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Laboratory Experiment

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Gravimetric Analysis of Bismuth in Bismuth Subsalicylate Tablets: A Versatile Quantitative Experiment for Undergraduate Laboratories

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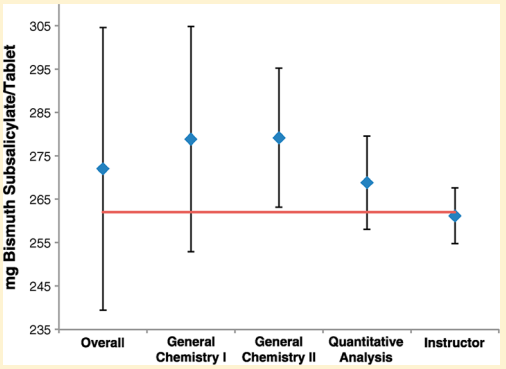
Supporting Information

ABSTRACT: In this laboratory experiment, lower- and upper-division students dissolved bismuth subsalicylate tablets in acid and precipitated the resultant Bi^{3+} in solution with sodium phosphate for a gravimetric determination of bismuth subsalicylate in the tablets. With a labeled concentration of 262 mg/tablet, the combined data from three different courses (general chemistry I and II and quantitative analysis) determined an average tablet concentration of 272.0 mg/tablet, with a standard deviation of ±48.46 mg. When analyzed by upper-division students, an average of 268.8 mg/tablet with a standard deviation of 16.02 mg was obtained, which confirmed the labeled concentration. In addition, students were given an opportunity to react the remaining solution with aluminum to reduce the ionic bismuth and produce a solid metal product, which reinforced the concepts of precipitation, stoichiometry, and reduction−oxidation in a single laboratory experiment.

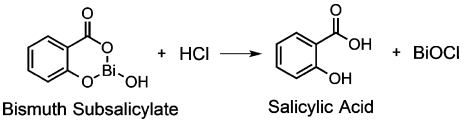


KEYWORDS: *High School/Introductory Chemistry, First-Year Undergraduate/General, Second-Year Undergraduate, Upper-Division Undergraduate, Analytical Chemistry, Laboratory Instruction, Gravimetric Analysis, Precipitation/Solubility, Quantitative Analysis*

INTRODUCTION

Gravimetric analysis provides a robust and simple method for the quantitation of an unknown compound that has been used historically in undergraduate laboratories.[1−6] When coupled with the topics commonly encountered in a general chemistry course, gravimetric laboratory exercises provide students with the opportunity to apply concepts of solubility, stoichiometry, concentration, moles, mass, data analysis, and statistics within a single experiment.[5,7] While gravimetric analysis has lost favor to more advanced techniques, its simplicity in execution and required materials makes it ideal to teach quantitative techniques to chemistry students of any undergraduate level;[7,8] however, many of the experiments commonly performed for this purpose hold little perceived interest for students without any real-world connection.[9]

A simple connection to the majority of general chemistry students lies within the medical field; many general chemistry students are on premedical school paths and thus have interests within biomedicine.[9] Thus, the investigation of pharmaceutical agents is a prime area to engage student interest while classroom topics are reinforced. Since the vast majority of pharmaceuticals utilize complex organic compounds, many of these agents cannot be easily investigated by the lower-division chemistry student; however, the indigestion agent bismuth subsalicylate (marketed as Pepto-Bismol) is the only current over-the-counter pharmaceutical that contains bismuth coordinated within its active ingredient. Recent interest has also sparked in the use of bismuth ion for antibacterial purposes.[10] A Popular Science article gave a method for the qualitative extraction of bismuth metal from bismuth subsalicyate tablets.[11] Through the addition of a precipitation reaction, the original quantity of bismuth subsalicylate within each pill may be calculated. The initial reaction liberates bismuth oxychloride from bismuth subsalicylate and leaves salicylic acid as a byproduct:



[1]

