INQUIRY-BASED LABORATORY WORK IN CHEMISTRY

TEACHER’S GUIDE

Derek Cheung
Department of Curriculum and Instruction
The Chinese University of Hong Kong
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Preface

Inquiry-based laboratory work can improve student learning of chemistry. It requires students to write their own procedures and decide how data are presented and analyzed. This guidebook is for secondary school chemistry teachers. It covers 10 examples of inquiry-based laboratory exercises developed and tested at the Chinese University of Hong Kong. They are suitable for Secondary 4-7 students (aged 14-17 years) and were piloted in several schools. Both teachers and students have found that the 10 laboratory exercises can make our school chemistry interesting, challenging, and engaging.

I would like to thank the Quality Education Fund for financial support of this project. I am grateful to Grace Lui, Christine Yu and Ken Lai for their assistance in developing the laboratory exercises. Many thanks are also due to the following chemistry teachers who willingly involved themselves in my project:

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As we developed and piloted the instructional materials, we learned a great deal of the characteristics of inquiry-based laboratory work, but we are very well aware that what we have learned is only the beginning of the journey. We invite any comments, questions and suggestions you may have. Please send them to: spcheung@cuhk.edu.hk.

Derek Cheung
June, 2006
Teacher’s Concerns about Inquiry-based Laboratory Work

Inquiry-based laboratory work is becoming increasingly important in chemistry teaching and learning. In Hong Kong, the Curriculum Development Council (2002) gave teachers the following note:

Teachers should avoid giving manuals or worksheets for experiments with ready-made data tables and detailed procedures, for this kind of instructional materials provide fewer opportunities for students to learn and appreciate the process of science. With an inquiry-based approach, students are required to design all or part of the experimental procedures, to decide what data to record, and to analyse and interpret the data. Students will show more curiosity and sense of responsibility for their own experiments leading to significant gains in their basic science process skills. (p. 40)

However, keeping up with new teaching methods can be a frightening experience for teachers, especially if support is inadequate. Research has revealed that teachers have many concerns about implementing inquiry-based laboratory work in school. The following section describes three major concerns and recommends strategies for alleviating them.

Lack of Class Time

This is the top concern of most teachers. Inquiry-based experiments take longer to complete than traditional lab work. Students need extra time to plan their experimental designs, read books, search for information from the Internet, formulate hypotheses, write procedures, modify procedures based on trials, and decide how data are presented and analyzed. In Hong Kong, chemistry teachers are under strong pressure to finish the content-dominated curriculum and to prepare students for high stakes examinations. Therefore, quite naturally, they organize few inquiry-based labs in order to leave time to cover large amounts of content.

It is worth noting that there are at least four levels of scientific inquiry: confirmation, structured inquiry, guided inquiry, and open inquiry. A confirmation inquiry requires students to verify concepts, principles, or laws by following a given procedure. For a structured inquiry, students are required to solve a problem (e.g., to determine the ‘best buy’ from among several brands of commercial vinegar). Although students do not know the answer, they are allowed to follow a given procedure to find it out. In a guided inquiry, the teacher provides a question and students need to design the procedure to resolve it. In an open inquiry, students are also required to design their own procedures, but the laboratory activities are directed by student questions or an open question set by the teacher (e.g., what factors affect reaction rate?).

It is not necessary that all chemistry laboratory work must be open inquiry because different kinds of laboratory work serve different purposes. The key is to have a variety of laboratory activities for students. To reduce class time, I recommend teachers to include two or three guided inquiries in each academic year and they should be embedded within the existing chemistry curriculum. Students may attempt open inquiries in extra-curricular time. Guided inquiry is useful even though a school has the resources to require students to carry out open inquiries because they need to have opportunities to understand scientific investigations conceptually and procedurally before undertaking open inquiries.

Another strategy to reduce classroom time is to design short investigations that can be performed in several teaching periods. This is particularly important if expensive equipment...
(e.g., data logger) is required. Also, technically simple investigations are preferred because complex techniques or sophisticated instruments may overshadow the conceptual objectives of the inquiry-based lab. Lastly, an excellent strategy to increase efficiency is to ask the entire class to share their raw data. This not only reduces laboratory time but also provides students with more data points to plot graphs, to calculate an average, etc.

**Lack of Effective Instructional Materials**

The second concern most frequently mentioned by teachers is the lack of high-quality instructional materials. Although the Education and Manpower Bureau (2003) and textbook publishers have provided several examples of inquiry-based lab, teachers generally find them ineffective because they are not related to students’ lives. Nor are they relevant to students’ personal needs and curiosity or future profession. They are also not challenging because students can easily find the solutions from textbooks. Thus, our challenge is to increase the degree of authenticity of the investigative problems so that students can exercise curiosity and engage in solving real-world problems that do not have a pre-determined solution. Regrettably, well-contextualized inquiry-based chemistry labs are scarce.

Formulating the problem is a very difficult part of developing a high-quality chemistry investigation. Deters (2004) suggested that ‘The fastest way to begin using inquiry is to take a fairly straightforward lab and simply delete the procedure, data recording, and analysis sections’ (p. 42). But this strategy does not work because most of the experiments prepared by publishers do not have a real-world context.

The 10 investigative problems included in this guidebook are not traditional-style chemistry experiments where students can just follow a given procedure to collect data and then complete worksheets. Instead, they provide Secondary 4-7 students genuine practice in scientific inquiry. The tasks are authentic because they are similar to the activities of scientists. Students are required to write experimental procedures and decide how data are analyzed. These experiences enable them to take an active role in their learning process. They are not only ‘hands-on’, but also ‘minds-on.’ The results of trials done in Hong Kong indicated that the 10 inquiry-based labs can enhance students’ motivation to learn chemistry. Common misconceptions held by students are also identified for each guided inquiry.

Furthermore, effective chemistry teaching requires instructional materials tailored to the abilities and interests of various types of students. Two of the 10 exemplars use lettuce as a source of catalyst (see pages 50-72), but their levels of difficulty are different. Several exemplars focus on acid-base chemistry and chemical kinetics; teachers may select one that is interesting to their students.

**Large Class Size**

The third barrier Hong Kong teachers must overcome is large classes. Inquiry-based laboratory work, seen as something very positive in the chemistry curriculum, is hard to be implemented in a whole-class setting. Investigative labs encourage multiple approaches to a common problem and thus different students can follow different experimental procedures. Ideally, in a scientific inquiry, each student in a class should have considerable autonomy to follow his or her procedure and learn at his or her own pace. Unfortunately, there are usually 40 students in a Secondary 4-5 class in Hong Kong. Even for Secondary 6-7 courses, a chemistry class can be crowded with as many as 35 students. Teachers are afraid that inquiry-based laboratory work can become a logistical nightmare and a source of chemical
hazards because they do not have control over exactly what their students do.

One strategy is to divide the class into groups of 3-4 students when they plan a guided inquiry. I suggest that an oral presentation session be included after students have submitted their plans but before they are allowed to perform the laboratory work (see Appendix A for details). To save class time, only two or three groups will do presentation. During the oral presentation, the teacher encourages students to find out limitations of an experimental design but does not offer the so-called ‘model’ answers directly. The teacher aims to facilitate the groups to finalize a feasible procedure through a consensus approach. It is important to note that the final version of the experimental procedure is derived from student ideas. Even though all groups are allowed to follow the same procedure after the oral presentation session, students have to write up the procedure by themselves to develop scientific communication skills and gain a deep understanding of the concepts involved. Furthermore, no data tables, mathematical equations, or graphs with labeled axes will be distributed as student handouts; students need to record and analyze the raw data by themselves.

Our trials indicated that the consensus approach has a number of advantages. First, the teacher does not lose control over what students do in a large class. Because the entire class will follow the same version of procedure for a given lab, only a limited amount of specific equipment and chemicals is required. Thus, technical support from lab technicians can be planned in advance and the lab becomes less chaotic. Second, safety issues are critically important when students perform investigative laboratory work. It is obvious that teaching large classes makes safety issues get worse. Of course, the teacher must check the plans submitted by students carefully, but there might be hidden, unsafe steps in a procedure. The consensus approach will result in an experimental procedure with less safety issues because the teacher or the lab technician can try it in a pre-lab. Finally, the teacher can manage the lab time easily as the time taken for all students in a large class to finish their practical work can be estimated.

In addition, I recommend teachers to distribute assessment criteria to students in advance. There are at least two advantages as far as large class size is concerned. First, students know the teacher’s expectations and thus they tend to ask less trivial questions about a scientific inquiry. This will reduce management problems. Second, the teacher can grade students’ plans or lab reports efficiently because he or she has already decided what performance must be assessed. Samples of assessment criteria are shown in Appendices B and C. Teachers are welcome to modify the assessment criteria to meet their needs. It is best to start with a simple checklist so that students can develop various process skills gradually.

During the lab work, the teacher may assess students’ performance by direct observation. Assessment criteria may include: the procedures are followed; safety guidelines are followed; proper manipulative skills for specific techniques (e.g., titration); good share of the group work; take measurements accurately; record observations and measurements in a clearly labeled table; repeat the experiment if results are strange; and proper cleanup of lab. A sample of student questionnaire (Appendix D) is also available for teachers to assess whether their students value inquiry-based laboratory work as a component of the chemistry curriculum.

References

Curriculum Development Council (2002). Chemistry curriculum guide (Secondary 4-5). Hong Kong: Printing Department.


How Much Sodium Bicarbonate Is in One Effervescent Tablet?

Redoxon® is the brand name of the first artificially synthesized vitamin C to be sold to the public. Each effervescent Redoxon® tablet contains sodium bicarbonate and other chemicals. The tablet releases carbon dioxide gas when it is placed in water.

The equation for the reaction between sodium bicarbonate and vitamin C (ascorbic acid) is shown below:

\[ 2 \text{NaHCO}_3(\text{aq}) + \text{H}_2\text{C}_6\text{H}_6\text{O}_6(\text{aq}) \rightarrow \text{Na}_2\text{C}_6\text{H}_6\text{O}_6(\text{aq}) + 2 \text{H}_2\text{O}(\text{l}) + 2 \text{CO}_2(\text{g}) \]

The bubbles provide the characteristic mouthfeel and flavour effects when the vitamin C solution is consumed. But too much or too little bubbles will change the flavour of the product. Unfortunately, the amount of sodium bicarbonate present in a Redoxon tablet has not been revealed by the manufacturer.

Imagine that you work as a chemist in a vitamin C factory. Your boss wants you to do a little industrial spying to find out the amount of sodium bicarbonate used by Redoxon®. You job is to design and carry out an investigation to determine the percent by mass of sodium bicarbonate in Redoxon® tablets. You may assume that the tablets do not contain other chemicals which can produce carbon dioxide. Submit your plan by ___________ (date).

On ___________ (date), representatives of your factory will give you an opportunity to share your plan. You will have 10 min to present your plan, followed by 10 min in which you will be expected to respond to queries. Your presentation needs to answer the following questions:

- What are your experimental procedures?
- What data do you need?
- How do you know your procedures can give accurate results?
- Will the proposed procedures be feasible and safe?
- How can you calculate the percent by mass of sodium bicarbonate in Redoxon® tablets?

NOTE: After reviewing your experimental design, the teacher will discuss any safety precautions that are specific to your design. Obtain teacher approval before beginning any lab work.
### Assessment Criteria for Planning the Redoxon® Investigation

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. The procedures for investigating Redoxon tablets are simple and accurate.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2. A proper chemical method is included to check the accuracy of results.</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>3. Calculations of % bicarbonate are correct.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4. Suitable choice of chemicals and apparatus.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5. Chemicals and apparatus are easily available.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6. Measurement errors are minimized by appropriate procedures or apparatus</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7. Steps are included to reduce risks.</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>8. No invalid assumptions.</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>9. The procedures are clear enough to be followed by other students.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10. Labelled drawings are used to help present the procedures.</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>11. Lab trials and repeats are stated.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12. Controls on variables are clearly stated.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>13. Chemicals that need accurate measurement are identified.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>14. Limitations of the experimental design are described.</td>
<td></td>
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</tr>
</tbody>
</table>

**TOTAL:**
Curriculum Links

- Acid-base chemistry
- Effervescent tablets
- Molar volume
- Stoichiometry

Background Information

1. Effervescent tablets release carbon dioxide when they are placed in water. The resulting carbonated solution masks the undesirable taste of any medicinal chemical (Allen, Popovich & Ansel, 2005). The evolution of carbon dioxide gas also agitates gastric juices so that the rate of absorption of drugs such as aspirin into the bloodstream is faster. The mass of bicarbonate or carbonate per tablet is calculated carefully by pharmacists. One of the reasons is to prevent violent and uncontrollable effervescence that could overflow the glass.

2. This guided inquiry is suitable for S4-5 students and was adapted from an experiment described by Peck, Irgolic and O’Connor (1980). In their original experiment, students were asked to determine the percent of bicarbonate by mass in an Alka-Seltzer® tablet. However, Hong Kong students can easily find the mass of sodium bicarbonate from the package. We recommend Redoxon® (double action vitamin C + zinc effervescent tablets) as a source of carbon dioxide because there is no information about the mass of bicarbonate from the package sold in Hong Kong. Student interest in chemistry is enhanced if the laboratory work aims to find out an unknown.

3. From the Internet, we found that the ingredients of Redoxon® effervescent tablets (double action vitamin C + zinc) include ascorbic acid, citric acid, sodium bicarbonate, sodium carbonate, sorbitol, colour (beta-carotene), zinc citrate trihydrate, sodium chloride, sweeteners (aspartame, acesulfame K) and flavouring. Both bicarbonate and carbonate can react with acids to produce carbon dioxide. Designing procedures for determining the percentages by mass of bicarbonate and carbonate in a Redoxon® tablet is too challenging for S4-5 students. That is why in this guided inquiry students are asked to assume that the tablets do not contain other chemicals which can produce carbon dioxide.

