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Laboratory Experiment

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Using a Laboratory Inquiry with High School Students To Determine the Reaction Stoichiometry of Neutralization by a Thermochemical Approach

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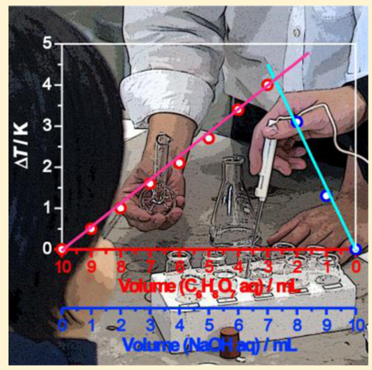
Supporting Information

ABSTRACT: This paper presents the design and practical application of a laboratory inquiry at high school chemistry level for systematic chemistry learning, as exemplified by a thermochemical approach to the reaction stoichiometry of neutralization using Job's method of continuous variation. In the laboratory inquiry, students are requested to propose the principles of Job's method and of data analysis for determining the reaction stoichiometry, in addition to experimental data acquisition and analysis. When applying the laboratory inquiry to the acid–base reaction learning unit that is positioned subsequent to thermochemistry, the chemical knowledge and concepts of thermochemistry are actively used to reinforce the previous learning. The laboratory inquiry also constructs the basis for the forthcoming learning of acid–base titration. Such a laboratory activity tightly links the different learning units and is expected to be effective in the educational strategies involving systematic chemistry learning in high schools.



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The acquisition of novel chemical knowledge and concepts is promoted by the active use of previously acquired knowledge and concepts. Laboratory inquiry provides a possible opportunity for such integrated chemistry learning. Many daily use materials and chemical phenomena can be used for educational purposes.[1−4] On the basis of a spiral curriculum at high schools, the introduction of such inquiry-based learning into systematic chemistry learning appears to be important for intentionally linking learning contents from different learning units.[5] In addition, a laboratory inquiry, which is based on the previous learning content and leads to the subsequent one, is expected to be a possible method to tightly link the two learning units and to make the educational strategy of the systematic chemistry learning effective. Such a laboratory inquiry reinforces the previously acquired chemical knowledge and concepts through practical use and lays the basis for subsequent learning.

For the introduction of such a laboratory inquiry, well organized learning situations and learning subjects are necessary. As a possible learning situation, we used the acid–base reaction learning unit, which is positioned after that of thermochemistry in a high school chemistry course. A thermochemical approach to the reaction stoichiometry of neutralization using Job's method of continuous variation[6−8] can form a suitable laboratory exercise. This study aims to apply effectively the laboratory exercise to high school chemistry courses by finding the most effective learning situation in the curriculum, carefully designing the inquiry-based learning, improving the instruments, and simplifying the experimental procedures. A 2 h laboratory exercise in a combined style of a structured/guided inquiry was designed and applied for use by 10 4-member groups at a high school chemistry level, in which proposals of the principles of Job's method and data analysis for determining the reaction stoichiometry are part of students' tasks, in addition to the experimental data acquisition and analysis. This paper presents the detailed design and practical use of the laboratory inquiry on the basis of the educational practices in high school chemistry courses. It is expected that the results of the present study provide an example of the carefully organized chemistry teaching/learning for improving high school chemistry in the ongoing curriculums and for developing high school chemistry for the next generation.

BASIS OF THE LABORATORY INQUIRY DESIGN

Learning Situation

The laboratory inquiry was designed for students of a class in a high school chemistry course. In the class, students were already aware of the learning units covering the structure of materials, chemical reaction stoichiometry, and thermochemistry. In the thermochemistry learning unit, students performed the measurements of the dissolution enthalpy of sodium hydroxide and the neutralization enthalpy of hydrochloric acid and an aqueous solution of sodium hydroxide, followed by the

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verification of Hess's law using the experimental results. The present laboratory inquiry has been introduced during the acid–base learning unit, after learning the definitions of acid, base, and neutralization and before experiencing acid–base titration in the laboratory. The learning situation was strategically developed as a part of the present research by expanding and rearranging the ongoing high school chemistry curriculum.

Learning Task

The laboratory inquiry was designed to construct a learning base for the stoichiometry of neutralization and acid–base titration by expanding the previously acquired knowledge and concepts of the chemical reaction stoichiometry and thermochemistry. Determination of the reaction stoichiometry between aqueous solutions of citric acid ($C_6H_8O_7$) and sodium hydroxide (NaOH) using a thermometric method was selected as the subject of the inquiry. The tasks that were assigned in sequence to the students during the laboratory inquiry are as follows.