However, bismuth oxychloride is an insoluble compound in a neutral or basic solution; thus, this procedure maintains the solution in acidic conditions in order to drive the formation of dissolved bismuth chloride:

$$Bi^{3+}(aq) + Cl^{-}(aq) + H_2O \rightleftharpoons BiOCl(s) + 2H^{+} \quad (2)$$

Since this is an equilibrium reaction, Le Chatelier's principle drives the reaction toward dissolved bismuth. Finally, the aqueous bismuth is reacted with excess phosphate ion to drive the formation of bismuth phosphate for gravimetric analysis:

$$Bi^{3+}(aq) + PO_4^{3-}(aq) \rightarrow BiPO_4(s) \quad (3)$$

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Thus, the nature of this laboratory procedure lends itself to versatility within the university space. Depending on the desired student learning outcomes of the class, the experiment may be utilized for introductory chemistry, advanced general chemistry, or upper-division quantitative analysis courses with simple modifications to the procedure and the scope of calculations performed by the student, which include such topics as stoichiometry, gravimetric analysis, and equilibrium chemistry.

EXPERIMENTAL DETAILS

Reagents

Bismuth subsalicylate pills (Bismuth Subsalicyate Tablets or generic) with a labeled concentration of 262 mg/tablet were obtained over-the-counter from a local pharmacy. Students analyzed a mixed variety of brand-name, generic, chewable, and traditional pills. Sodium phosphate (tribasic) (J.T. Baker, Center Valley, PA) was used at a concentration of 0.1 M, and 2 M hydrochloric acid (VWR, Radnor, PA) was used for pill dissolution.

Experimental Procedure

For general chemistry, the laboratory was designed to utilize a 2 h laboratory time slot and require 2 weeks to complete. The entire procedure could be finished in a single, 3 h laboratory time as well. Each student or group was provided 10 bismuth subsaclicylate pills. After each pill was weighed to find an average mass, the pills were pulverized into a fine powder via mortar and pestle. The resultant powder was quantitatively transferred to a 250 mL beaker, and 100 mL of 2 M HCl was added to the powder in 10 mL increments with continuous stirring from a glass rod. The reaction produces significant gas formation with each addition of HCl, but these usually ceased after approximately 50 mL had been added. The resultant solution was vacuum filtered with number 42 (fine texture) filter paper. Depending on the specific brand of bismuth subsalicylate tablets used, some solutions required multiple filtering steps through the same paper until no particulates remained in the solution (depending on the brand or flavor of tablets utilized, some solutions will be pink in color, while others will be clear). In these cases, centrifugation may be used to initially pellet the suspended solids prior to filtration. The solid was rinsed with approximately 50 mL of distilled water, vacuumed to dryness, and discarded. The remaining solution was brought to a total volume of 250 mL with distilled water in a volumetric flask.

Twenty-five milliliter aliquots of solution were reacted with excess (25 mL) 0.1 M Na_3PO_4 to produce a fine, white precipitate. The solution was vacuum-filtered onto preweighed number 311 (extra-fine texture) filter paper. Because of the extremely fine particulate size, the resulting solution often required 2−3 (or more) passes through the same piece of filter paper before the solution was clear of all precipitate. In a few rare cases, the precipitate required over seven filtrations to produce a clear solution. The filter paper and precipitate were dried under vacuum at 50 °C for 15−20 min and weighed to find a total mass of $BiPO_4$ obtained from the aliquot. If a vacuum oven is not available, the samples may be dried at 80 °C for approximately 1 h, or covered and placed in a secure location for drying before being weighed in subsequent weeks.

