An Introductory Laboratory Exercise on Solution Preparation: A Rewarding Experience

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Solution preparation is one of the most common tasks of a laboratory worker or researcher. Yet in the introductory laboratory, chemistry students rarely encounter the need to prepare their own solutions. This is unfortunate, since the lecture part of introductory chemistry often places significant emphasis on solution concentration, dilution, and solution stoichiometry. Students do homework exercises and answer exam questions involving molarity (M), percentage-by-weight (%w/w), percentage by weight–volume (%w/v), parts per million (ppm), etc. Their textbooks (*1*) may even include step-by-step graphical illustrations of a solution being prepared in a volumetric flask. However, many of them draw a blank when it comes to actually preparing a solution in the laboratory. This lack of ability to prepare simple solutions has been noted among advanced chemistry students (*2, 3*). Quigley (*2*) cautioned against the presumption that "second- or third-year undergraduate students know how to prepare their own standard solutions" in the analytical chemistry laboratory. Marino (*3*) proposed: "Maybe the 'solution' to this laboratory void is to provide sufficient lab time in general chemistry for students to prepare their own solutions."

The laboratory exercise described here takes another approach to fill this "laboratory void". It *is* a solution preparation exercise, to be completed within a two-hour lab session. Designed to help beginning students connect conceptual understanding to hands-on practice, this exercise has served the purpose successfully for more than 14 years in different types of introductory chemistry courses I have taught. Students, science and non-science majors alike, consistently consider this experiment one of their top-favorite labs. Upon completion of this exercise, most, if not all, of the students are able to write clear, step-by-step procedures for preparing solutions commonly used in the laboratory.

Merits of This Exercise

Several factors contribute to the effectiveness of this lab exercise. First, it links solution preparation to the iodine clock oscillating reaction developed by Briggs and Rauscher (*4*). This reaction displays striking cyclic color changes from colorless to amber to blue using simple reagents. It has been widely recognized as an excellent lecture demonstration (*5, 6*) along with many other demonstrations based on chemical oscillations (*7, 8*) and iodine clock reactions (*9–11*). Several student investigations based on oscillation reactions were reported in this *Journal* (*12–15*). These experiments center around examining the dynamics of a class of chemical oscillators in what is known as the Belousov–Zhabotinskii (BZ) reaction (*16*). All but one of them are physical chemistry labs. The one exceptional exercise (*12*) directed students to compare the periodic behaviors of the BZ reaction and a pendulum in a three-way freshman course integrating general chemistry, general physics, and calculus.

In the introductory exercise described here, students are simply asked to prepare the solutions for the Briggs–Rauscher (BR) oscillation reaction. The lab session begins with a demonstration of the BR reaction. After being awed by the almost magical phenomenon, students welcome the prospect of making it happen again with their own solutions. This motivates them to carefully employ proper laboratory procedures in solution preparation.

Second, this exercise involves a variety of situations commonly encountered in solution preparation. Four solutions are necessary for the BR reaction; each provides certain specific opportunities for hands-on practice. Solution 1 contains two solutes, 0.15 M malonic acid and 0.020 M $MnSO_4$. This solution introduces standard techniques for preparing molar solutions of the solid-in-liquid type. Since manganese sulfate is supplied as $MnSO_4{\cdot}H_2O$, students need to calculate the formula mass for the monohydrate. This alerts them to the fact that solid crystals may not always be anhydrous. Solution 2 is 0.20 M KIO_3 in 0.080 M H_2SO_4. Since potassium iodate dissolves rather slowly at room temperature, heating is necessary to speed up the process. (It is soluble in 12 parts of water and 3.1 parts boiling water [*17*].) This procedure allows students to observe firsthand the effect of temperature on solubility. Solution 3 is 3.6 M H_2O_2, which is prepared from 30% (w/w) H_2O_2. Students learn to convert percentage-by-weight to molarity in this case. Laboratory procedure introduces dilution and volume measurements with a buret. Solution 4 is a 3% (w/v) boiled-starch solution, which involves another concentration unit, percentage by weight–volume.

Third, oscillation phenomenon can occur (at least to a certain extent) even when solution concentrations are not exactly accurate. Though this seems to contradict the intent of this exercise, it affords some latitude in defining what "success" means to the students. As a first attempt in solution preparation, 80–85% beginning students have succeeded in making their solutions "work" in this exercise. This "success rate" appears to be just right for the exercise to be challenging but not overly so. Upon meeting the challenge, students gain confidence and derive quite a sense of satisfaction. The exercise has even served as a vehicle for public outreach. Some students asked to take their solutions home for "show-and-tell" to family members, roommates, etc. Others designed science fair projects and, in one case, a Sunday school demonstration, using the "recipe" from this exercise.