4. Since the mass of sodium bicarbonate in a Redoxon® tablet (double action vitamin C + zinc) has not been revealed by the manufacturer, there is no way to check the accuracy of students’ experimental results. The challenge of this guided inquiry is that students have to include a chemical method to estimate the accuracy of their results. Go over students’ plans before starting the oral presentation session. Let students have ample time to carry out calculations and repeat experiments to get the best accuracy possible. Encourage them to consider what changes they could make to improve the accuracy. They may need to modify their procedures, the amounts of chemicals, or the type of solvent in order to improve the results. Furthermore, students will learn from this guided inquiry that even though they repeat a procedure with care, the results are often not reproducible. As Dudek (1991) pointed out, the deviation has pedagogical value. Reliability is a very fundamental procedural concept for students to learn.

5. Students generally propose to use eudiometry (Dudek, 1991; Kildahl & Varco-Shea,
They dissolve an effervescent tablet in water with subsequent measurement of the carbon dioxide gas produced. Based on the volume of carbon dioxide gas, they calculate the amount of sodium bicarbonate contained in the original tablet. There are three common methods to collect and measure the volume of carbon dioxide: (a) displacing water held in a measuring cylinder inverted in a trough of water; (b) syringe; and (c) eudiometer (see page 12).

Table 1. Comparing the percent error of various methods

<table>
<thead>
<tr>
<th>Method</th>
<th>% error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water displacement using HCl as reactant</td>
<td></td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.1 M HCl (50 cm³)</td>
<td></td>
</tr>
<tr>
<td>Tap water in the trough</td>
<td>11 – 13</td>
</tr>
<tr>
<td>NaCl solution in the trough</td>
<td>8 – 12</td>
</tr>
<tr>
<td>Water displacement using citric acid as reactant</td>
<td></td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + citric acid(s) + distilled water (50 cm³)</td>
<td></td>
</tr>
<tr>
<td>Tap water in the trough with dissolved CO₂ from one tablet</td>
<td>12 – 15</td>
</tr>
<tr>
<td>Glass syringe using HCl as reactant</td>
<td></td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 1.0 M HCl (15 cm³)</td>
<td>4 – 6</td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.5 M HCl (15 cm³)</td>
<td>4 – 6</td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.5 M HCl (25 cm³)</td>
<td>6 – 7</td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.1 M HCl (45 cm³)</td>
<td>13 – 14</td>
</tr>
<tr>
<td>Glass syringe using citric acid as reactant</td>
<td></td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + citric acid(s) + distilled water (15 cm³)</td>
<td>6 – 7</td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + citric acid(s) + NaCl(aq) (30 cm³)</td>
<td>10 – 11</td>
</tr>
<tr>
<td>Eudiometer filled with tap water</td>
<td></td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.1 M HCl (20 cm³)</td>
<td>2 – 10</td>
</tr>
<tr>
<td>Eudiometer filled with CO₂-saturated distilled water</td>
<td></td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.5 M HCl (15 cm³)</td>
<td>-2 – 4</td>
</tr>
<tr>
<td>Eudiometer filled with distilled solution</td>
<td></td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.5 M HCl (15 cm³)</td>
<td>1 – 3</td>
</tr>
<tr>
<td>Eudiometer filled with salt solution</td>
<td></td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.5 M HCl (20 cm³)</td>
<td>3 – 4</td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.5 M HCl (15 cm³)</td>
<td>1 – 2</td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.25 M HCl (20 cm³)</td>
<td>4 – 5</td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + 0.25 M HCl (15 cm³)</td>
<td>1 – 3</td>
</tr>
<tr>
<td>Eudiometer filled with salt solution</td>
<td></td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + citric acid(s) + distilled water (15 cm³)</td>
<td>3 – 5</td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + citric acid(s) + NaCl(aq) (25 cm³)</td>
<td>1 – 3</td>
</tr>
<tr>
<td>Reactants: NaHCO₃(s) + citric acid(s) + NaCl(aq) (20 cm³)</td>
<td>1 – 3</td>
</tr>
</tbody>
</table>
We tried out the three methods and tested different experimental conditions. We set up a eudiometer as that described by Peck, Irgolic and O’Connor (1980). We also adopted some of the procedures used by Irgolic, Peck and O’Connor (1977), and Bedenbaugh, Bedenbaugh and Heard (1988). Our trials found that the eudiometer filled with salt water using 15 cm³ of 0.5 M HCl as reactant provided the smallest inaccuracy and smallest deviation (see Table 1), but we had not exhausted all possible experimental conditions. We do not recommend a eudiometer filled with CO₂-saturated distilled water due to three reasons. First, we need to let the solution stand aside for several hours to remove excess carbon dioxide gas; otherwise a lot of tiny bubbles will stick on the walls of the leveling bulb, delivery tube and burette. Second, the water level tends to fluctuate with time even though the chemical reaction is completed. Third, the procedure often results in a negative percent error (see Table 1); extra carbon dioxide gas can come from the solution in the burette. Dudek (1991) also reported that her students usually obtained a negative percent error when they used a saturated solution of carbon dioxide.

6. Under no circumstances should the reaction of Redoxon® with water or acid be performed in a closed container. An explosion could result.

7. Do not buy Redoxon® chewable tablets as they do not give effervescence when they are placed in water.

8. Students should understand the effect of pressure on gaseous volume. The lab pressure may not be exactly one atmosphere. But for S4-5 chemistry, it is not necessary for students to convert the volume of carbon dioxide gas by calculation using Charles’ Law (i.e., V/T = constant) or the ideal gas equation. They may assume that the molar volume of a gas at room temperature and pressure is 24.0 dm³. Also, S4-5 students do not need to take the vapour pressure of water into account.

Students’ Misconceptions and Difficulties

1. Some students have the misconception that the accuracy of their results can only be estimated by repeating the experiment with an effervescent tablet of known amount of bicarbonate or carbonate (e.g., Alka-Seltzer® and Sandoz®). This method will fail because the amount of carbon dioxide released per tablet may not be consistent with the information shown on the package label. For example, according to the manufacturers, the amounts of calcium carbonate in the following two tablets are shown below:

Each tablet of Ca-C 1000 Sandoz (orange flavoured) contains 0.327 g of CaCO₃.
Each tablet of Calcium-D-Redoxon (orange flavoured) contains 0.625 g of CaCO₃.

However, we found that both Ca-C 1000 Sandoz and Calcium-D-Redoxon released much more carbon dioxide gas than the predicted amounts. One possible explanation is that there are other chemicals in these two brands of tablets, which can release carbon dioxide gas. Thus, students must use a pure sample of bicarbonate or carbonate to check the accuracy of their proposed experimental procedures.

2. To check the accuracy of experimental results, some students have the misconception that they must use vitamin C to react with sodium bicarbonate because vitamin C is
present in the Redoxon® tablet. They believe that vitamin C can only be replaced by a diprotic acid. In fact, acids with different numbers of ionizable hydrogen atoms can be used; the molar ratio of NaHCO₃ to CO₂ remains unchanged (i.e., 1:1). The equations for the reactions with hydrochloric acid, vitamin C (ascorbic acid), and citric acid are shown below.

\[
\text{NaHCO}_3 (aq) + \text{HCl}(aq) \rightarrow \text{NaCl} (aq) + \text{H}_2\text{O}(l) + \text{CO}_2(g)
\]

\[
2 \text{NaHCO}_3 (aq) + \text{H}_2\text{C}_6\text{H}_6\text{O}_6(aq) \rightarrow \text{Na}_2\text{C}_6\text{H}_6\text{O}_6(aq) + 2 \text{H}_2\text{O}(l) + 2 \text{CO}_2(g)
\]

\[
3 \text{NaHCO}_3 (aq) + \text{H}_3\text{C}_6\text{H}_5\text{O}_7(aq) \rightarrow \text{Na}_3\text{C}_6\text{H}_5\text{O}_7(aq) + 3 \text{H}_2\text{O}(l) + 3 \text{CO}_2(g)
\]

The net ionic equation for the above three reactions is

\[
\text{HCO}_3^-(aq) + \text{H}^+(aq) \rightarrow \text{H}_2\text{O}(l) + \text{CO}_2(g)
\]

3. Few students can name a chemical test to distinguish between sodium bicarbonate and sodium carbonate. Thermal decomposition of sodium bicarbonate will produce carbon dioxide. But when students heat Redoxon® tablets, yellowish brown fumes will also be formed.

4. Some students determine the mass of carbon dioxide released by comparing the mass of the dry tablet, flask and water to the mass of the dissolved tablet, flask and water. However, many electronic balances with a precision of ±0.001 g do not have the capacity to measure the mass of a flask filled with water. The mass loss experiment will result in large experimental errors because carbon dioxide gas dissolves in water and some water vapour will escape.

5. Many students plan to use a syringe to collect and measure carbon dioxide. When they perform the experiment, they find that the movement of the plunger is affected by friction. Few students think about how their procedures may be modified to reduce the effect of friction (e.g., use hydrochloric acid instead of water to speed up the reaction, set up an eudiometer).

5. Some students plan to use a syringe to collect the carbon dioxide gas released by an
effervescent tablet. Then, they find out the mass of carbon dioxide by weighing the syringe. This method cannot give accurate results due to two main reasons: (a) there is buoyancy of the syringe and plunger when weighed in air; and (b) the reaction flask is filled with air and thus the carbon dioxide collected is impure.

6. A number of students believe that they must collect pure carbon dioxide gas to determine the percent of sodium bicarbonate by mass in a Redoxon® tablet.

7. Some students plan to measure the volume of carbon dioxide by water displacement. They assume that the amount of carbon dioxide dissolved in water is negligible. But carbon dioxide does dissolve in water. At room temperature, 100 cm³ of water can dissolve up to about 80 cm³ of carbon dioxide gas (Peck, Irgolic and O’Connor, 1980).

9. Some students propose to use sodium hydroxide solution to absorb the carbon dioxide released by an effervescent tablet and then perform a back titration with hydrochloric acid to find out any excess sodium hydroxide. But sodium hydroxide reacts with carbon dioxide to form sodium carbonate. Therefore, the resulting solution is a mixture of carbonate ions and hydroxide ions.

\[
2\text{NaOH(aq)} + \text{CO}_2(g) \rightarrow \text{Na}_2\text{CO}_3(aq) + \text{H}_2\text{O(l)}
\]

During the back titration, hydrochloric acid will react with both carbonate ions and hydroxide ions. The composition of the hydroxide-carbonate mixture can be found by the double indicators method (Skoog, West, Holler & Crouch, 2000). Alternatively, barium chloride solution may be added to the mixture to selectively precipitate the carbonate ions before the back titration is done (Wink, Gislason & Kuehn, 2005). But these two methods are beyond S4-5 chemistry students.
**Sample Procedure**

**IMPORTANT:** This sample procedure is for teacher information only. It should *not* be given to students as a cookbook-style experiment. Risk assessments should be done in advance by the teacher. Obtain MSDS information on all hazardous chemicals involved. Label chemicals with the appropriate safety hazard warning labels.

**Materials (per group)**

- Safety goggles (1 pair per student)
- Gloves (1 pair per student)
- Apron (1 per student)
- Sodium bicarbonate (analytical grade)
- Redoxon® effervescent tablets (double action vitamin C + zinc)
- 0.5 M hydrochloric acid
- 25-cm$^3$ measuring cylinder
- Small weighing bottle (glue a magnetic clip at the bottom)
- Magnet bar
- Mortar and pestle
- Aluminium foil or plastic food wrap
- Forceps
- Access to a balance (±0.001 g) and a desiccator

To set up a eudiometer, you need the following:

- 50-cm$^3$ burette
- 250-cm$^3$ conical flask
- 250-cm$^3$ separating funnel to work as a leveling bulb
- 600-cm$^3$ beaker
- Distilled or deionized water
- Table salt (from supermarket)
- Glass rod to stir salt solution
- Funnel
- Transparent delivery tube (about 100 cm long, to connect the burette with the leveling bulb)
- Delivery tube (with two stoppers, about 100 cm long, to connect the burette with the conical flask)
- Clamp and stand (2)
Experimental Details

(A) Construction of a eudiometer

1. Set up the apparatus as shown below. Be sure all connections are air-tight. If you are not familiar with the proper techniques for using clamps and stands, consult your teacher before setting up the eudiometer.
2. Add about 300 cm$^3$ of distilled or deionized water to a 600-cm$^3$ beaker. Dissolve about 100 g of table salt. The salt solution is nearly saturated. Stir the solution with a glass rod. Be sure no bubbles stick on the wall of the beaker.

3. Remove the stopper from the top of the burette. Pour the salt solution into the leveling bulb and the burette. Alternately raise and lower the leveling bulb until all the air has been expelled from the lower delivery tube and no further bubbles rise in the burette.

4. Then, connect the upper delivery tube to the top of the burette. The eudiometer must be air-tight. Check for any leaks by raising the leveling bulb. The salt solution in the burette should not rise continuously. If it does, there is gas leakage.

5. Finally, remove both stoppers from the top of the burette and the conical flask. Adjust the level of water in the burette to about 0.3 cm above the zero mark by raising or lowering the leveling bulb. The eudiometer is now ready for use.

