Determination of Al Content in Commercial Samples through Stoichiometry: A Simple Experiment for an Advanced High-School Chemistry Olympiad Preparatory Course

ARTICLE *in* JOURNAL OF CHEMICAL EDUCATION · JUNE 2014

Impact Factor: 1.11 · DOI: 10.1021/ed400683r

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JOURNAL OF CHEMICAL EDUCATION
Laboratory Experiment
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Determination of Al Content in Commercial Samples through Stoichiometry: A Simple Experiment for an Advanced High-School Chemistry Olympiad Preparatory Course

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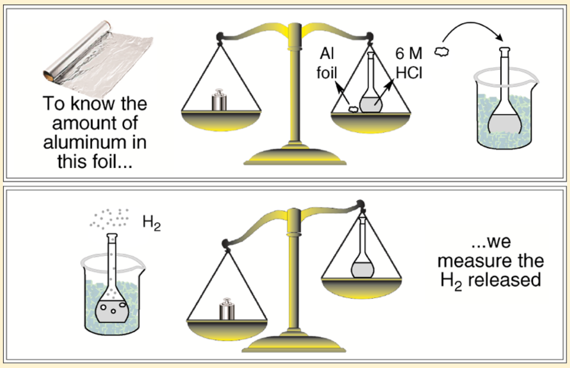
Ⓢ *Supporting Information*

ABSTRACT: Stoichiometry has always been a puzzling subject. This may be partially due to the way it is introduced to students, with stoichiometric coefficients usually provided in the reaction. If the stoichiometric coefficients are not given, students find it very difficult to solve problems. This article describes a simple 4-h laboratory experiment for the determination of aluminum content in commercial samples through the stoichiometric relationship between aluminum and released hydrogen gas. The experiment was performed by high-school students at an advanced preparation course for the Chemistry Olympiad. The aluminum content in foil samples was determined by measuring the amount of formed hydrogen gas through mass balance of the reaction system. The experiment is appropriate for high-school chemistry or a first-semester general chemistry course.



KEYWORDS: *High School/Introductory Chemistry, First-Year Undergraduate/General Chemistry, Laboratory Instruction, Hands-On Learning/Manipulatives, Applications of Chemistry, Oxidation/Reduction, UV−Vis Spectroscopy*

"We may lay it down as an incontestable axiom, that, in all operations of art and nature, nothing is created; an equal quantity of matter exists both before and after the experiment; the quality and quantity of elements remain precisely the same; and nothing takes place beyond changes and modifications in the combination of these elements."[1]

In Brazil, basic sciences suffer from a deficit of 250,000 public school teachers,[2] thus it is not difficult to identify chemistry as one of the most negatively affected disciplines. In order for Brazil to be affirmed as a technology leader, there must be a substantial improvement of science education and training of qualified personnel. The Chemistry Olympiad[3] is perfectly suited for this mission because it provides a discussion-rich environment and produces citizens endowed with intelligence, solidarity, and ethical standards, qualities that form the foundation of any modern society. In 2012, the Brazilian Chemistry Olympiad program was instituted in 13 states, involving undergraduate students of chemistry courses. This national project is aimed at promoting enthusiasm for chemistry, stimulating interest for sciences, and encouraging integration of Brazilian universities with public schools to develop teaching strategies.

The present chemistry laboratory experiment was performed by high-school students from public schools in the state of Rio Grande do Norte, Brazil, at a preparatory course for the Chemistry Olympiad. The goal was to determine the aluminum content in commercial aluminum foil based on the released hydrogen gas from the reaction between Al and HCl. This experimental activity focused on overcoming the dichotomy between theory and practice through the development of a conceptual appreciation and motivation for the study of stoichiometry.[4] This chemistry laboratory experiment successfully engaged the students in the course content with integration of the course objectives into the student's life experience. By dealing with an actual problem, students showed enthusiasm and motivation to work in a case in which stoichiometric coefficients are unknown.