While the samples dried under vacuum, students placed the remaining solution into a 400 mL beaker and added approximately 15 g of aluminum pellets. The reduction−oxidation reaction between the aluminum metal and bismuth ion produced a solid bismuth precipitate that was filtered and melted in a crucible over a Bunsen burner into a "bb"-sized pellet, which the student could keep if desired. An important note is that the side-products of the melting of the bismuth permanently coated the container. It is suggested to perform this step in a dedicated or disposable container. If this laboratory method is utilized in an upper-division setting, additional experiments may be performed on a large sample of pure bismuth metal. Milán et al. demonstrated a method for the synthesis and characterization of bismuth crystals in an undergraduate laboratory setting.[12]

HAZARDS

Approved safety goggles, nitrile-based gloves, and standard laboratory personal-protective equipment must be worn at all times while this experiment is performed. Hydrochloric acid and sodium phosphate can cause severe caustic burns. All chemicals must be collected in permitted, labeled waste containers at the end of the experiment. The bismuth metal should be melted in the hood because of the outgassing of remaining chemicals, and care should be taken because of the extreme heat required to melt bismuth metal. Because of the extreme heat, any flaws in the crucible used for melting result in sudden, catastrophic failure of the ceramic; keep the hood-sash lowered during the melting process to avoid possible injury if this occurs.

RESULTS AND DISCUSSION

This experiment was performed by three classes of students at various levels of experience in chemistry to demonstrate the applicability of this method across a chemistry curriculum. These courses included the first and second semesters of college-level introductory (general) chemistry and an upper-division quantitative analysis course. The results are summarized in Figure 1 and tabulated in Table 1. An instructor also performed the experiment.

The course instructor found an average mass of bismuth subsalicylate per tablet of 261.2 mg with a standard deviation of ±7.64 mg, which resulted in a p_{crit} value of 0.7622 when compared to the anticipated 262 mg. Upper-division chemistry students in the quantitative analysis course produced an average mass of 268.8 mg/tablet with a standard deviation of ±16.02 mg, which resulted in a p_{crit} value of 0.1680 when compared to 262 mg/tablet. First semester general chemistry students found an average mass of 278.8 mg/tablet with a standard deviation of ±39.36 mg and a p_{crit} value of 0.1864. The increased average mass was most likely due to insufficient drying times, while the large standard deviation was likely due to the inexperience of these students. Finally, second semester general chemistry students produced an average mass of 279.2 mg/table with a standard deviation of ±22.00 mg and a p_{crit} value of 0.0293. In this case, the masses obtained resulted in a statistically different value. Again, the higher-than-anticipated mass was likely due to insufficient drying times in the time-constrained general chemistry laboratory.

When used as a general chemistry laboratory (first or second semester), this procedure was found to provide students with a practical application of stoichiometric theory and precipitation-based gravimetric analysis. In these courses, laboratory groups of two students dissolved ten pills and precipitated and weighed one aliquot from the total resultant solution. For general

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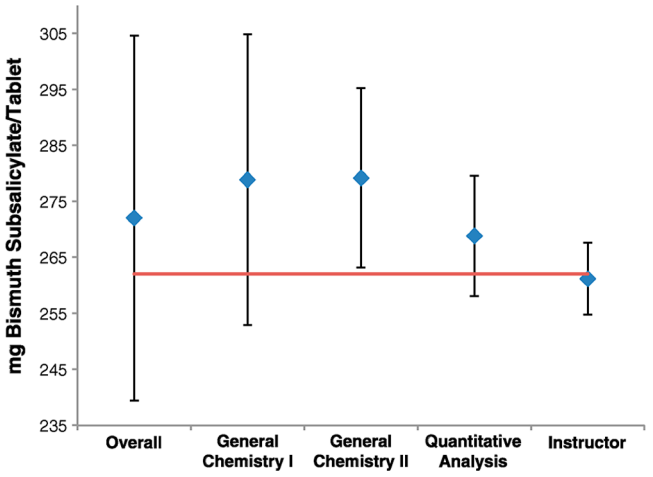

Figure 1. Summary of course data for the gravimetric analysis of bismuth subsalicylate. Error bars indicate 95% confidence interval of the data. Each course produced data that were statistically identical to the labeled concentration of bismuth subsalicylate for the tablets (262 mg/tablet) except for the general chemistry II course. In the referenced data, the general chemistry II course was taught by a teaching assistant (TA) inexperienced with this procedure, which led to insufficient drying of the samples and thus a greater-than-expected average mass; however, the data from the general chemistry II course may be considered statistically identical at the 90% confidence interval.