- Understanding the exothermic nature of the reaction
- Quantitative understanding of the variation in temperature change when varying the mixed volume (mass) ratio of the reactant solutions with the same molar concentration
- Proposing an experimental design based on Job's method
- Experimental data acquisition
- Determination of the reaction stoichiometry using an extrapolation procedure
- Calculation of the neutralization enthalpy from the experimental data
- Qualitative prediction of the pH of the product solutions obtained by mixing the reactant solutions with the same molar concentration in different volume (mass) ratios

■ LABORATORY INQUIRY

Introduction to the Subject

Two questions followed by demonstrations are effective for introducing the subject of the laboratory inquiry. The primary question is about the temperature change when mixing commercially available lemon juice that is used for cooking and an aqueous solution of sodium hydroxide. Students easily answered the question; they said that they expect an exothermic behavior of the reaction between the organic acid and sodium hydroxide. The students' expectation is successfully verified by a demonstration of measuring the temperature change during mixing the same volume of commercially available lemon juice and 0.5 M NaOH(aq), when a temperature increase by 2–3 K is observed. Some students mention citric acid as the major organic acid present in the lemon juice. Using this students' response, the instructor can replace lemon juice with an aqueous solution of citric acid (0.5 M) for the subsequent laboratory inquiry. If the lemon juice has the description of contents on the label of the bottle, students accept this replacement more easily. The chemical formula of citric acid is mentioned by the instructor as $C_6H_8O_7$, when the laboratory subject is introduced.

The other question concerns the relation of the quantities of acid and base solutions of the same concentration for mixing and the temperature change in the mixed solution. Students are asked about the magnitude of the temperature change when mixing different quantities of acid (0.5 M HCl(aq)) and base (0.5 M NaOH(aq)) to give 10 mL of mixed solutions, for example, 8 mL of HCl(aq) and 2 mL of NaOH(aq) or 6 mL of HCl(aq) and 4 mL of NaOH(aq). For this question, students appropriately selected the case of the larger temperature increase by considering the reaction stoichiometry of the neutralization between HCl(aq) and NaOH(aq). The answer is verified by the demonstration of mixing HCl(aq) and NaOH(aq) in different volume ratios by the instructor. Through answering the questions and discussing the results of the accompanying demonstrations, students' understanding of the thermochemical relationship of the neutralization is reinforced and the basis for proposing the experimental principle and procedures for the determination of reaction stoichiometry for the neutralization by the thermometric method are constructed.

Proposal of the Experimental Principle

Based on the introductory demonstrations, the determination of reaction stoichiometry for the neutralization between $C_6H_8O_7$(aq) and NaOH(aq) is suggested as the subject of the laboratory exercise. Students are requested to propose the experimental procedure to determine the reaction stoichiometry using temperature measurements.

After discussing in groups for approximately 10 min, students proposed the experimental procedure in line with Job's method by referencing the experiments in the introductory demonstrations. At the same time, the students listed the necessary procedures and conditions for the experiments, which are as follows.

- Equivalent concentrations of $C_6H_8O_7$(aq) and NaOH(aq)
- Equivalent temperatures of $C_6H_8O_7$(aq) and NaOH(aq)
- Systematic change of the volume ratios of $C_6H_8O_7$(aq) and NaOH(aq) for mixing and precise measurement of the volumes
- Temperature measurements obtained before and after mixing the solutions and precise measurement of temperatures
- Safety countermeasures for treating acid and base solutions
- Reduction of the amount of chemicals used for the experiment in light of environmental issues and efficient use of resources

For each procedure and condition listed by the students, the instructor inquires about the reasoning and the possible solution in the proposed experiment. After discussing each procedure and condition in the class, the instructor introduces the instrument prepared for the students' experiment, with the necessary instructions for its use. The idea proposed by the students is also emphasized to closely correspond to the continuous variation method for determining the reaction stoichiometry proposed by Job in 1925.[9]

Experimental Work

A complete manual of the experimental procedures in line with the students' proposal and class discussion is provided by the instructor after the discussion, that is, at the beginning of the experimental work, as the second part of the student handout (see Supporting Information). The experimental procedure is practically the same as those proposed previously,[6–8] but reduction of the amount of reagents and improvement of the calorimetric capability were accomplished in this study. After being provided the complete manual of the experimental

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procedures, students are required to look into the appropriateness of the procedures, from which students' recognition of the roles of each experimental procedure is deepened.

Figure 1 illustrates the instruments and experimental procedures. Simplified calorimetric vessels comprising 20 mL glass sample bottles (totally 18 pieces) arranged in two rows in a block of polystyrene foam with holes for each bottle are used for the measurements (Figure 1a). The 0.5 M $C_6H_8O_7$(aq) and 0.5 M NaOH(aq) in the continuously varying volume ratio of 1:9–9:1 and a total volume of 10 mL are carefully transferred into one of the pair of the sample bottles for each solution using 10 mL measuring cylinders (Figure 1b). A pen-type digital thermometer is used for the temperature measurements. Using the thermometer, the temperature of one of the solutions in the pair of sample bottles with the larger volume is measured as the initial temperature of the sample solutions (Figure 1c). Then, the other solution with the smaller volume is added to the solution with the larger volume, and the temperature of the mixed solution is measured as the final temperature (Figure 1d). Students repeat the procedure for nine different volume ratio pairs and record the initial and final temperatures of the solutions.