■ EXPERIMENTAL PROCEDURE

Determination of the Ratio between the Stoichiometric Coefficients of Aluminum and Hydrogen Gas

Hydrochloric acid, 10 mL of 6.0 mol L^{-1}, in a volumetric flask and approximately 0.100 g of pure aluminum were weighed as shown in Figure 1A. The volumetric flask containing the hydrochloric acid was then inserted into an ice bath for about 10 min until the temperature dropped to ~0 °C. While still in the ice bath, the pure aluminum was carefully dropped into the volumetric flask as shown in Figure 1B. After about 5 min, hydrogen began to evolve (Figure 1C). Reaction completion

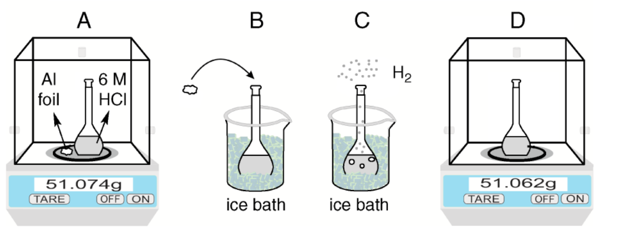


Figure 1. Experimental arrangement: (A) preweighing, (B) addition of the aluminum to the cooled HCl solution, (C) evolution of the hydrogen gas, and (D) postweighing.

was realized by cessation of hydrogen gas evolution, which took approximately 30 min. Afterward, the volumetric flask was removed from the ice bath, dried with a paper tissue, and weighed as shown in Figure 1D.

Determination of the Aluminum Content in a Commercial Aluminum Foil

The previous procedure was repeated with the pure aluminum replaced by the commercial Al sample.

■ HAZARDS

The HCl solution is corrosive and can cause burns upon skin contact. The students should wear safety goggles and rubber gloves.

■ RESULTS

To illustrate how the law of mass conservation and the mole concept were utilized, the stepwise resolution of the problems is presented in Tables 1 and 2. Table 1 is a guide to calculate

Table 1. Experimental Data Analysis Using Pure Aluminum

procedure	step
$a\text{Al(s)} + b\text{HCl(aq)} \rightarrow c\text{Al}^{3+}\text{(aq)} + d\text{Cl}^{-}\text{(aq)} + e\text{H}_2\text{(g)}$ Unknown stoichiometric coefficients	1
$\frac{n_{Al}}{a} = \frac{n_{H_2}}{e}$ Mole concept	2
$n_{Al} = \frac{m_{Al}}{M_{Al}} = \frac{0.1136\ \text{g}}{26.9\ \text{g/mol}} = 4.22 \times 10^{-3}\ \text{mol}$ Mole concept	3
$m_{H_2} = m_1 - m_2 = 51.0748 - 51.0620 = 0.0128\ \text{g}$ Law of mass conservation, where m_1 is the preweight and m_2 is the postweight	4
$n_{H_2} = \frac{m_{H_2}}{M_{H_2}} = \frac{0.0128\ \text{g}}{2.02\ \text{g/mol}} = 6.34 \times 10^{-3}\ \text{mol}$ Mole concept	5
$\frac{a}{e} = \frac{n_{Al}}{n_{H_2}} = \frac{4.22 \times 10^{-3}\ \text{mol}}{6.34 \times 10^{-3}\ \text{mol}} = 0.665 = \frac{2}{3}$ Mole concept	6

the a/e ratio, the mole ration between Al and H_2, by using a pure aluminum sample, which ensures the H_2 formation to be solely due to Al oxidation. First the general chemical equation is written and the stoichiometric coefficients are left unidentified. In step 6, the a/e ratio is found to be 0.66 (2/3), which is exactly the relationship between aluminum and hydrochloric acid found in textbooks.[5] However, to reach this result, the reaction must be carried out in an ice bath. The first