(B) Estimation of percent error in accuracy

1. Using a dry, small weighing bottle, weigh 0.160 g of sodium bicarbonate powder. Record the exact mass.

2. Using a 25-cm$^3$ measuring cylinder, add 15.0 cm$^3$ of 0.5 M of HCl into the conical flask connected to the eudiometer.

3. With the aid of a pair of forceps, carefully put the weighing bottle into the conical flask.

4. Stopper the reaction flask. Then stopper the top of the burette. The water level in the burette should drop slightly due to the volume of the stopper.

5. Adjust the leveling bulb until its water level matches that of the burette. Record the initial water level in the burette.
6. Tilt the weighing bottle with a magnet to start the chemical reaction. Shake the reaction flask gently and continuously to mix the reactants.

7. Observe the water level in the burette. Stop shaking when the movement of the water level comes to a halt. Lower the leveling bulb to reduce the pressure in the burette and then shake the reaction flask again.

8. Stop shaking the reaction flask when the water level in the burette becomes stationery. Wait for about 1 min. The reaction is complete if no gas bubbles are formed in the reaction mixture.

9. Equilibrate the pressure on the eudiometer by adjusting the leveling bulb until its water level matches that of the burette. Record the final water level in the burette.

10. Remove the stopper from the top of the burette. Raise the leveling bulb to bring the water level to about 0.3 cm above the zero mark again. Empty and clean the conical flask. Repeat the experiment to obtain consistent data.

(C) Determination of the % by mass of NaHCO$_3$ in Redoxon® tablets

NOTE: Redoxon® tablets absorb atmospheric moisture easily. Do not handle them with your bare hands. Keep unused tablets in a desiccator.

1. Take half of a Redoxon® effervescent tablet (double action vitamin C + zinc). Quickly wrap the remainder of the tablet using aluminium foil or plastic food wrap to prevent contact with atmospheric moisture.

2. Get a dry and clean mortar and pestle. Crush the half-tablet into fine powder.

3. Using a dry weighing bottle, weigh 0.730 – 0.740 g of the powder. Record the exact mass.

4. Using a 25-cm$^3$ measuring cylinder, add 15.0 cm$^3$ of 0.50 M HCl into the conical flask connected to the eudiometer.

5. With the aid of a pair of forceps, carefully put the weighing bottle into the conical flask.

6. Stopper the reaction flask. Then stopper the top of the burette. The water level in the burette should drop slightly due to the volume of the stopper.

7. Adjust the leveling bulb until its water level matches that of the burette. Record the initial water level in the burette.
8. Tilt the weighing bottle with a magnet to start the chemical reaction. Shake the reaction flask gently and continuously to mix the reactants.

9. Observe the water level in the burette. Stop shaking when the movement of the water level comes to a halt. Lower the leveling bulb to reduce the pressure in the burette and then shake the reaction flask again.

10. Stop shaking the reaction flask when the water level in the burette becomes stationery. Wait for about 1 min. The reaction is complete if no gas bubbles are formed in the reaction mixture.

11. Equilibrate the pressure on the eudiometer by adjusting the leveling bulb until its water level matches that of the burette. Record the final water level in the burette.

12. Remove the stopper from the top of the burette. Raise the leveling bulb to bring the water level to about 0.3 cm above the zero mark again. Empty and clean the conical flask. Repeat the experiment to obtain consistent data.

NOTES:

1. Avoid using tap water to prepare the sodium chloride solution. If tap water is used, students should leave it overnight to degas.

2. Do not use saturated salt solution to fill the eudiometer; crystals of salt may form, which can block the flow of solution in the burette.

3. Students should calculate the mass of pure sodium bicarbonate and Redoxon powder to produce a volume of carbon dioxide (46-48 cm$^3$) that can utilize most of the capacity of the burette. This is one of the ways to increase of the reliability of data.

4. To minimize risks, avoid using hydrochloric acid with concentration greater than 0.5 M.

5. The volume of carbon dioxide is sensitive to change in temperature. Hold the neck of the conical flask when you shake it to mix the reactants.
Sample Data and Results

Six runs were conducted to estimate the accuracy of results obtained by the eudiometer. The results are shown below:

Lab temperature = 20.0 °C
Volume of 0.50 M HCl used = 15.0 cm$^3$
Molar mass of NaHCO$_3$ = 23.0 + 1.0 + 12.0 + 16.0 x 3 = 84.0 g
Assume that the molar volume of gases = 24.0 dm$^3$
For run #1, the predicted volume of CO$_2$ is $\frac{0.165}{84.0} \times 24000 = 47.1 cm^3$
The % error is $\frac{47.1 - 46.45}{47.1} \times 100% = 1.4%$

<table>
<thead>
<tr>
<th>Run</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of NaHCO$_3$ used (g)</td>
<td>0.165</td>
<td>0.166</td>
<td>0.166</td>
<td>0.167</td>
<td>0.167</td>
<td>0.167</td>
</tr>
<tr>
<td>Final burette reading (cm$^3$)</td>
<td>46.45</td>
<td>46.65</td>
<td>46.45</td>
<td>46.95</td>
<td>46.95</td>
<td>46.95</td>
</tr>
<tr>
<td>Initial burette reading (cm$^3$)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.10</td>
<td>0.05</td>
<td>0.10</td>
<td>0.05</td>
</tr>
<tr>
<td>Measured volume of CO$_2$ (cm$^3$)</td>
<td>46.45</td>
<td>46.65</td>
<td>46.35</td>
<td>46.90</td>
<td>46.85</td>
<td>46.90</td>
</tr>
<tr>
<td>Predicted volume of CO$_2$ (cm$^3$)</td>
<td>47.1</td>
<td>47.4</td>
<td>47.4</td>
<td>47.7</td>
<td>47.7</td>
<td>47.7</td>
</tr>
<tr>
<td>Error in accuracy (%)</td>
<td>1.4</td>
<td>1.6</td>
<td>2.3</td>
<td>1.7</td>
<td>1.8</td>
<td>1.7</td>
</tr>
</tbody>
</table>

The average % error is $\frac{(1.4 + 1.6 + 2.3 + 1.7 + 1.8 + 1.7)}{6} = 1.8%$

Three runs were carried out to determine the percent by mass of NaHCO$_3$ in Redoxon® tablets (double action vitamin C + zinc). The results are displayed in Table 3.

Table 3. The Redoxon experiments

<table>
<thead>
<tr>
<th>Run</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Redoxon powder used (g)</td>
<td>0.750</td>
<td>0.750</td>
<td>0.735</td>
</tr>
<tr>
<td>Final burette reading (cm$^3$)</td>
<td>46.80</td>
<td>47.75</td>
<td>47.95</td>
</tr>
<tr>
<td>Initial burette reading (cm$^3$)</td>
<td>0.00</td>
<td>0.05</td>
<td>0.10</td>
</tr>
<tr>
<td>Measured volume of CO$_2$ (cm$^3$)</td>
<td>46.80</td>
<td>47.70</td>
<td>47.85</td>
</tr>
<tr>
<td>Mass of NaHCO$_3$ based on the measured volume of CO$_2$ (g)</td>
<td>0.164</td>
<td>0.167</td>
<td>0.167</td>
</tr>
<tr>
<td>Mass of NaHCO$_3$ (%)</td>
<td>21.9</td>
<td>22.3</td>
<td>22.7</td>
</tr>
</tbody>
</table>

For run #1, the volume of carbon dioxide collected = 46.80 cm$^3$
Based on the volume of carbon dioxide, the mass of NaHCO$_3$ = $\frac{46.80}{24000} \times 84.0 = 0.164 g$
The % by mass of NaHCO$_3$ in Redoxon® tablets = $\frac{0.164}{0.750} \times 100\% = 21.9\%$

The % by mass increased from run 1 to run 3, indicating that some carbon dioxide gas may have dissolved in the salt solution in the eudiometer.

The average % by mass of NaHCO$_3$ = \( \frac{21.9 + 22.3 + 22.7}{3} \) = 22.3%

Possible sources of error include:

1. We assumed no leakage of gases during the experiments.
2. We assumed no loss of carbon dioxide gas due to solubility in the hydrochloric acid and salt solution.
3. There were human errors when we matched the water levels.
4. We used half of a tablet for experiment, but sodium bicarbonate may not have been evenly distributed in a Redoxon® tablet.
5. Although Redoxon® tablets were kept in the original container, they may have absorbed moisture.

References


What is the Rate of a Lightstick Reaction?

Did you ever play with lightsticks on Middle Autumn Festival night or Halloween night? The lightstick consists of a thin glass ampule inside a plastic tube. The ampule contains hydrogen peroxide solution. The plastic tube is filled with a solution of an ester and a dye. These two solutions are kept separate by the glass ampule. The lightstick can be activated by bending the plastic tube to break the ampule. This allows the hydrogen peroxide and the ester to react, producing intermediates. The intermediates then react with the dye to form high-energy molecules which emit light when they reform stable molecules. Thus, the lightstick is an excellent example of chemical energy converted into light energy. Reactions that produce light without heat are called chemiluminescent reactions.

If you increase the rate of the chemiluminescent reaction, the lightstick will give off more light. Can you plan and carry out an investigation to test the following two hypotheses? Submit the plan as group work by ________________ (date).

(1) The chemiluminescent reaction in a lightstick becomes slower and slower as time proceeds.

(2) The rate of chemiluminescent reaction in a lightstick increases about 2 fold for every 10 °C rise in temperature.

Your group will be presenting on ________________ (date). You will have 10 minutes to present your plan, followed by 10 minutes in which you will be expected to respond to queries. Your presentation needs to answer the following questions:

- How can you measure the rate of reaction in a lightstick?
- Is a fair test essential in this investigation? Why?
- Will your proposed procedure be feasible and safe?

NOTE: After reviewing your experimental design, the teacher will discuss any safety precautions that are specific to your design. Obtain teacher approval before beginning any lab work.
Assessment Criteria for Planning the Lightstick Investigation

Names of Students: ____________________________

__________________________________________

__________________________________________

Date: ____________________________

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment</th>
<th>Self</th>
<th>Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Proper procedure and apparatus are proposed to measure the reaction</td>
<td></td>
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<tr>
<td>rate in a lightstick.</td>
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<tr>
<td>2. Clear arguments for or against a fair test are presented.</td>
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<tr>
<td>3. Correct method of data analysis is presented to test the two</td>
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<td></td>
<td></td>
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<tr>
<td>hypotheses.</td>
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<tr>
<td>4. Safety precautions are specified and steps are included to reduce</td>
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<td></td>
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<tr>
<td>risks.</td>
<td></td>
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<tr>
<td>5. Measurement errors are minimized by appropriate procedures or</td>
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<tr>
<td>apparatus.</td>
<td></td>
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<tr>
<td>6. No invalid assumptions are made.</td>
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<tr>
<td>7. The methods are clear enough to be followed by other students.</td>
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<tr>
<td>8. Lab trials are stated.</td>
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<tr>
<td>9. Repeats are stated.</td>
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<tr>
<td>10. Chemistry vocabulary is used correctly.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>11. Labeled drawings are used to help present the procedure.</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>12. Limitations of the experimental design are described.</td>
<td></td>
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</tr>
</tbody>
</table>

TOTAL: ____________________________
Background Information

1. Chemistry textbook writers always define chemical reaction rates in terms of concentration changes with time. They rarely describe measurement of reaction rates using a light sensor.

2. Reactions that produce light without heat are called chemiluminescent reactions. There is a lot of information about the use of luminol from the Internet. But to make this guided inquiry more authentic, S4-5 students are required to investigate a commercial chemiluminescent reaction that occurs inside a lightstick.

3. Although information about the effect of temperature on the brightness of a lightstick is available from the Internet, quantitative data are seldom presented. This inquiry-based experiment provides S4-5 chemistry students an opportunity to investigate the effect of temperature on lightstick reaction quantitatively. It is also a good opportunity for S4-5 students to develop an ability to plot graphs and interpret information from graphs.

4. Different manufacturers may use different chemicals to make lightsticks. The lightsticks of some brands keep the dyes in the glass ampule rather in the plastic tube. The basic mechanism is as follows:

   (i) oxalate ester + hydrogen peroxide $\rightarrow$ intermediate + products

   (ii) intermediate + dye $\rightarrow$ dye* + products

   (iii) dye* $\rightarrow$ dye + $hv$

5. The first substitution of hydrogen peroxide for a phenol in the oxalate ester is rate determining (Hadd et al., 1999). The light intensity at a fixed point from the lightstick is directly proportional to the rate of the chemiluminescent reaction (Bindel, 1996).

   \[ \text{Rate} \propto \text{Light Intensity} \]

6. The colour of light emitted by the excited state of the dye molecules depends upon the type of fluorescent dye used. Three examples are shown below. To measure the change in light intensity accurately, a light sensor must be used. A darkened room is not needed if the measurements are done inside a black cardboard box.

<table>
<thead>
<tr>
<th>Colour</th>
<th>Dye</th>
</tr>
</thead>
<tbody>
<tr>
<td>Green</td>
<td>9,10-bis(phenylethynyl) anthracene</td>
</tr>
<tr>
<td>Orange-yellow</td>
<td>Rubrene</td>
</tr>
<tr>
<td>Blue</td>
<td>9,10-diphenylanthracene</td>
</tr>
</tbody>
</table>
7. The higher the temperature, the faster the reaction, the more intense the chemiluminescence. Students should understand that rate measurements are usually made under conditions such that all of the factors which might affect the rate are held constant, except one, so that the dependence of the rate on each variable is determined separately. To investigate the effect of temperature on the chemiluminescent reaction, the reaction rate should be determined before the initial concentrations of reactants have changed significantly. The initial rate of a reaction is the instantaneous rate determined just after the reaction begins (just after t = 0).

