.

The resulting mixture consisting of the excess sodium hydroxide and sodium carbonate is titrated with standard HCl. Titration to the first colorless phenolphthalein endpoint neutralizes the excess sodium hydroxide and converts all of the sodium carbonate into sodium bicarbonate. Continuation of the titration to the second methyl orange endpoint converts the sodium bicarbonate to water and carbon dioxide. The difference in milliliters between the first and second endpoints is used to calculate the carbon dioxide present in the sample or the grams of substance being sought. When this technique was applied to the analysis of carbon dioxide evolved from standard sodium carbonate, recoveries between 98 and 100% were obtained.



CO_2 absorption apparatus.

The following equation is used and its derivation should also be carried out by the student.

$$\frac{\text{volume of titrant (L)}}{\text{(1st to 2nd endpoint)}} \times \frac{\text{molarity of}}{\text{standard acid}} \times \frac{\text{molecular}}{\text{weight of } CO_2} = \frac{\text{mass of}}{CO_2\text{ (g)}}$$

In general, soluble carbonates are usually determined by direct titration with standard acid and insoluble carbonates by the addition of excess acid followed by back titration with NaOH. However, these determinations can be carried out by the method illustrated above, which is also the foundation for analyses that cannot be determined by direct titration or by the back-titration method.

Blank determinations indicate that the amount of contamination of the NaOH by atmospheric carbon dioxide is undetectable in these titrations (*4*). Also, some samples

---

Based on the paper presented at the 205th meeting of the American Chemical Society, Denver, March 1993.

need to be treated with acid to evolve the carbon dioxide; then atmospheric carbon dioxide absorption by the sample is of no concern. Furthermore, carbonate-free sodium hydroxide was prepared and used in these experiments (*5*). Based on this technique, the method was extended to the following student experiments (*6, 7*).

Student Experiments

The Determination of Carbon Dioxide in Carbonated Beverages

Carefully pour the cold 12-oz. beverage to be analyzed into a 500-mL Erlenmeyer flask. Pour about 100 mL of NaOH (approximately 1 M) into an adequately large balloon. Immediately secure the balloon around the neck of the flask; be sure not to spill any of the NaOH into the flask. If the balloon is positioned as illustrated in the figure there is no danger of the NaOH entering into the reaction flask.

NaOH leaches out some color from the balloon. Because this could interfere with the detection of the endpoints, a colorless balloon is preferred. Do not use red, orange, yellow, or pink balloons. If any NaOH is consumed by the balloon dye, there is no effect on the results because the results are determined by the difference between the first and second endpoints, which are solely dependent on the CO_2 evolved.

With a bath sonicator or magnetic stirrer, slowly agitate the flask at first. Then after the vigorous fizzing has ended, continue with greater agitation for 30 min. Allow the flask to stand overnight or a minimum of 10 h, and the balloon will shrink and collapse, demonstrating the absorption of the CO_2 by the NaOH. (If NaOH is allowed to stand in contact with the balloon for more then one week, the balloon may disintegrate some.)

Quantitatively transfer the contents of the balloon into a 250-mL Erlenmeyer flask. Add three drops of phenolphthalein to the flask. Using a magnetic stirrer, titrate the solution with standard HCl (approximately 1 M) until colorless. Record the volume used to the first endpoint. Next, add four drops of methyl orange and complete the titration until an orange color is obtained. Record the volume used to the second endpoint and calculate the amount of carbon dioxide in the beverage. If desired, the volume of carbon dioxide in the beverage at STP can be calculated. The student results ranged from 730 to 800 mL of carbon dioxide and compared favorably with the manufacturer's estimates of 700 to 1000 mL of carbon dioxide dissolved in a 12-oz. container of 7-Up.

The Determination of Sodium Bicarbonate in Alka-Seltzer

Place one tablet of Alka-Seltzer in a 150-mL filter flask. The side arm should be snugly fitted with a serum cap. Measure out approximately 40 mL of NaOH (approximately 1 M) and pour into an adequately large balloon, which is then fitted around the neck of the flask. Using a plastic syringe, inject 50 mL of HCl (approximately 1 M) through the serum cap. Any possible volatilization of HCl in the acid media does not affect the difference between the first and second endpoints. After evolution of the gas ceases, inject enough distilled water into the flask to almost reach the side arm, forcing the carbon dioxide into the balloon. Agitate the flask and allow it to stand for a minimum of 10 h.

Proceed as described in the previous experiment. From the first and second endpoints calculate the amount of sodium bicarbonate present in the tablet. Student results ranged from 1.65 to 1.70 g as compared to the manufacturer's estimates of 1.710 g. The same technique can be applied to the analysis of carbonates and commercially available antacids such as Tums, Maalox, and Rolaids, which contain $CaCO_3$ and $MgCO_3$ or both.

Determining the Molecular Weight of Carbon Dioxide

- ***Caution***: Safety goggles must be worn when using HCl and NaOH.

Measure out approximately 40 mL of 1 M NaOH and pour into a 250-mL Erlenmeyer flask. Attach a rubber balloon (about 15-in. inflatable diameter) to the neck of the flask and accurately weigh the entire apparatus. Now remove the balloon and drop a piece of dry ice (about the size of a pea (0.3–0.7 g)) into the flask. Then quickly reattach the balloon to keep the carbon dioxide from escaping. Allow the flask to stand for a minimum of 10 h. Weigh the entire apparatus again and determine the mass of dry ice by the difference in weights. Remove the balloon and titrate the contents of the flask as before with standard HCl (two endpoints).

Calculate the molecular weight of carbon dioxide using the following equation.

$$\frac{\text{mass of dry ice (g)}}{\begin{array}{c}\text{volume of titrant (L)}\\ \text{(1st to 2nd endpoint)}\end{array} \times \begin{array}{c}\text{molarity}\\ \text{of titrant}\end{array}} = \begin{array}{c}\text{molecular weight}\\ \text{of carbon dioxide}\end{array}$$

Only a minimal amount of inexpensive equipment, normally available at most institutions, is required for these determinations. Furthermore, the use of commercially available real-life samples and the use of balloons serve to intensify student interest.

Literature Cited

1. Hargis, L. G. *Analytical Chemistry*; Prentice: New Jersey, 1988; pp 179–181.
2. Dudek, E. *J. Chem. Educ.* **1991**, *68*, 948–950.
3. Arnaiz, F. J. *J. Chem. Educ.* **1993**, *70*, 1020–1021.
4. Macca, C. *J. Chem. Educ.* **1986**, *63*, 691–693.
5. Skoog, D. A.; West, D. M. *Analytical Chemistry*, 2nd ed.; Holt: New York, 1974; p 263.
6. Bent, H. A. *J. Chem. Educ.* **1987**, *64*, 167–171.
7. Kalbus, L. H.; Petrucci, R. H. *J. Chem. Educ.* **1971**, *48*, 107.