Lastly, this exercise lends itself to further take-home studies or class discussions suitable for different types of introductory chemistry courses. For example, in a general chemistry course for science and engineering majors, students may identify, complete, and balance some of the redox reactions involved in the oscillation reaction. In a course for nursing and allied health professionals, students can write a short essay comparing and contrasting chemical oscillations with some periodic phenomena found in living organisms, such as heartbeat and the menstrual cycle.

Description of the Exercise

This lab is best scheduled shortly after lecture discussions on solution concentration and dilution. Students are instructed to prepare three solutions:

1. 50 mL 0.15 M malonic acid ($CH_2(COOH)_2$) and 0.020 M manganese sulfate ($MnSO_4$)
2. 50 mL 0.20 M potassium iodate (KIO_3) in 0.080 M sulfuric acid (H_2SO_4)
3. 25 mL 3.6 M hydrogen peroxide (H_2O_2)

For each solution, students need to first show calculations to find out how much solute is needed. (They are to complete similar calculations for the fourth solution, 3% [w/v] starch, though the solution is provided in dropper bottles for their use.) Then, they follow laboratory procedures to prepare the actual solutions. When all solutions are ready, they measure equal amounts (10 mL) of the three solutions and pour them simultaneously into a beaker with several drops of the starch solution. If prepared correctly, the mixture will display characteristic cyclic color changes all on its own for about ten minutes. A lack of the oscillating phenomenon indicates one or more of the solutions may have been prepared incorrectly. If time allows, students can examine the oscillating reaction more carefully. They may record time intervals between color changes, note changes to the pattern if one or more solutions are added, check how temperature affects the reaction in terms of oscillating frequency, evolution of a gas, and formation of a solid, etc.

The following materials and equipment are provided in the laboratory: reagent grade $CH_2(COOH)_2$(s), $MnSO_4 \cdot H_2O$(s), KIO_3(s), 0.080 M H_2SO_4(aq), 3% (w/v) boiled-starch indicator solution, 25- and 50-mL volumetric flasks, 250-mL beaker, 10-mL graduated cylinders, metal spatula, stirring rod, timer, hot-plate, balance, and thermometer. An additional solution used in this exercise, 30% (w/w) H_2O_2(aq), requires special handling and storage attention (*7, 17*). It is a very strong oxidizing agent. To minimize handling hazards, I have always delivered desired aliquots of the 30% solution (9.18 mL needed to prepare 25 mL of 3.6 M solution) from a buret directly into a student's volumetric flask upon request.

For those instructors who wish to avoid using 30% H_2O_2 in student labs altogether, the Instructor Notes section of the lab write-up describes two alternative ways to replace it in this exercise.[W] The first is to replace 30% H_2O_2 with 15% H_2O_2. Since 15% H_2O_2 is not widely used as is the 30% solution, it is not available for direct purchase from most chemical suppliers. Its density may differ significantly from the 1.11 g/mL given for the 30% solution (*17*). The instructor may need to prepare the 15% solution by diluting the 30% and perform an accurate measurement of its density. This should be done shortly before the lab period to avoid deterioration of the solution. The second alternative is taken from Shakhashiri (*7*). It eliminates the dilution procedure by using 3% H_2O_2 directly as solution 3. Concentrations of the other solutes are changed slightly as follows: solution 1 = 0.20 M malonic acid and 0.026 M manganese sulfate, solution 2 = 0.27 M potassium iodate and 0.10 M sulfuric acid. Instead of mixing equal volumes of solutions 1, 2, and 3 to generate the BR reaction, this procedure calls for equal volumes of new solutions 1 and 2 and a double volume of the new solution 3, 3% H_2O_2. Disadvantages of using the 3% solution (*7*) include a longer waiting period before oscillations begin (one minute versus instantaneous), less spectacular color changes, and students' missing the opportunity to do dilution in this solution preparation exercise.

The student handout for this exercise was recently rewritten to match other learning activity packets (LAPs) in my *Hands-On Chemistry* series (*18*). The LAPs employ a format with text and questions interspersed among laboratory procedures to promote active learning in laboratory settings. A description of the LAP approach, a sample LAP on *Atoms and Molecules*, and a list of other available LAPs are available on the Internet at the Web site *http://www.scc.spokane.cc.wa.us/RWang/*.

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[W]Supplemental Material

Supplemental material for this article is available in this issue of *JCE Online*.

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