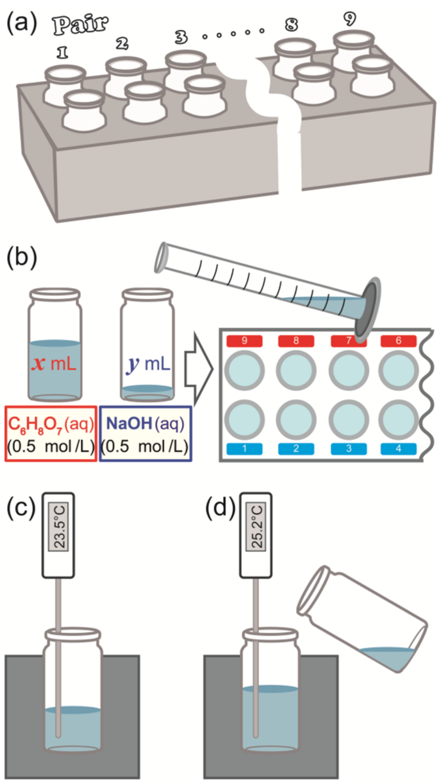

Figure 1. Schematics of the instruments and experimental procedures: (a) instruments; (b) setup for the reactant solutions; (c) temperature measurement of the reactant solution; and (d) temperature measurement of the resultant solution.

Data Analysis

The experimental measurements for the temperature change of the solution at different mixed ratios are analyzed by each student group. In the student handout, it is suggested to draw a graph by plotting the temperature change against the mixed ratio of $C_6H_8O_7$(aq) and NaOH(aq) solutions. From the graph (Figure 2a) and the previous discussions for determining the number of the acid–base pairs for the experiment, the students find that the mixed ratios examined experimentally are not necessarily the correct ratios of the reaction stoichiometry and start to discuss a possible method to determine the reaction stoichiometry.

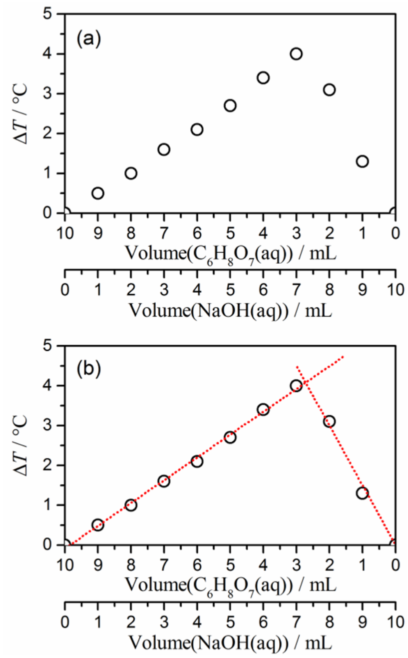

Figure 2. Typical experimental result using Job's method: (a) data points and (b) data analysis.

Through discussion in the groups, they make an important observation on each side in the graph: there are two linear relationships between the mixed ratio and the temperature change. Considering this observation and the preliminary expectation of the maximum temperature change at the mixed ratio that corresponds to the reaction stoichiometry, students propose the intersection point of the two linear regression lines drawn on each side of the graph (Figure 2b). All student groups determine the mixed ratio of 1:3 for $C_6H_8O_7$(aq) and NaOH(aq) to give the maximum temperature change without exception. Some student groups also report that the ratio of the slopes drawn on both sides is 1:3. From the experimental result, the chemical equation that the students develop for the neutralization reaction of $C_6H_8O_7$(aq) and NaOH(aq) is as follows.

$$C_6H_8O_7(aq) + 3NaOH(aq) \rightarrow 3H_2O(l) + Na_3C_6H_5O_7(aq) \quad (1)$$

On receiving the students' result, the instructor presents the molecular structure of citric acid to the students and asks for a possible explanation of the reaction stoichiometry of the neutralization reaction of $C_6H_8O_7$(aq) and NaOH(aq) determined experimentally. Students find three carboxyl groups in $C_6H_8O_7$, and they identify the groups as the reaction sites for the neutralization.

■ HAZARDS

Students are required to wear safety glasses and gloves when handling $C_6H_8O_7$(aq) and NaOH(aq). These solutions are hazardous in case of skin contact, eye contact, or ingestion. The waste solutions should be disposed of after neutralization. If all

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of the solutions used by each student group are mixed, the pH of the mixed solution is in the 5−6 range, and then it can be disposed of safely down the drain.