Table 2. Experimental Data Analysis Using Commercial Aluminum Foil

Procedure	Step
$\frac{a}{e} = \frac{n_{Al}}{n_{H_2}} = 0.66$ Mole concept	1
$m_{H_2} = m_1 - m_2 = 50.8418 - 50.8277 = 0.0141\ \text{g}$ Law of mass conservation	2
$n_{H_2} = \frac{m_{H_2}}{M_{H_2}} = \frac{0.0141\ \text{g}}{2.02\ \text{g/mol}} = 7.05 \times 10^{-3}\ \text{mol}$ Mole concept	3
$\frac{a}{e} = \frac{n_{Al}}{n_{H_2}} = \frac{n_{Al}}{7.05 \times 10^{-3}\ \text{mol}} = 0.66 = n_{Al} = 4.65 \times 10^{-3}\ \text{mol}$ Mole concept	4
$m_{Al} = n_{Al} \times M_{Al} = 4.65 \times 10^{-3}\ \text{mol} \times 26.9\ \text{g/mol} = 0.1251\ \text{g}$ Mole concept	5
$\%\text{Al} = \frac{m_{Al}(\text{found})}{m_{Al}(\text{weighed})} \times 100 = \frac{0.1251\ \text{g}}{0.1369\ \text{g}} \times 100 = 91.38\%$ Aluminum content	6

experiments with the system at room temperature yielded an a/e ratio of 0.050 (1/20), meaning that 1 mol of aluminum would generate 20 mol of hydrogen gas! The students were then asked about possible sources of errors. Some of them realized that the reaction vessel after the experiment was warmer than in the beginning. The conclusion was that some gaseous HCl also evolved due to heating causing an overestimation of the mass loss. This is a valid assumption since HCl is far heavier than H_2. By keeping the reaction flask in an ice bath, the temperature is low during the entire reaction, hence suppressing HCl evolution.

In order to find the amount of Al in a commercial sample, the a/e ratio determined in Table 1 was applied as demonstrated in Table 2, with the percentage of Al being 91.4%. This result was confirmed by the authors using colorimetry[6] and wavelength-dispersive X-ray spectroscopy (WDS).[7]

■ DISCUSSION

A total of 15 high school students were divided into 3 groups of 5 students each. The activity began with a 30 min briefing by the undergraduate teaching assistant (TA). The TA presented the students with the problem to be solved using stoichiometry concepts and provided the students with the above-mentioned experimental procedure. The groups carried out the experiment independently. During the postlaboratory discussion, the TA used the students' data to guide them into the understanding of the quantitative relationships between reactants and products, as well as the concept that known quantities of reactants can be used to predict the quantity of products, following the fundamental statement of stoichiometry: "the known defines, delimits, or determines the unknown".[8]

To our surprise none of the groups was able to solve the proposed problem. The students encountered difficulty in comprehending the law of conservation of mass and concept of the mole. In addition, an interesting point in the experiment was that the measured mass of the system after the reaction was, in all experiments, lower than the mass of the system before the reaction. This was an ideal opportunity to question the students about the quantitative relationship between the

dx.doi.org/10.1021/ed400683r | J. Chem. Educ. XXXX, XXX, XXX−XXX

amount of aluminum undergoing reaction and the amount of hydrogen being produced, especially because most of the stoichiometry problems deals with closed systems: "the mass before and after was different, was the conservation law violated?" "How would you go about establishing the connection at the molecular level between moles of aluminum and moles of hydrogen?" "What are the coefficients for the balanced chemical equation?" "Is it necessary to use cross multiplication to solve this experiment?"

One explanation for this is that most chemistry textbooks we reviewed[9,10] introduce the concept of stoichiometry through reactions in which the compounds and their stoichiometric coefficients are *all known a priori.* Some students advanced in balancing the equation based on the charge 3+ of the aluminum ion; however, this action was discouraged by the TA because the present method is general and enables the resolution of the problem even if the charge of the cation is not known. As this experiment was designed to simulate a Chemistry Olympiad, we took the opportunity to introduce the fundamental laws for the resolution of any stoichiometric problem: (i) the law of mass conservation and (ii) the mole concept.