chemistry, students were not asked to statistically verify their results against the "actual" value as stated on the packaging. Instead, students were tasked with performing a percent yield calculation with the assumption that each pill had 262 mg of bismuth subsalicylate (laboratory manual and data sheet may be found in the Supporting Information). The extra solution provided a simple means for the reduction of Bi^{3+} to solid bismuth metal, which allowed a visible, qualitative experiment that reinforced reduction−oxidation reactions and provided a sample of bismuth metal large enough to be handled (each solution produced a pellet of bismuth the approximate size of a bb).

Lower-division student response to this laboratory was positive. The students appreciated the connection of use of a pharmaceutical agent with which they have likely had experience and the laboratory techniques that they had learned and practiced. In addition, the added factor of producing a solid metal from a pill they may have previously ingested reinforces the concept of ionic metals versus solid metals and the ability to change an oxidation state through a chemical reaction. When applied for first semester chemistry students, the emphasis was set on percent yield and stoichiometry. When utilized in second semester chemistry courses, additional emphasis may be added to the final step of adding Al to the cell. By using standard reduction potentials, students were asked to determine if a reaction was expected between solid aluminum and aqueous bismuth ion through an E_{cell} calculation. They were then able to test their hypotheses experimentally and comment on the results. While not a vital portion of the experiment, the dramatic formation of a black solid within their solution lends an excellent added teaching moment for the students in reduction−oxidation chemistry.

For upper-division quantitative analysis students, the focus was placed on the statistical analysis of the results. With the increased time allotted to these upper-division laboratories (2 credit hour laboratory; meeting two times per week for 3 h each; this lab consumed 1 week) and the increased laboratory experience of the students, the students each dissolved 10 pills and precipitated at least four aliquots from their resultant solutions. From these data, the students statistically analyzed their results initially for outliers and finally for a significant difference from the stated value. One additional variable in this laboratory could be the use of varying brands or batches of bismuth subsalicylate pills to compare consistency and quality across multiple producers. Finally, this experiment may also be applied toward the comparison of wet-bench chemical techniques to modern instrumentation if an atomic absorption or emission instrument is available.

CONCLUSIONS

A versatile undergraduate-level chemistry experiment was developed that may be applied within a general chemistry or upper-division quantitative analysis setting. This method allowed the accurate quantitation of bismuth subsalicylate in bismuth subsalicyate tablets as well as a qualitative reinforcement of reduction−oxidation theory.

ASSOCIATED CONTENT

Supporting Information

Document draft of the laboratory procedure utilized by the students who performed this experiment, which contains a student-oriented introduction, a stepwise laboratory procedure, data collection sheets, calculation walkthroughs, and postlaboratory questions utilized in the operation of this laboratory. Feel free to use or modify the attached document in any way necessary. This material is available free of charge via the Internet at http://pubs.acs.org.

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Table 1. Data Summary of the Gravimetric Determination of Bismuth Subsalicylate in Bismuth Subsalicyate Tablets[a]

	parameters					
results by course	average mass of bismuth subsalicylate per tablet (mg)	standard deviation	confidence interval (high)	confidence interval (low)	*p*-critical for 262 mg/tablet	total tablets analyzed
average	272.0	48.46	304.5	239.4	0.4792	250
general chemistry I	278.8	39.36	304.8	252.9	0.1864	110
general chemistry II	279.2	22.00	295.2	263.1	0.0293	90
quantitative analysis	268.8	16.02	279.6	258.1	0.1680	30
instructor	261.2	7.64	267.5	254.8	0.7622	20

[a]Student-developed data provided an accurate, quantitative comparison to the package labeled 262 mg/tablet upon statistical comparison.

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Notes

The authors declare no competing financial interest.

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