POSTLAB LEARNING EXERCISE

Reinforcement of the Previous Learning

For the experimental results obtained by the students, two questions are asked to reinforce the previous learning on thermochemistry. The first concerns the calculation of the neutralization enthalpy, Δ*H*, from the expected maximum temperature change. For the calculation, the calibration constant, *K*, for the simplified calorimeter used in the student laboratory (*K* is for compensating the overall loss of the reaction heat during measurement and approximately 1.2−1.4 in the present type of calorimetric system as described in the Supporting Information) is given by the instructor. Students are required to calculate the neutralization enthalpy, which is based on the enthalpy change during the formation 1 mol of $H_2O(l)$ by the reaction between $H^+(aq)$ and $OH^-(aq)$, by formalizing the equation

$$\Delta H\ (\mathrm{kJ/mol}) = -\frac{m\ (\mathrm{g}) \times \frac{C_p\ (\mathrm{J/(K \cdot g)})}{1000} \times \Delta T\ (\mathrm{K})}{\frac{V_{\mathrm{NaOH}}\ (\mathrm{mL})}{1000} \times c\ (\mathrm{mol/L})} \times K \tag{2}$$

where *m* is the mass of the resultant solution ($m = 10$ g with an assumption of the specific gravity of the solution to be unity), C_p is the specific heat capacity ($C_p = 4.18$ J·(K·g)$^{-1}$ by approximately using that of water at 291 K), V_{NaOH} is the volume of NaOH(aq), and *c* is the molar concentration of NaOH(aq). Owing to the simplified calorimetric measurement and the determination of the maximum temperature change at the intersection point of two extrapolated lines, a precise determination of Δ*H* is not expected. However, the value of Δ*H* reported by the students is usually −50−60 kJ mol^{-1}, which is acceptable for discussing the origin of the reaction heat as the neutralization.

The students are also requested to theoretically explain the reason for the linear relationship between the temperature change and mixed ratio of the solutions observed on both sides of the graph (Figure 2b). Using eq 2, the temperature change can be explained to be only a function of the volume of one of the reactant solutions.

Introduction to the Subsequent Learning

Based on the graph in Figure 2, the students are also requested to show experimental evidence that the mixed ratio of the solutions at the intersection point of the two extrapolated lines corresponds to the reaction stoichiometry. Through discussion in the student groups and class, the addition of acid−base indicator to the resultant mixed solutions is proposed by the students to detect excess acid or base. The resultant solutions obtained by adding the acid−base indicator (phenolphthalein for this reaction) indicate a clear color change before and after the mixed ratio at the intersection point. From the activity, students are aware of the use of the acid−base indicator for determining the reaction ratio of acid and base. Subsequently, a demonstration of the titration experiment of 0.5 M $C_6H_8O_7(aq)$ by 0.5 M NaOH(aq) using phenolphthalein as the indicator is performed by the instructor, by which the students confirm the correctness of the reaction stoichiometry determined by Job's method. This demonstration also acts as an introduction to the experimental procedure of acid−base titration that is the subsequent laboratory exercise for the students. If laboratory time is available, this experiment can also be subjected as one of the student tasks.

The final question to the students is how to determine the concentration of an acid, when the concentration is unknown. Here, the students are required to transfer their understanding of the reaction stoichiometry of the acid−base reaction for applying to the neutralization titration. After a short discussion, the class agrees on the use of a neutralization titration. The forthcoming laboratory exercise involves the selection of the most appropriate acid−base indicator for the respective neutralization titrations of HCl−NaOH, CH_3COOH−NaOH, and HCl−NH_3 systems using the empirical methods comparing the colorations of the mixed solutions with different ratio of acid and base. After practicing the neutralization titrations for the acid−base reactions with the reaction stoichiometry of 1:1, the determination of the concentration of citric acid in the lemon juice is given as the final subject.

CONCLUSION

In a high school chemistry course in which a systematic learning curriculum is used, the laboratory inquiry for determining the reaction stoichiometry of neutralization via Job's method using thermochemical relationships is a suitable laboratory exercise for students who have studied the learning units covering the structure of materials, chemical reaction stoichiometry, and thermochemistry. The laboratory exercise provides the opportunity to actively use the students' learned chemical concepts and knowledge. The inquiry is based on the scientific methodology proposed by the students themselves, and this promotes their motivation for the laboratory exercise; the accomplishment of the inquiry gave them intense pleasure. At the same time, the laboratory inquiry generates the basis for subsequent learning of acid−base reactions and neutralization titration. The present laboratory inquiry can be an example to integrate different learning units and promote the effectiveness of systematic chemistry learning in a high school chemistry course.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/ed500947t.

Student handout and information for instructor (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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