Two analytical methods, colorimetry and WDS, were employed by the instructors to confirm the percentage of aluminum in the commercial foil. Colorimetry is based on the color change due to the reaction between Eriochrome Cyanine R (ECR) and aluminum ions. This technique was only employed to confirm the results found with stoichiometry, and it is not intended to be carried out by students. Full experimental details for this step are given in the student handout in the Supporting Information. Figure 2 shows a series

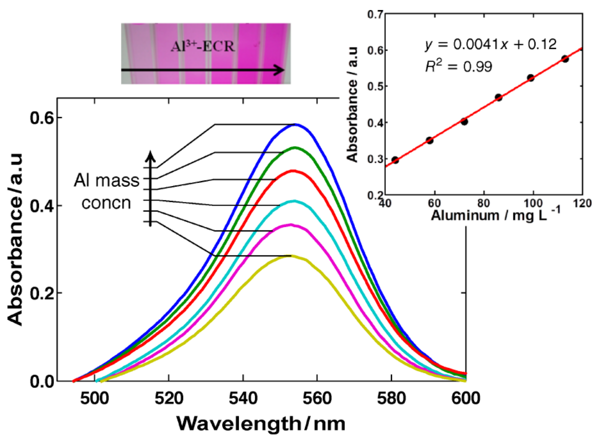


Figure 2. Spectra of the Al^{3+}-ECR complex for different Al^{3+} concentrations.

of spectra whose absorbance increased with the Al^{3+} concentration. By plotting the maxima of the curves against the respective concentration, one generates the calibration curve (inset of Figure 2). The mean concentration of aluminum for three determinations was 89.5 mg/L, which gives 87.7% Al in the commercial sample, therefore corroborating the result found through stoichiometry. The value found with colorimetry was somewhat lower than that determined with the present method (91.4%), and reasons for this are discussed with the help of WDS.

WDS has been employed only to confirm the results found with stoichiometry. It is not intended to be carried out by students. Table 3 shows the elements present in the commercial sample as well as their respective percentages.

Table 3. WDS Results for the Commercial Aluminum Foil

Element	Mass (%)
aluminum	87.4
carbon	7.2
iron	5.4

One can see that carbon and iron appear as impurities. As iron is a non-noble metal, it is also subjected to oxidation by HCl contributing to generation of H_2, leading to an overestimation of the Al content in the sample. From WDS results iron constitutes 5.4% of the commercial sample. In Table 2 the amount of commercial aluminum used was 0.1369 g, of which 7.40 mg are, therefore, iron. Considering that mainly Fe^{2+} was generated due to the strong acid environment,[11] 0.264 mg of H_2 was generated from the complete oxidation of Fe. The correct H_2 released solely due to Al is hence (see step 2) 14.1 mg − 0.264 mg = 13.8 mg. Going down the steps of Table 2 with this new value, one reaches 88.7% of aluminum, a closer value to those determined by independent techniques.

Non-noble metals such as Mg, Zn, and Fe present in the sample can be regarded as interfering species. This was an excellent opportunity to discuss with the students possible sources of errors and emphasize that care must be taken when analyzing the results.

This simple experiment can be used to explore the fundamental concepts of stoichiometry during a preparatory course for the chemistry Olympiads exam. Because an open system is used in this laboratory exercise, it is not obvious that conservation of mass is maintained. In textbook examples and most laboratory experiments involving stoichiometric concepts the systems are closed. The experiment developed in this article is also appropriate for an undergraduate general chemistry or instrumental analysis course exploring concepts of colorimetry and microscopy.

■ CONCLUSION

Stoichiometry is indeed a puzzling subject. Even above-average students encountered difficulties in solving the herein proposed activity. Not providing stoichiometric coefficients for the reaction was found detrimental to solving the problem. We believe that high school chemistry textbooks should include problems similar to the one proposed, in which stoichiometric coefficients are unknown. Finally, we learned with the feedback from the students that they were more motivated in carrying out the activity because there was an actual problem to be solved, namely, finding the amount of aluminum in a commercial sample.

■ ASSOCIATED CONTENT

Supporting Information

Note for instructors, student handout, the experimental details of the colorimetric determination of aluminum, and the basics of WDS analysis. This material is available via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

The authors are grateful to Coordenação de Aperfeiçoamento de Ensino Superior (Capes) and Institute of Chemistry (UFRN).

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