8. Do NOT allow S4-5 students to break the plastic tube and glass ampule and separate the reactants because it is dangerous to break the thin glass ampule. Some dyes are suspected carcinogens (Hadd et al., 1999). For this investigation, it is not necessary to carry out the chemiluminescent reaction in a test tube.

9. To avoid melting the plastic tube of a lightstick, the temperature must not exceed 70 °C (Shakhashiri, 1983).

10. This inquiry-based experiment can be carried out by a pair of students, one of whom starts the reaction by bending the lightstick while the other student turns on a stopwatch.

11. If the teacher wants to save on lightsticks and time, she or he may divide the class into several groups and let each group have one lightstick. Different groups may activate their lightsticks at different temperatures and then share the data.

12. The effect of temperature on reaction rates depends upon the values of activation energy. The reaction rates of a lot of chemical reactions do not increase about 2 fold for every 10 °C rise in temperature.

13. There are two pathways for the production of light in a lightstick: one of short duration, and one of long duration (Estell, 1991). This guided inquiry focuses on the reaction of short duration.

**Students’ Misconceptions and Difficulties**

1. Many students try to test the first hypothesis by plotting light intensity against time and then examining the change in slope along the curve. They believe that the slope or gradient shows the reaction rate.

\[
\text{reaction rate} = \frac{\text{change in light intensity}}{\text{time elapsed}}
\]

Light sensors usually measure light intensity expressed as lux. It is a metric unit that indicates the density of light that falls on a surface. One lux is defined as one lumen per square meter. Lumen is a unit for measuring the rate of flow of electromagnetic energy. Therefore, lux is a unit that indicates the amount of radiant energy per second that falls on a surface.

To test the first hypothesis, all the students have to do is to plot light intensity against time and show that the light intensity decays quickly and then “settles down.” Figure 1 shows an example.
2. To test the second hypothesis, many students do not recognize that changes in light intensity due to changes in concentration of reactants must be avoided. If they perform the experiments at different temperatures, the concentrations of reactants are the same only at the beginning of the reaction. Thus, students must try to compare the initial light intensities at different temperatures.

3. Some students have the misconceptions that they can compare the reaction rates at two different temperatures by measuring the time taken for the light intensity to decrease a certain value (e.g., five lux) or to settle down. They are not aware that the concentrations of reactants will change over the time intervals. Thus, it is not a fair test.

4. Some students try to test the second hypothesis by comparing the decrease in light intensity after a time interval (e.g., 2 min). This method does not work because the value indicates the change of reaction rate with time.

\[
\text{rate} \propto \text{intensity}
\]

\[
\frac{\Delta(\text{intensity})}{\text{time}} = \frac{\Delta(\text{rate})}{\text{time}}
\]

Similarly, calculation of the slope of a line tangent to the intensity-time curve at a particular time is also inappropriate.

5. The procedures proposed by S4-5 students are seldom fast enough to determine the initial light intensity of the chemiluminescent reaction. There are at least two possible ways to partially overcome the difficulty. First, students may try to reduce the time lag to, for example, 10 seconds and assume that the concentrations of reactants will remain unchanged during the short time interval. Second, students may extrapolate the intensity-time curve to estimate the initial intensity at a particular temperature. Note that extrapolation is different from interpolation. Extrapolation is the estimation of a value
beyond a given series of values, while interpolation is the estimation of a value between two values in a table or experimental data on a graph (Iwunze, 2005). However, it is not easy to extrapolate a curve with high accuracy; only an approximate value can be obtained.

6. It is difficult for S4-5 students to prepare a water bath at a specific temperature (e.g., 60 °C). If only two lightsticks are given to each group of students, they may have difficulty in testing the second hypothesis as ΔT may not be exactly 10 °C. One way to overcome the difficulty is to ask the class to share data and then plot a graph of initial light intensity against temperature. The second hypothesis can be tested based on the values estimated by interpolation.

7. It is difficult to keep temperature constant during the reaction. Measurement of change in light intensity should be limited to a very short duration (e.g., 4 minutes) so that the decrease in temperature in the reaction mixture is not significant.
Sample Procedure

IMPORTANT: This sample procedure is for teacher information only. It should not be given to students as a cookbook-style experiment. Risk assessments should be done in advance by the teacher. Obtain MSDS information on all hazardous chemicals involved.

Materials (per group)

Safety goggles (1 pair per student)
Apron or lab coat (1 per student)
Lightstick (2, same brand, colour and size)
1-L beaker
Glass rod to stir the water bath
Stopwatch
Black cardboard box
Thermometer
Bunsen burner, electric heater or hotplate
Access to a light sensor, data logger, computer, and Blu-tack

Experimental Details

1. Turn on the interface and your computer. Plug the light sensor into the computer USB port. This will automatically launch the DataStudio® program. A graph display window should appear.

2. Set the sampling rate at 1 measurement per second.

3. Insert the light sensor into the hole of a black cardboard box. Seal the hole with blu-tack. Select the intensity range for a light bulb. Click the “Start” button to check whether the light sensor can work satisfactorily.
4. Add about 800 cm$^3$ of tap water into a 1-litre beaker. Prepare a 60 °C water bath **CAUTION:** Do NOT exceed 70 °C because the plastic tube of your lightstick may melt. Adjust the heating to keep the temperature of the water steady.

5. Remove a lightstick from its wrapper. Immerse the lightstick in the hot water bath. Wait at least 10 minutes to allow time for the chemicals inside the lightstick to come to the same temperature as the water.
6. **Carry out steps 6 and 7 quickly.** Remove the lightstick from the water bath, and record the exact temperature of the water bath immediately.

7. Dry the outside of the lightstick. Gently bend the lightstick until you hear the glass ampule inside snap. Start the stopwatch immediately. Vigorously and quickly shake the lightstick 10 times to mix its contents. The lightstick should glow. Place the activated lightstick into the black cardboard box and fix it to the box with blu-tack. Cover the box to prevent room light from entering the light sensor. After 10 seconds have passed away, click the “Start” button to begin recording of data.

8. Collect data for about 4 minutes. Click the “Stop” button to end data recording.

9. Click the “Scale to Fit” button. A graph of light intensity against time will appear. Save the file.

10. Remove the lightstick from the cardboard box. It can be thrown away in a rubbish bin.

11. Repeat steps 4 -10 by preparing a water bath at 50 °C. Use a new lightstick of the same brand, size and colour. Fix the lightstick to the same position in the cardboard box.

**NOTES:**

- The above sample procedure uses the DataStudio® program, sensor and interfaces manufactured by Pasco®. The procedure can be easily modified if students use other programs, light sensors, and interfaces. Make sure that the light sensor is set correctly for measuring chemiluminescence.

- The 4-min data collection time is short. So, the investigation can be performed by several groups of students even though the teacher can only provide one or two light sensors.

- To share data, students must use the same brand, colour and size of lightsticks.
Sample Data and Results

Green Cyalume® lightsticks (13 cm long) were used to perform the experiments. Results are shown in Figure 2. The two curves indicate that the light intensity, and therefore the reaction rate, decreased quickly with time and then settled down. Thus, the first hypothesis is consistent with the real data.

Figure 2. Chemiluminescence intensity against time at 50 and 60 °C

However, the second hypothesis is not supported by the data. The rate only increased by 1.3 fold when temperature increased from 50 to 60 °C.

\[ \text{rate } \propto \text{ intensity} \]

\[
\frac{\text{rate}_{60}}{\text{rate}_{50}} = \frac{I_{60}}{I_{50}} = \frac{23 \text{ lux}}{18 \text{ lux}} = 1.3
\]

Possible sources of error include:

1. We are not sure whether the lightsticks contained the same concentrations of chemicals before they were bent.
2. The real initial light intensities were not measured.
3. It was difficult to keep the temperature constant during the experiment.
4. We are not sure whether we shook the lightsticks to the same extent.
5. Data at only two temperatures (i.e., 50 and 60 °C) were collected and analyzed. The second hypothesis might be correct if temperatures below 50 °C were tested.

References


Does Toothpaste Protect Teeth?

We use toothpaste for cleaning our teeth. Which brand of toothpaste do you prefer? Can toothpaste slow down the rate at which acids attack our teeth?

Imagine that you have been hired by the Consumer Council to investigate whether toothpaste can really slow down the rate of reaction between teeth and acids. It is your job to plan and carry out experiments to compare at least three different brands of toothpaste. You may use chicken eggshells to substitute for human teeth. The major ingredient of eggshell is calcium carbonate. Submit your plan by ____________ (date).

Your group will be presenting on ____________ (date) in front of the Consumer Council representatives. You will have 10 minutes to present your plan, followed by 10 minutes in which you will be expected to respond to queries. Your presentation needs to answer the following questions:

- How will you measure the rate of tooth decay for each brand of toothpaste?
- What variables will you need to keep constant in this investigation?
- Will the proposed procedure be feasible and safe?

NOTE: After reviewing your experimental design, the teacher will discuss any safety precautions that are specific to your design. Obtain teacher approval before beginning any lab work.
## Assessment Criteria for Planning the Toothpaste Investigation

**Names of Students:**

________________________

________________________

________________________

**Date:** __________________

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Identify the problem and state it clearly in a way that can be tested.</td>
<td>____</td>
<td>____</td>
<td>____</td>
</tr>
<tr>
<td>2. Use proper apparatus, techniques and safety precautions.</td>
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<td>15. Labeled drawings are used to help present the procedure.</td>
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**TOTAL:** ____ ____ ____
Background Information

1. This guided inquiry is for Secondary 4 students and was adapted from an experiment described by Parkin (1998). In his original design, students need to investigate the effect of fluoride ions on the rate of reaction between eggshells (substitute teeth) and dilute ethanoic acid. They need to soak eggshells in sodium fluoride solution of various concentrations (0.25 M – 1.00 M). The treated eggshells are then placed in a conical flask, along with a small vial containing ethanoic acid. The flask is sealed and is connected to a home-made, S-shape manometer containing a small volume of coloured water to create an air-lock. Finally, the flask is tilted to overturn the vial so that the eggshells can react with dilute ethanoic acid. Students need to record the time taken to produce a given volume of carbon dioxide gas measured by the change in the level of coloured water. However, the degree of authenticity of Parkin’s experiment is low (Cheung, 2005). Also, solid sodium fluoride is toxic and Secondary 4 students should avoid using it to carry out experiments. Furthermore, when we tried out Parkin’s experimental set-up, we found that the use of a manometer to measure the rate of formation of carbon dioxide gas was unreliable; the movement of the coloured water inside the tube was easily affected by factors such as friction and the shape of the tube.

2. Many brands of toothpaste contain fluoride, but its concentration is very low (500 ppm – 1500 ppm). Our toothpaste investigation focuses on the effect of toothpastes on the rate of reaction between eggshell and acid. It is difficult for Secondary 4 students to obtain enough human teeth for investigation. Although teeth may be taken from other
mammals such as pigs, there are hygiene hassles. Therefore, we recommend the use of chicken eggshells as a substitute for teeth. Chicken eggshells contain mainly calcium carbonate and are inexpensive. The results of trials indicated that we can significantly increase the reaction rate by using hydrochloric acid rather than ethanoic acid.

3. In order to compare several brands of toothpaste, students need to keep all-but-one variable constant in their experimental design (i.e., the concept of a fair test). The practical work is interesting and resembles the kind of tasks undertaken by professionals.

4. Bolduc and Bagonda (n.d.), and also Blais and Poirier-Garneau (n.d.) tested human teeth. Their lab reports can be downloaded from the Internet.

5. Hydrochloric acid is used to simulate the environment in which our teeth are when we eat acidic food or after eating some fermentable carbohydrates (Cheung, 2005). Fluoride can prevent dental caries due to three major mechanisms of action (Featherstone, 2000). However, the exact mechanisms are too difficult for Secondary 4 students. The mechanisms may be simplified as follows:

   The main component of tooth enamel is hydroxyapatite, $\text{Ca}_5(\text{PO}_4)_3\text{OH}$. It dissolves to a small extent in our mouth to form ions.

   $$\text{Ca}_5(\text{PO}_4)_3\text{OH}(s) \rightarrow 5 \text{Ca}^{2+}(aq) + 3 \text{PO}_4^{3-}(aq) + \text{OH}^-(aq)$$

   After we have eaten carbohydrates, certain bacteria in our mouth react with sugars and produce acids when they metabolize them. These acids react with hydroxide ions near our teeth. To compensate for the loss of hydroxide ions, more hydroxyapatite dissolves, resulting in cavities (i.e., decaying teeth). When we brush our teeth with fluoride toothpaste, the fluoride ions react with calcium ions and phosphate ions to form fluorapatite, $\text{Ca}_5(\text{PO}_4)_3\text{F}$.

   $$5 \text{Ca}^{2+}(aq) + 3 \text{PO}_4^{3-}(aq) + \text{F}^-(aq) \rightarrow \text{Ca}_5(\text{PO}_4)_3\text{F}(s)$$

   Because fluorapatite is less soluble than hydroxyapatite, our enamel is more acid resistant.


7. The assessment criteria for assessing students’ planning skills and oral presentation skills should be given to students together with the toothpaste problem. Teachers are welcome to modify the assessment criteria to suit their special needs, but they should not be too specific to give students a clue to the answer.
Students’ Misconceptions and Difficulties

A large majority of students suggest immersing a known mass of eggshells in a toothpaste-water mixture for a few minutes. Then, they wash the eggshells with water and add an acid to produce carbon dioxide. However, some students have the following misconceptions or limitations in their proposed experimental designs:

1. Some students plan to use limewater to measure the rate of formation of carbon dioxide gas. They pass carbon dioxide to a test-tube of limewater through a delivery tube and record the time taken for the limewater to turn milky. However, it is difficult to detect the colour change reliably.

2. Many students want to use a syringe to collect the carbon dioxide produced. They record the time taken to collect a given volume of carbon dioxide. However, the gas syringe method does not result in accurate data because the movement of the plunger is seriously affected by friction, particularly if plastic syringes are used.

3. Students may forget to include a control set-up in their experimental design. This is essential as some brands of toothpaste may speed up the reaction between calcium carbonate and acid.

4. Some students may suggest that the effect of toothpastes on the rate of reaction between the calcium carbonate in eggshells and an acid can be investigated by (a) measuring the decrease in mass of the mixture during the reaction, or (b) recording the change in pressure during the reaction using a data-logger. These procedures can be used if your school has adequate quantity of requisite equipment (e.g., electronic balances with a precision of ±0.001 g, pressure sensors, data-loggers) for students to perform the experiments in groups.
Sample Procedure

IMPORTANT: This sample procedure is for teacher information only. It should not be given to students as a cookbook-style experiment. Risk assessments should be done in advance by the teacher. Obtain MSDS information on all hazardous chemicals involved. Label chemicals with the appropriate safety hazard warning labels.

Materials (per group)

Safety goggles (1 pair per student)
Gloves (1 pair per student)
Apron or lab coat (1 per student)
Toothpaste (different brands, such as Darlic and Colgate)
Chicken egg
1.00 M hydrochloric acid (350 cm$^3$)
0.005 M sodium hydroxide (100 cm$^3$)
0.5% phenolphthalein indicator solution (5 cm$^3$)
Deionized or distilled water
250-cm$^3$ conical flask (4)
A stopper with bent glass delivery tubing
Glass rod to stir mixtures
Weighing bottle (glue a magnetic clip at the bottom)
Magnetic bar
250-cm$^3$ beaker
100-cm$^3$ beaker (4)
50-cm$^3$ measuring cylinder
5-cm$^3$ pipette
25-cm$^3$ pipette
Pipette filler
Test tube (4)
Test tube rack
Mortar and pestle
Stopwatch
Access to a balance (±0.001 g), oven, and oven mitts
Experimental Details

(A) Preparation of eggshells

1. Remove the white and the yolk from a chicken egg to leave a clean shell. Immerse the eggshell in a 250-cm$^3$ beaker of distilled water for 5 minutes. Then remove all the membranes from the inside of the eggshell. Wash the eggshell with distilled water.

2. Tare your balance to the weight of a clean and dry 100-cm$^3$ beaker. Weigh 3.00 g of toothpaste. Record the exact mass of toothpaste.

3. Add 20 cm$^3$ of distilled water to the 100-cm$^3$ beaker and stir the mixture until it becomes homogenous.

4. Crush the eggshell into several large pieces. Immerse about 2 g of the eggshell in the toothpaste-water mixture for 30 minutes.

5. Repeat Steps 2-4 with other brands of toothpaste. Use distilled water as your control. Remember to label the four beakers.

6. Wash the eggshells in beakers A, B and C with tap water and then distilled water.

7. Dry the eggshells in an oven at 110 °C for about 20 minutes.

8. Remove the eggshells from the oven (use an oven mitt). Let the eggshells cool down to room temperature.

9. Grind the eggshells to a very fine powder using a mortar and pestle. Keep the eggshell powder in the four labeled beakers.

(B) Effect of toothpastes on the rate of reaction between HCl and eggshell

1. Pour about 70 cm$^3$ of 0.005 M NaOH solution into a 250-cm$^3$ beaker. Add a few drops of phenolphthalein indicator solution to give a pink color.

2. Pipette 5.0 cm$^3$ of the NaOH-phenolphthalein solution into a test tube.

3. Pipette 25.0 cm$^3$ of 1.00 M HCl(aq) into a 250-cm$^3$ conical flask.

4. Tare your balance to the weight of a clean and dry weighing bottle (with a magnetic clip at the bottom). Weigh 0.500 g of eggshell powder from beaker A and record the exact mass.
5. Carefully put the weighing bottle into the flask. Set up the apparatus as shown below.

![Apparatus Diagram]

- 25.0 cm³ of hydrochloric acid
- Eggshell powder
- 5.0 cm³ of sodium hydroxide solution with phenolphthalein indicator

6. Tilt the weighing bottle with a magnet to start the chemical reaction and switch the stopwatch on immediately.

7. Shake the flask gently until the NaOH solution changes from pink to colourless. Stop the stopwatch and record the time in seconds.

8. Repeat Steps 2-7 with eggshell powder from beakers B, C and D.

NOTES:

- Be sure that your students wear safety goggles at all times in the lab. It is also a good idea for students to wear aprons.

- Phenolphthalein indicator solution is flammable. Use the suggested concentrations of HCl and NaOH to reduce risks; 1.00 M HCl and 0.005 M NaOH are not corrosive or irritant. This is important because the hazard of a solution depends upon its concentration. Ask your students how they should handle chemical spills properly. For example:
  
  Chemical splash on skin – immediately flush the skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Get medical aid if irritation develops or persists. Wash clothing before reuse.
  
  Chemical splash into the eyes – immediately flush the eyes with plenty of water for at least 15 minutes using an eye washer and seek medical advice immediately.

- Remind students that they should handle glassware carefully (e.g., beaker, pipette, test tube, conical flask). They should not pick up fragments of broken glass with bare hands.

- Be sure that your students wear oven mitts when removing hot beakers from the oven.
Sample Data and Results

Concentration of HCl = 1.00 M  
Concentration of NaOH = 0.0050 M  
Mass of toothpaste (different brands) used = 3.00 g

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Run 1</th>
<th>Run 2</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>Mass of eggshell used (g)</td>
<td>Time taken (s)</td>
</tr>
<tr>
<td>20 cm$^3$ distilled water</td>
<td>0.500</td>
<td>177</td>
</tr>
<tr>
<td>Colgate toothpaste + 20 cm$^3$</td>
<td>0.500</td>
<td>210</td>
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<tr>
<td>distilled water</td>
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<td></td>
</tr>
<tr>
<td>Crest toothpaste + 20 cm$^3$</td>
<td>0.500</td>
<td>240</td>
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<tr>
<td>distilled water</td>
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<td></td>
</tr>
<tr>
<td>Salz toothpaste + 20 cm$^3$</td>
<td>0.500</td>
<td>133</td>
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<tr>
<td>distilled water</td>
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</table>

Each group of students should perform several runs to find an average. The teacher may ask the class to lump their data together to facilitate them to draw conclusions. The above sample data indicate that the Crest toothpaste can slow down the rate of reaction between CaCO$_3$ and HCl more effectively than Colgate or Salz toothpaste. Interestingly, Salz toothpaste actually speeded up the reaction because it took less time for Salz to turn the NaOH solution from pink to colourless than distilled water (i.e., the control). Since Salz toothpaste contains chloride rather than fluoride, this may imply that fluoride is important to slow down the rate of reaction between CaCO$_3$ and HCl.

This investigation is also a good opportunity for students to apply what they have learned about reaction stoichiometry. Students may calculate the rate of reaction between HCl and the CaCO$_3$ in eggshell in terms of the rate of formation of carbon dioxide as follows:

The balanced equation for the reaction is CO$_2$ + 2 NaOH $\rightarrow$ Na$_2$CO$_3$ + H$_2$O

Number of moles of NaOH in the test tube = 0.0050 M $\times$ $\frac{5.0}{1000}$ L = 2.5 x $10^{-5}$ mol

Number of moles of CO$_2$ required to neutralize NaOH = $\frac{2.5 \times 10^{-5}}{2}$ = 1.25 x $10^{-5}$ mol

Rate of formation of CO$_2$ = $\frac{1.25 \times 10^{-5} \text{mol}}{\text{time taken}}$

Possible sources of error include:

1. The eggshell samples used in the experiments were not homogeneous. Thus, the samples may have contained different amounts of CaCO$_3$ even though same mass of eggshell was used.
2. The conical flasks were not shaken uniformly during the reaction. This may have affected the timing.
3. Different amounts of carbon dioxide gas dissolved in the HCl in various experiments. So, not all CO₂ passed into the NaOH solution.

4. Not all the carbon dioxide gas reacted with the NaOH solution.

References


What is the Mass of Carbon Dioxide Released by Dead Leaves?

Plant debris decomposes in the soil, releasing carbon dioxide. This is because bacteria and fungi break down dead leaves by the following chemical reaction:

$$C_x(H_2O)_y + x O_2 \rightarrow x CO_2 + y H_2O + \text{energy}$$

This reaction is one of the components of the global carbon cycle. Different types of plant material decompose at different rates. Green, leafy matter decomposes easily, but woody, stem debris takes longer. Low temperatures, dry conditions and flooding will also slow down decomposition.

Unfortunately, human activities – especially burning of fossil fuels – have led to changes in the natural carbon cycle. The cycle is now out of balance. The amount of carbon dioxide in the atmosphere has increased from 280 ppm to 370 ppm over the past 140 years, resulting in global warming via the greenhouse effect. The slight warming of the Earth’s surface may cause bacteria and fungi to decompose dead leaves more rapidly, releasing even more carbon dioxide into the atmosphere. This investigation will provide you an opportunity to understand part of the carbon cycle dynamics.

Your task:

Suppose that you work as a chemist in an environmental protection company. Your challenge
is to plan and do an investigation to determine the number of grams of CO₂ released by one gram of dead leaves per day. Submit the plan as group work by ____________ (date).

Your group will be presenting on ____________ (date) in front of the company representatives. You will have 10 minutes to present your plan, followed by 10 minutes in which you will be expected to respond to queries. Your presentation needs to answer the following questions:

- How will you measure the rate of formation of CO₂ for a particular type of leaf (e.g., oak)?
- What variables will you need to keep constant in this investigation?
- Will the proposed procedure be feasible and safe?

NOTE: After reviewing your experimental design, the teacher will discuss any safety precautions that are specific to your design. Obtain teacher approval before beginning any lab work.
Assessment Criteria for Planning the Carbon Dioxide Investigation

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<tr>
<th>Criteria</th>
<th>Marks Possible</th>
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<td>Self</td>
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<td>Teacher</td>
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<tr>
<td>1. Identify the problem and state it clearly in a way that can be investigated.</td>
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<td>2. Use proper apparatus, techniques and safety precautions.</td>
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<td>11. Lab trials are stated.</td>
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<td>13. Chemistry vocabulary is used correctly.</td>
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**Curriculum Links**

- Acid-base chemistry
- Mole concept
- Global carbon cycle
- Air pollution and global warming

**Background Information**

1. This guided inquiry is for S4-5 chemistry students. It can provide students with an opportunity to understand part of the global carbon cycle (Buell & Girard, 2003; Field & Raupach, 2004; Stanitski et al., 2003). The problem is authentic because it resembles the kind of tasks undertaken by soil scientists or ecologists (Grogan, 1998; Raich, 1998).

2. Students are seldom aware that bacteria and fungi feed on the dead remains of plants and, via respiration, release carbon dioxide to the atmosphere. An electron micrograph of bacteria on the surface of a decomposing leaf can be found in Cheung (2006).

3. Each year, the mass of carbon dioxide released by decomposing leaves is approximately equal to that released by animal and plant respiration. It is also about half of the amount of carbon dioxide removed by photosynthesis.

4. In the lab, the mass of carbon dioxide released by one gram of dead leaves per day depends upon many factors such as the type of plant, amount of bacteria and fungi, and temperature. Although there is no easy way to check the accuracy of the results obtained by students, this guided inquiry is a good opportunity to deepen their conceptual and procedural understanding (Gott & Duggan, 1995).

5. The dead leaves of various types of plant can be used for this guided inquiry. Our trials (Cheung, 2006) indicated that the following two types can generate measurable results after one week:

*Liquidambar formosana* Hance (金縷梅科: 楓樹、楓、香楓)
Students’ Misconceptions and Difficulties

1. Some students do not recognize that carbon dioxide is present in the air even without the decomposing leaves. They have to determine how much more carbon dioxide is produced as a result of the leaf decomposition. A control setup is needed.

2. Many students want to use limewater to absorb the carbon dioxide released by dead leaves and to determine the mass of carbon dioxide by weighing a bottle of limewater before and after a measurement period. This method does not work because limewater cannot absorb carbon dioxide efficiently.

3. Some students use sodium hydroxide solution to absorb the carbon dioxide released by dead leaves and then perform a back titration with hydrochloric acid to find out any excess sodium hydroxide. But sodium hydroxide reacts with carbon dioxide to form sodium carbonate. Therefore, the resulting solution is a mixture of carbonate ions and hydroxide ions.

\[
2\text{NaOH(aq)} + \text{CO}_2(\text{g}) \rightarrow \text{Na}_2\text{CO}_3(\text{aq}) + \text{H}_2\text{O(l)}
\]

During the back titration, hydrochloric acid will react with both carbonate ions and hydroxide ions. The composition of the hydroxide-carbonate mixture can be found by the double indicators method (Skoog, West, Holler & Crouch, 2000). Alternatively, barium chloride solution may be added to the mixture to selectively precipitate the carbonate ions before the back titration is done (Wink, Gislason & Kuehn, 2005). Unfortunately, these two methods are beyond S4-5 chemistry students.

4. We recommend soda lime. Students should note that solid sodium hydroxide alone is not satisfactory to absorb carbon dioxide in this guided inquiry because it absorbs water
vapour from the air and puddles of extremely concentrated (and corrosive) sodium hydroxide solution will be formed. A lot of heat will also be released when sodium hydroxide solution is formed.

5. Students should also note that absorption of carbon dioxide using solid calcium hydroxide alone is not efficient because carbon dioxide must be dissolved in water before it can react with calcium hydroxide. Soda lime is more efficient as sodium hydroxide is added to calcium hydroxide to absorb moisture. The moisture in soda lime granules is not visible when the water content is less than 20%. Because carbon dioxide is chemically bound but the moisture is not, soda lime can be dried and weighed before and after a measurement period to determine the amount of carbon dioxide absorbed.

The mechanism of CO$_2$ absorption is too complicated for S4-5 students. The equations (Iwona & Ryszard Klos, 2004) shown below are for teacher information:

\[
\begin{align*}
H_2O + CO_2 & \Leftrightarrow H_2CO_3 \\
H_2CO_3 & \Leftrightarrow H^+ + HCO_3^- \\
HCO_3^- & \Leftrightarrow H^+ + CO_3^{2-} \\
2H^+ + CO_3^{2-} + 2NaOH & \rightarrow Na_2CO_3 + 2H_2O \\
Na_2CO_3 + Ca(OH)_2 & \rightarrow CaCO_3 + 2NaOH
\end{align*}
\]

6. Although soda lime is a variable mixture of sodium hydroxide and calcium hydroxide, we do not need to know the exact percentages of these chemicals in order to calculate the mass of carbon dioxide absorbed. The teacher may guide students to apply chemistry concepts to solve this problem as follows:

Absorption of carbon dioxide occurs by the following overall chemical reactions:

(i) \( Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \)
(ii) \( 2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O \)

For every mole of CO$_2$ that is reacted with Ca(OH)$_2$ in the soda lime, one mole of H$_2$O is formed that is subsequently evaporated during oven-drying. Thus, the increase in mass of dried soda lime measured before and after the experiment is not equal to the mass of CO$_2$ absorbed. From the first chemical equation, if one mole (44 g) of CO$_2$ has been absorbed, the increase in mass is equal to

\[
\text{molar mass of CaCO}_3 - \text{molar mass of Ca(OH)$_2$} = 100 - 74 = 26 \text{ g}
\]

The mass of CO$_2$ absorbed by Ca(OH)$_2$ is proportional to the increase in mass after the experiment. Thus,

\[
\frac{\text{Mass of CO}_2 \text{ absorbed}}{44g} = \frac{\text{Increase in mass}}{26g}
\]

\[
\text{Mass of CO}_2 \text{ absorbed} = \text{Increase in mass} \times \frac{44g}{26g} = \text{Increase in mass} \times 1.69
\]
Similarly, from the second chemical equation, if one mole (44 g) of CO\textsubscript{2} has been absorbed by two moles of NaOH in the soda lime, the increase in mass is equal to

molar mass of Na\textsubscript{2}CO\textsubscript{3} – the mass of two moles of NaOH = 106 – 2 \times 40 = 26 g

Like Ca(OH)\textsubscript{2}, the mass of CO\textsubscript{2} absorbed by NaOH = increase in mass \times 1.69. Thus, the relative amounts of Ca(OH)\textsubscript{2} and NaOH in a sample of soda lime is not important in this guided inquiry. Some chemical suppliers also add potassium hydroxide to soda lime, but the mass of CO\textsubscript{2} absorbed by potassium hydroxide can also be obtained by multiplying the increase in mass by 1.69.
Sample Procedure

**IMPORTANT:** This sample procedure is for teacher information only. It should not be given to students as a cookbook-style experiment. Risk assessments should be done in advance by the teacher. Obtain MSDS information on all hazardous chemicals involved. Label chemicals with the appropriate safety hazard warning labels.

**Materials (per group)**

- Safety goggles (1 pair per student)
- Rubber gloves (1 pair per student)
- Apron or lab coat (1 per student)
- Two airtight containers (at least 28 cm x 21 cm x 6 cm)
- 10 dry dead leaves (same type of leaf)
- Soda-lime (1.0 – 2.5 mm granular size, about 40 g, hazard warning label = corrosive)
- Deionized or distilled water
- Two glass Petri dishes (at least 9 cm diameter)
- 10-cm³ measuring cylinder
- Spatula
- Access to a balance (±0.001 g), oven, and oven mitts

**Experimental Details**

(A) Pre-lab to be done by technician

1. Put a thin layer of granulated soda lime in the bottom plate of the Petri dish. Find out the mass of soda lime used. This is approximately half of the mass of soda lime to be used by one group of students. Estimate the total amount of soda lime needed by the whole class.

2. To inactivate soda lime, put the estimated total amount of soda lime in a beaker and dry it in an oven at 105 °C for 24 hours. Place the soda lime in a desiccator immediately upon removal from the oven.

(B) Student experiment

1. Put on your safety goggles. Wear lab apron and gloves. Obtain two airtight containers. Label one container as “Experimental” and the other as “Control.”

2. Obtain 8 to 10 leaves (use the same type of leaf and if the leaves are large in size, reduce the number of leaf). Record the total mass of leaves. Put the leaves into the experimental container.

3. Obtain two glass Petri dishes. Fill the top plates of the dishes half full of distilled water. Place one plate of water into the Experimental container and the other plate into the control container as shown in Figure 1.
4. Using a marker, label the bottom plates of the two Petri dishes as “experimental” and “control.” Weigh each plate separately and record its exact mass. Using a spatula, spread out a thin layer of the oven-dried soda lime granules in the plates. **CAUTION:** Soda lime is corrosive. Do not touch it with your bare hands. Weigh each plate separately and record the exact mass of soda lime used. Place the plates containing soda lime into the experimental and control containers.

5. Using a 10-cm³ measuring cylinder, carefully add 5.0 cm³ of distilled water to the soda lime in the experimental and control containers to activate the soda lime. Immediately seal the containers.

6. Place the two airtight containers in the location specified by your teacher for one week.

7. After one week, open the experimental and control containers and remove the two soda lime dishes. Dry the soda-lime at 105 °C in an oven for 24 hours. Find out the mass of CO₂ absorbed by re-weighing the two soda lime dishes.
NOTES:

- Do NOT clean the leaves. We need to keep the bacteria and fungi.
- Since soda lime is inefficient for CO\textsubscript{2} absorption unless moisture is available, the above sample procedure allows water to evaporate from a dish to increase humidity. Remind students that soda lime is corrosive and thus they should not touch it with their bare hands.
- We recommend granular (1.0 – 2.5 mm) soda lime. The teacher may use other granular sizes, but it should be small enough for a large surface-to-volume ratio and large enough to prevent losses of fine particles during drying and handling.
- A balance with adequate sensitivity is a must.

**Sample Data and Results**

Mass of dry dead leaves used = 3.073 g  
Name of plant = *Liquidambar formosana*  
Duration = 7 days

<table>
<thead>
<tr>
<th></th>
<th>Experimental</th>
<th>Control</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final mass of soda lime (g)</td>
<td>20.310</td>
<td>20.352</td>
</tr>
<tr>
<td>Initial mass of soda lime (g)</td>
<td>20.275</td>
<td>20.347</td>
</tr>
<tr>
<td>Increase in mass (g)</td>
<td>0.035</td>
<td>0.005</td>
</tr>
<tr>
<td>Mass of CO\textsubscript{2} absorbed (g)</td>
<td>0.035 x 1.69 = 0.059</td>
<td>0.005 x 1.69 = 0.008</td>
</tr>
</tbody>
</table>

To find the amount of CO\textsubscript{2} released by decomposing leaves, students need to subtract the mass of CO\textsubscript{2} absorbed in the control container from the mass of CO\textsubscript{2} absorbed in the experimental container. Therefore, the mass of CO\textsubscript{2} released per gram of leaf per day

\[ \frac{(0.059 - 0.008)g}{3.073g \times 7 days} \]

= 0.0024 g

Possible sources of error include:

1. We are not sure whether the soda lime in the experimental setup has absorbed all of the CO\textsubscript{2} released by the decomposing leaves.
2. The soda lime may have absorbed some CO\textsubscript{2} from the air during the final oven-drying phase in the experiment.
3. Normal air flow was not allowed in the experimental setup. This might have affected the respiration rate of bacteria and fungi.
4. The quantity and/or the surface area of soda lime granules may have limited the CO$_2$ absorption during the measurement period.

References


What is the Order of Reaction?

Metabolism of fats and carbohydrates in humans produces toxic hydrogen peroxide as a by-product. One of the harmful effects of hydrogen peroxide is that it causes damage to our DNA and cell membranes, resulting in aging. Fortunately, there is an enzyme called catalase in our cells, which can speed up the decomposition of $H_2O_2$ into harmless $H_2O$ and $O_2$.

$$2\ H_2O_2 \rightarrow 2\ H_2O + O_2$$

Catalase activity is particularly high in our liver, kidney and red blood cells. But the mechanism of this catalase-catalyzed reaction is not fully understood. The relationship between reaction rate and concentration of $H_2O_2$ can be represented as follows:

$$\text{Rate} = k\ [H_2O_2]^x[catalase]^y$$

where $k$ is the rate constant, the exponent, $x$, is the order of the reaction with respect to $H_2O_2$, and the exponent, $y$, is the order of the reaction with respect to catalase.

Catalase is present in some vegetables and fruits. Examples are onion, carrot, celery, lettuce and papaya. Measurement of catalase activity is important in industrial food production. Imagine that you work as a biochemist in a canning factory. Your boss has asked you to find out how catalase in lettuce catalyzes the decomposition of hydrogen peroxide. To explore the reaction mechanism, you need to carry out kinetic analysis. Your challenge is to design and carry out simple, quick experiments to determine the value of $x$ accurately. Submit your plan by _____________ (date).

You will be presenting on _____________ (date) in front of a group of biochemists. You will have 10 min to present your plan, followed by 10 min in which you will be expected to respond to queries. Your presentation needs to answer the following questions:

- What method will you use to measure the reaction rate? Why?
- How will you find out the value of $x$?
- Will your proposed procedure be rapid and safe?

NOTE: After reviewing your experimental design, the teacher will discuss any safety precautions that are specific to your design. Obtain teacher approval before beginning any lab work.
Assessment Criteria for Planning the H₂O₂ Investigation

Names of Students: ____________________________
______________________________
______________________________
______________________________
Date: ____________________________

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. The method for measuring the reaction rate is simple, quick and accurate.</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
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<tr>
<td>2. Calculations of the value of x are shown correctly.</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
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<tr>
<td>3. Suitable choice of chemicals and apparatus.</td>
<td>_______</td>
<td>_______</td>
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<tr>
<td>4. Chemicals and apparatus are easily available.</td>
<td>_______</td>
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<tr>
<td>5. Measurement errors are minimized by appropriate procedures or apparatus.</td>
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<td>6. Specific hazardous chemicals are identified.</td>
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<td>7. Steps are included to reduce risks.</td>
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<td>8. No invalid assumptions are made.</td>
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<td>9. The methods are clear enough to be followed by other students.</td>
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<td>10. Reagents that need accurate measurement are identified.</td>
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<td>11. Lab trials are stated.</td>
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<td>12. Repeats are stated.</td>
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<tr>
<td>13. Chemistry vocabulary is used correctly.</td>
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<tr>
<td>14. Limitations of the experimental design are described.</td>
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<td>_______</td>
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</tbody>
</table>

TOTAL: _______ _______ _______
Curriculum Links

- Rate equation
- Order of reaction
- Factors affecting reaction rate
- Catalysis
- Enzymatic reactions

Background Information

1. Some biology teachers provide students an opportunity to compare the relative catalase activity of different types of vegetables using hydrogen peroxide as a substrate. In our sixth form chemistry curriculum (CDC-HKEAA, 2005), catalytic decomposition of hydrogen peroxide is also listed as a learning activity for the sub-topic ‘Catalysis’ but manganese(IV) oxide serves as the catalyst. Students often complain that chemistry laboratory work is not relevant to their everyday life.

2. Actually, catalase is a very important human enzyme. In this inquiry-based experiment, Secondary 6-7 students are required to investigate the rate of catalase-catalyzed decomposition of hydrogen peroxide and determine the order of reaction with respect to hydrogen peroxide.

3. A catalyst concentration should be included in the rate law expression for the overall reaction if the catalyst is involved in the rate-determining step. Unfortunately, few teachers point out this important concept when they teach chemical kinetics.

4. For the catalase-catalyzed decomposition of hydrogen peroxide, the rate law expression is rate = k[H_2O_2]^x[catalase]^y. The value of x can be determined by kinetic analysis if we vary the initial concentration of hydrogen peroxide but keep the temperature and the concentration of catalase constant. The enzymatic reaction is first order with respect to hydrogen peroxide (Kimbrough, Magoun & Langfuir, 1997; Stauffer, 1989). However, hydrogen peroxide solutions at high concentrations should be avoided because catalase will be inactivated and the reaction rate may be limited by the catalase concentration and consequently the reaction is zero order rather than first order with respect to hydrogen peroxide.

5. Avoid asking students to verify that the reaction is first order with respect to hydrogen peroxide. They will have the feel of discovery if they need to find out the answer based on real data.

6. Catalase is a heme protein. The iron in catalase serves as a redox reagent. The Fe(III) is reversibly oxidized and reduced, acting as an electron exchanger. But the detailed mechanism for the reaction is not yet available. A two-step process has been proposed by researchers (Mate et al., 2001; Walsh, 1979):

\[
[catalase-Fe(III)] + H_2O_2 \rightarrow H_2O + [catalase-Fe(IV)=O]
\]

\[
[catalase-Fe(IV)=O] + H_2O_2 \rightarrow H_2O + O_2 + [catalase-Fe(III)]
\]
7. The reaction rate can be measured by following either the decomposition of hydrogen peroxide or the generation of oxygen gas. Enzymologists measure the reaction rate by modern analytical methods such as UV spectrophotometry and chemiluminescence. In this investigation, Secondary 6-7 students are required to design a simple, quick and accurate method to determine the value of x. Titrimetric methods are time-consuming and may not be suitable for coloured vegetable extracts. Our trials indicated that direct measurement of volume of gases from water displacement in a measuring cylinder can determine the order of reaction efficiently. This method is sufficiently accurate for secondary school chemistry if appropriate steps are included to guarantee good reliability of data.

8. The assessment criteria for planning the investigation and oral presentation should be distributed to students together with the problem. Teachers are most welcome to adapt the criteria to meet their needs but the criteria should not be too specific to give students a clue to the answer (e.g., a proper method to collect oxygen gas).

9. To make the investigation more authentic, the teacher should avoid buying a commercial sample of catalase from a chemical company. Instead, students should be given an opportunity to extract catalase from a natural source. Our trials demonstrated that fresh lettuce (西生菜) can produce a suitable concentration of catalase for this investigation. Do not use fruits such as pineapples and bananas (see the table below) as the volume of oxygen gas evolved is relatively small and thus it is difficult to measure the volume of oxygen gas reliably.

<table>
<thead>
<tr>
<th>Relative Catalase Activity</th>
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<tbody>
<tr>
<td><strong>High</strong></td>
</tr>
<tr>
<td>Lettuce</td>
</tr>
<tr>
<td>Celery</td>
</tr>
<tr>
<td>Green Cabbage</td>
</tr>
<tr>
<td>Carrots</td>
</tr>
<tr>
<td>Chinese Cabbage</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

10. The disproportionation reaction, \( 2\text{H}_2\text{O}_2 \rightarrow 2\text{H}_2\text{O} + \text{O}_2 \), can be speeded up by light. Therefore, hydrogen peroxide should be stored in dark bottles. Also, the gas pressure resulting from formation of oxygen gas could cause a bottle to explode. Store hydrogen peroxide in a cool place.

11. Let students know that hydrogen peroxide is sold as a 3-percent aqueous solution for medical use as an antiseptic.
Students’ Misconceptions and Difficulties

1. Some students think that they have to collect pure oxygen gas.

2. Some students plan to mix the reactants in a test-tube or boiling tube. But reactants cannot be mixed well in a tube. Even worse, a think layer of foam will form and collection of oxygen will be affected.

3. Many students want to use a syringe to collect oxygen gas. However, the gas syringe method cannot give reliable data on reaction rate because there is friction between the plunger and barrel. Our trials have confirmed that less gas will be collected if a glass syringe is used; the use of plastic syringes is even worse.

4. Many students plan to measure the reaction rate at room temperature by recording the oxygen produced after a period of time (e.g., 2 min) and then calculate the average rate. But measurement of reaction rates is easily affected by numerous factors such as pH changes and the presence of side reactions. To minimize those factors, the method of initial rate should be used.

5. The initial rate can be found by drawing the tangent to the volume-time curve at time equal to zero. However, if students look at the plot carefully, they will find that the initial segment of the curve is quite straight. Below is an example. Note that the line from 0 – 40 seconds is fairly straight. Therefore, the volumes of oxygen collected in 40 seconds using different concentrations of hydrogen peroxide solutions may be taken to indicate the initial rates.
6. A lot of students plan to add the lettuce extract to H₂O₂ solution directly in a flask, assuming that no oxygen gas or air will be lost by closing the flask with a stopper quickly. A way to minimize loss of gases should be included in the experimental plan.

7. To prevent loss of oxygen or air, some students suggest that a tap funnel may be used to add catalase (see the figure below). However, they do not recognize that the catalase solution will displace air. As a result, they forget to subtract the volume of catalase solution added from the total volume of gases collected. Another disadvantage of the use of a tap funnel is that it takes several seconds for the catalase solution to pour into the flask and this may affect the measurement of reaction rate.
Sample Procedure

**IMPORTANT:** This sample procedure is for teacher information only. It should not be given to students as a cookbook-style experiment. Risk assessments should be done in advance by the teacher. Obtain MSDS information on all hazardous chemicals involved. Label chemicals with the appropriate safety hazard warning labels.

**Materials (per group)**

- Safety goggles (1 pair per student)
- Rubber gloves (1 pair per student)
- Apron or lab coat (1 per student)
- Lettuce (fresh lettuce must be used to extract catalase)
- 1.5% (0.441 M) hydrogen peroxide solution
- Deionized or distilled Water
- Mortar and pestle
- Filter funnel
- Tea bag paper or cheesecloth
- 50-cm³ or 100-cm³ wide-necked conical flask (with a stopper and bent glass delivery tubing)
- Small weighing bottle (glue a magnetic clip at the bottom of the bottle and check whether it fits the wide-necked conical flask)
- Magnetic bar
- 100-cm³ conical flask (with a stopper)
- 100-cm³ volumetric flask
- 50-cm³ measuring cylinder
- 25-cm³ measuring cylinder
- 25-cm³ pipette
- 10-cm³ graduated pipette
- 5-cm³ graduated pipette (2)
- Pipette filler
- Droppers
- Forceps
- Wash bottle
- Glass rod to stir the catalase extract
- Rubber tubing for collecting gases over water
- Stopwatch
- Stand, boss and clamp
- Water trough
- scissors
- Access to a balance and knife
Experimental Details

(A) Dilution of hydrogen peroxide solution

1. Pipette 50.0 cm$^3$ of 1.5% (0.441 M) H$_2$O$_2$ solution into a clean 100-cm$^3$ volumetric flask and bring to volume with deionized water. Mix the contents well and label the flask.

2. Keep the 1.5% and 0.75% H$_2$O$_2$ solutions for Part B.

(B) Preparation of catalase extract

NOTE: The amount of catalase may vary from one preparation to another. You should use one single preparation for all trials. Also, catalase activity will decrease rapidly due to standing. You need to complete all trials within one hour.

1. Halve a lettuce. Weigh about 100 g of lettuce for extraction of catalase.

2. Using a pair of scissors, cut the lettuce into small pieces in a large, clean mortar. Avoid losing any juice.

3. Using a 50-cm$^3$ measuring cylinder, add 50 cm$^3$ of deionized water to the lettuce.

4. To extract catalase from cells, crush the lettuce using a pestle until it has become a fine pulp. This step may take 10 minutes.

5. Cut one side of a tea bag with a pair of scissors to make a cone. Put it over a filter funnel. (Alternatively, cheesecloth may be used to filter the lettuce suspension.)

6. Filter the resulting lettuce suspension through the tea-bag to remove solid particles. Collect the filtrate using a 100-cm$^3$ conical flask.

7. Squeeze the tea bag to obtain more lettuce juice.

8. When filtration is done, stopper the conical flask immediately to minimize oxidation of catalase by air. Dispose of the lettuce solid residue.

(C) Determination of the volume of oxygen gas

1. Using a 5-cm$^3$ graduated pipette, pipette 2.0 cm$^3$ of 1.5% H$_2$O$_2$ solution into a small weighing bottle (with a magnetic clip at the bottom).
Using a 10-cm³ graduated pipette, pipette 8.0 cm³ of lettuce juice into a 50-cm³ wide-necked conical flask.

With the aid of a pair of long forceps, carefully put the weighing bottle into the conical flask. Avoid splashing H₂O₂ about.

Fill a 25-cm³ measuring cylinder with tap water. Invert it in a water trough. Set up the apparatus as shown in Figure 1. CAUTION: Make sure that all connections are gas tight.

Tilt the weighing bottle with a magnet to start the enzymatic reaction. Start the stopwatch immediately. Vigorously shake the contents at a rate of about 3 shakes per second.

After 40 seconds have passed away, remove the rubber tubing to stop collection of gases. Make the pressure on the gases inside the measuring cylinder equal to atmospheric pressure by lowering the cylinder until the water level inside is the same as that outside. Record the final volume and note the number of significant figures.

Repeat the experiment at least two times to ensure consistency of results and calculate an average. The same 1.5% hydrogen peroxide solution must be used. Rinse the reaction flask with deionized water after each trial.

Repeat steps 1 - 7 but use the 0.75% H₂O₂ prepared in part A.

Figure 1. An experimental set-up

NOTES:

- Extraction of catalase should only be done with fresh lettuce tissues.
- All of the trials are allowed to proceed for 40 seconds and thus the rate measurement is a very close approximation to the initial rate of formation of oxygen gas from the enzymatic decomposition of hydrogen peroxide.
- To reduce risks and inactivation of catalase, we recommend the use of 1.5% hydrogen peroxide solution, that is, 1.5 g of hydrogen peroxide per 100 g of solution.
- It is not necessary to standardize the 1.5% and 0.75% hydrogen peroxide solutions because only a ratio rather than absolute values of the concentrations is needed in the
calculation.

- To improve accuracy, graduated pipettes should be used to measure the hydrogen peroxide solution and catalase extract.

- Alternatively, if students attempt to measure the rate at more than two different concentrations of hydrogen peroxide, they may plot log initial rate against log [H₂O₂]. The plot should yield a straight line with slope equal to the order. Students may adjust the total volume of the reaction mixture so that it is the same in each run. This allows them to use the number of cm³ of the hydrogen peroxide solution as a measure of concentration, rather than the actual number of moles/dm³.

- A wide-necked 50 cm³ conical flask is recommended to allow for placement of a small weighing bottle inside it. Check whether such a type of flask is available in the lab. Larger conical flasks are not recommended because the total volume of catalase and hydrogen peroxide is just 10 cm³. The photo below shows three types of flasks: wide-necked 50 cm³ conical flask (right); wide-necked 100 cm³ conical flask (middle); and ordinary 100 cm³ conical flask (left).

Sample Data and Results

<table>
<thead>
<tr>
<th></th>
<th>Run 1</th>
<th>Run 2</th>
<th>Run 3</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of O₂ collected using 1.5% H₂O₂ (cm³)</td>
<td>17.0</td>
<td>17.0</td>
<td>16.8</td>
<td>16.9</td>
</tr>
<tr>
<td>Volume of O₂ collected using 0.75% H₂O₂ (cm³)</td>
<td>8.5</td>
<td>8.5</td>
<td>8.4</td>
<td>8.5</td>
</tr>
</tbody>
</table>

The rate equation for the enzymatic reaction is

\[ \text{Rate} = k \ [\text{H}_2\text{O}_2]^x[\text{catalase}]^y \]

To find \( x \), we can divide the rate equation for 1.5% \( \text{H}_2\text{O}_2 \) by the rate equation for 0.75% \( \text{H}_2\text{O}_2 \),

\[ \frac{rate_1}{rate_2} = \frac{k[\text{H}_2\text{O}_2]^x[\text{catalase}]^y}{k[\text{H}_2\text{O}_2]^2[\text{catalase}]^2} \]

59
The value of $k$ cancels from such a ratio because it is constant at a particular temperature. The initial concentrations of catalase were equal in all the experiments, so they too cancel. Thus, the expression simplifies to

$$\frac{\text{rate}_1}{\text{rate}_2} = \frac{[H_2O_2]_{i_1}^x}{[H_2O_2]_{i_2}^x}$$

We measured the reaction rate in terms of the change in volume of oxygen evolved with time.

$$\text{rate} = \frac{\text{volume of oxygen}}{\text{time}}$$

In all the experiments, the time was fixed at 40 seconds. So, the expression simplifies to

$$\frac{\text{rate}_1}{\text{rate}_2} = \frac{\text{volume}_1}{\text{volume}_2} = \frac{[H_2O_2]_{\text{initial}}^x}{[H_2O_2]_{\text{final}}^x}$$

The value of $x$ can be found by substituting data into the above expression.

$$\frac{16.9}{8.5} = \left(\frac{1.5}{0.75}\right)^x$$

$$2.0 = (2.0)^x$$

$$x = 1$$

Possible sources of error include:

1. Oxygen gas is slightly soluble in water. Some oxygen gas dissolved in the reaction mixture and the water in the trough.
2. The crude extract of lettuce, from which we obtained catalase, may have contained other substances that can react with hydrogen peroxide to produce gases.
3. It was difficult to measure accurately the bottom of the meniscus of water inside the measuring cylinder.
4. The lettuce catalase deteriorated in air.

References


**Is Catalase An Efficient Catalyst?**

The decomposition of hydrogen peroxide into water and oxygen can be catalyzed by inorganic substances such as iodide ion, iron(II) ion, and manganese(IV) oxide.

\[
2 \text{H}_2\text{O}_2 \rightarrow 2 \text{H}_2\text{O} + \text{O}_2
\]

Actually, this is an important reaction taking place in human cells. Metabolism of fats and carbohydrates in humans produces toxic hydrogen peroxide as a by-product. One of the harmful effects of hydrogen peroxide is that it causes damage to our DNA and cell membranes, resulting in aging. Fortunately, there is an enzyme called catalase in our cells, which can speed up the decomposition of hydrogen peroxide into harmless water and oxygen molecules. Catalase is also present in some vegetables and fruits. Examples are onion, carrot, celery, lettuce and papaya.

*Your task:*

Kinetic studies play a significant role in investigating the mechanisms of reactions. A catalyst offers an alternative reaction pathway with a lower activation energy, thereby increasing the proportion of molecules that have enough energy to form products. Imagine that you work as a biochemist and want to investigate the catalase activity in lettuce. Your challenge is to design and carry out an investigation to compare the activation energies for the catalase-catalyzed and iodide-catalyzed decomposition of hydrogen peroxide. Based on the activation energies, you can determine whether iodide or catalase is a more efficient catalyst. Submit your experimental plan by _______ (date).

You will be presenting on _______ (date) in front of a group of biochemists. You will have 10 minutes to present your plan, followed by 10 minutes in which you will be expected to respond to queries. Your presentation needs to answer the following questions:

- What method will you use to measure the reaction rates? Why?
- How will you determine the activation energies?
- Will your proposed procedures be feasible and safe?

NOTE: After reviewing your experimental design, the teacher will discuss any safety precautions that are specific to your design. Obtain teacher approval before beginning any lab work.
**Assessment Criteria for Planning the Activation Energy Investigation**

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment</th>
<th>Self</th>
<th>Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. The method for measuring the reaction rate is simple, quick and accurate.</td>
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<td></td>
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<tr>
<td>2. The way to calculate the value of activation energy is shown correctly and clearly.</td>
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<tr>
<td>3. Suitable choice of chemicals and apparatus.</td>
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<tr>
<td>4. Chemicals and apparatus are easily available.</td>
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<tr>
<td>5. Measurement errors are minimized by appropriate procedures or apparatus.</td>
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<td>7. Steps are included to reduce risks.</td>
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<tr>
<td>8. No invalid assumptions are made.</td>
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<td>9. The methods are clear enough to be followed by other students.</td>
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<tr>
<td>14. Limitations of the experimental design are described.</td>
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</table>

**TOTAL:** | | | | |
Curriculum Links

- Catalysis
- Enzymatic reactions
- Activation energy
- The Arrhenius equation

Background Information

1. This inquiry-based experiment may serve as an extension of the determination of the order of reaction with respect to hydrogen peroxide. Students are required to compare the activation energies for the catalase-catalyzed and iodide-catalyzed decomposition of hydrogen peroxide. Chemistry teachers always emphasize that a catalyst increases the rate of a reaction by providing an alternative pathway with a lower activation energy than the uncatalyzed pathway. Enzymes are biological catalysts. For a given enzymatic reaction, the enzyme is more efficient than inorganic catalysts. However, Secondary 6-7 students generally have few opportunities to plan procedures for comparing the efficiency of enzymes and inorganic catalysts.

2. To make the guided inquiry more authentic, the teacher should avoid buying a commercial sample of catalase from a chemical company. Instead, students should extract catalase from a natural source. Our trials demonstrated that lettuce (西生菜) can produce a suitable concentration of catalase for this guided inquiry.

3. Owing to constraints on equipment and time, most students in Hong Kong secondary schools plan to measure the reaction rate by collecting and measuring the oxygen gas evolved over water in an inverted cylinder. This method is acceptable if appropriate steps are taken to reduce experimental errors. For example, it is important to shake the reaction flask vigorously and continuously after the chemicals are mixed. Otherwise, less oxygen will be collected. All the joints must also be tight. Professional biochemists use pure catalase and sophisticated equipment such as spectrophotometer to measure reaction rates. Obviously, the methods proposed by Secondary 6-7 students are not as accurate as those used by professional biochemists. Nevertheless, this guided inquiry is a good opportunity for students to analyze and interpret kinetic data.

4. Catalase is a very efficient catalyst. Each molecule of catalase can convert $4 \times 10^7$ H$_2$O$_2$ molecules to products per second under optimal conditions.

Students’ Misconceptions and Difficulties

1. Some students have the misconception that they cannot determine the activation energy for a chemical reaction experimentally unless the absolute values of rate constants are found. The rate constants at different temperatures can be calculated either from the rate law or from the integrated rate equation. Because the rate law was not given in the problem and the concentration of catalase in lettuce is unknown, students did not know how to proceed. Actually, the determination of activation energy can be done without knowing the rate law of a reaction. Below is the explanation:

For the catalase-catalyzed reaction, the relationship between reaction rate and
concentration of hydrogen peroxide can be represented as follows:

\[
\text{rate} = k [\text{H}_2\text{O}_2]^x [\text{catalase}]^y
\]

where \( k \) is the rate constant, the exponent, \( x \), is the order of the reaction with respect to hydrogen peroxide, and the exponent, \( y \), is the order of the reaction with respect to catalase.

If we apply the method of initial rate, the concentrations of hydrogen peroxide and catalase can be assumed to be unchanged. Thus, the above rate law may be replaced by

\[
\text{initial rate} = ck
\]

where \( c \) is a constant.

The variation of reaction rate with temperature is usually expressed as the Arrhenius equation, which in its integrated form is

\[
k = A e^{-\frac{E_a}{RT}}
\]

where \( A \) is a constant called the frequency factor, \( E_a \) is the activation energy, \( R \) is the universal gas constant (8.314 J/mol-K), and \( T \) is the absolute temperature. Since \( \text{initial rate} = ck \), the initial rate can be found by

\[
\text{initial rate} = cA e^{-\frac{E_a}{RT}}
\]

Taking logarithms,

\[
\ln \text{initial rate} = \ln cA - \frac{E_a}{RT}
\]

or

\[
\log \text{initial rate} = \log cA - \frac{E_a}{2.303RT}
\]

Thus, we can plot \( \log \text{initial rate} \) against \( 1/T \) to obtain a straight line. The slope is multiplied by \(-2.303R\) to get \( E_a \). The rates can be expressed in volume of oxygen per second or in arbitrary units because the slope of the straight line will not be affected.

The activation energy can also be calculated from the following form of the Arrhenius equation:

\[
E_a = \frac{RT_2 T_1 \ln \left( \frac{\text{initial rate}_2}{\text{initial rate}_1} \right)}{T_2 - T_1}
\]

But determination of activation energy by collecting data at two different temperatures cannot test the Arrhenius equation. More importantly, large experimental errors may result. A more reliable method is to calculate the activation energy from the Arrhenius plot.
2. Some students forget to check the optimum temperature for the catalase-catalyzed reaction. The optimum temperature for catalase is determined by the balance between the effect of temperature on the rate of the enzymatic reaction and its effect of the rate of inactivation of catalase. It also varies with factors such as the source and purity of catalase. The optimum temperature is around 40 °C (Kimbrough, Magoun & Langfur, 1997). Students should first conduct preliminary experiments at two temperatures (e.g., 40 and 45 °C) to test the effect of temperature on lettuce catalase activity. Then, at least four different temperatures below the optimum temperature are selected for the main study. The temperature intervals should be at least 5 °C.

3. A lot of students believe that the two activation energies for catalase-catalyzed and iodide-catalyzed reactions must be measured under the same experimental conditions in order to have a fair test. Therefore, they plan to control the temperature range and the concentrations of hydrogen peroxide and catalysts. In fact, activation energy is essentially independent of temperature and concentrations of reactants and catalysts. For each reaction, all the students have to do is to carry out preliminary experiments to find out suitable concentrations and volumes of hydrogen peroxide and catalyst solutions so that a measurable volume of oxygen is produced within a short period of time.

4. Many students planned to measure the reaction rate by recording the oxygen gas produced after a period of time (e.g., 2 min) and then calculate the average rate. However, measurement of reaction rates is easily affected by numerous factors such as pH changes and the presence of side reactions. To minimize those factors, the method of initial rate is best to determine activation energies. Students should plot volume of oxygen against time and focus on the initial segment of the curve.
Sample Procedure

IMPORTANT: This sample procedure is for teacher information only. It should *not* be given to students as a cookbook-style experiment. Risk assessments should be done in advance by the teacher. Obtain MSDS information on all hazardous chemicals involved. Label chemicals with the appropriate safety hazard warning labels.

**Materials (per group)**

- Safety goggles (1 pair per student)
- Gloves (1 pair per student)
- Apron or lab coat (1 per student)
- Lettuce (fresh lettuce is best)
- 100 cm$^3$ of 0.44 M (1.5%) hydrogen peroxide solution (for the catalase experiments)
- 200 cm$^3$ of 0.22 M (0.75%) hydrogen peroxide solution (for the iodide experiments)
- 50 cm$^3$ of 0.1 M potassium iodide solution
- Deionized or distilled water
- Mortar and pestle
- Filter funnel
- Tea bag paper or cheesecloth
- 1000-cm$^3$ beaker (for preparing an ice-water bath)
- 50-cm$^3$ or 100-cm$^3$ wide-necked conical flask (with a stopper, thermometer and bent glass delivery tubing)
- Small weighing bottle (glue a magnetic clip at the bottom of the bottle and check whether it fits the wide-necked conical flask)
- Magnetic bar
- 250-cm$^3$ conical flask (with a stopper)
- 50-cm$^3$ measuring cylinder
- 25-cm$^3$ measuring cylinder
- 10-cm$^3$ measuring cylinder
- 10-cm$^3$ graduated or bulb pipette
- 5-cm$^3$ graduated or bulb pipette
- 2-cm$^3$ graduated or bulb pipette
- Pipette filler
- Droppers
- Forceps
- Wash bottle
- Glass rod to stir the catalase extract
- Rubber or plastic tubing for collecting gases over water
- Stopwatch
- Stand, boss and clamp (2)
- Water trough
- Deep bowl (diameter $\geq$ 14 cm) to serve as a water bath
- Electric hot plate or Bunsen burner to heat up water
- Thermometer to check the temperature of water bath
- Scissors
- Access to a balance, knife, and ice
Experimental Details

(A) Extraction of catalase from lettuce

NOTE: The amount of catalase may vary from one preparation to another. You should use one single preparation for all experiments. Also, catalase activity will decrease rapidly due to standing. You need to complete all experiments within two hours.

1. Cut a lettuce into quarters.
2. Weigh a quarter of the lettuce. Record its mass. Using a pair of scissors, cut it into small pieces in a large, clean mortar. Avoid losing any juice.
3. Using a 50-cm³ measuring cylinder, add deionized water to the mortar. The mass of water should be about half of that of lettuce.
4. Crush the lettuce using a pestle until it has become a fine pulp. This step may take 10 minutes.
5. Cut one side of a tea bag with a pair of scissors to make a cone. (Alternatively, cheesecloth may be used.) Put it over a filter funnel.
6. Filter the resulting lettuce suspension through the tea bag to remove solid particles. Collect the filtrate using a 250-cm³ conical flask. Squeeze the tea bag and contents to obtain more lettuce juice.
7. When filtration is done, stopper the flask immediately to minimize oxidation of catalase by air. Put the flask into an ice-water bath. Dispose of the lettuce solid residue.
8. Repeat steps 2-7 using another quarter of lettuce to get a total of about 120 cm³ of catalase extract.

(B) Measurement of the rate of the catalase-catalyzed reaction

1. Pipette 4.0 cm³ of 0.44 M H₂O₂ into a small weighing bottle (with a magnetic clip at the bottom).
2. Pipette 20.0 cm³ of the lettuce extract into a 50 cm³ wide-necked conical flask.
3. With the aid of a pair of long forceps, carefully put the weighing bottle into the conical flask. Avoid splashing H₂O₂ about.
4. Prepare a water bath at 20 °C. Set up the apparatus as shown in Figure 1. CAUTION: Make sure that all connections are gas tight. Wait at least 10 min to allow time for the chemicals to come to the same temperature as the water. Record the exact temperature.
5. Tilt the weighing bottle with a magnet to start the enzymatic reaction. Start the stopwatch immediately. Vigorously shake the contents at a rate of about 3 shakes per second. Record the volumes of oxygen gas collected after 10, 15, 20, 25 and 30 seconds.
6. Repeat steps 4-5 at four different temperatures below the optimum temperature of catalase (e.g., 25, 30, 35, 39 °C).
(C) Measurement of the rate of the iodide-catalyzed reaction

1. Pipette 5.0 cm$^3$ of 0.1 M KI solution into a small weighing bottle (with a magnetic clip at the bottom).

2. Pipette 20.0 cm$^3$ of 0.22 M H$_2$O$_2$ into a 50-cm$^3$ wide-necked conical flask.

3. Prepare a water bath at 30 °C. Set up the apparatus as shown in Figure 2. CAUTION: Make sure that all connections are gas tight. Wait at least 10 min to allow time for the chemicals to come to the same temperature as the water. Record the exact temperature.
4. Tilt the weighing bottle with a magnet to start the chemical reaction. Start the stopwatch immediately. Vigorously shake the contents at a rate of about 3 shakes per second. Record the times necessary to produce 2, 3, 4, 5, 6, 7 and 8 cm$^3$ of oxygen.

5. Repeat steps 1-4 at 35, 40, 45 and 50 °C.

NOTES:
To get consistent results in this inquiry-based lab, there are several important points to keep in mind.

- The state of ripeness of lettuce will influence catalase activity and the amount of catalase varies from tissue to tissue. Extraction of catalase should only be done with fresh tissues.

- At room temperature, catalase in the extract dissociates and loses its activity rapidly. Keep the catalase solution in an ice-water bath. Use the same catalase solution for all experiments and try to complete the lab work within two hours.

- Low temperatures should be avoided as it is difficult for the oxygen gas to escape from the reaction mixture.

- Accuracy in maintaining the temperature of the water bath is essential for consistent results. Wait for at least 10 min to allow time for the chemicals to come to the same temperature as the water bath. Measure the temperature of the reaction mixture rather than the temperature of the water bath.

- To reduce risks, the use of hydrogen peroxide solution with concentration higher than 0.44 M (i.e., 1.5%) should be avoided. Also, concentrated hydrogen peroxide solution will inactivate catalase and oxidize iodide to produce excessive iodine.

- The volumes of oxygen collected after 10, 15, 20, 25 and 30 seconds may be recorded. But reading the measuring cylinder is harder than reading the stopwatch when the reaction proceeds rapidly. So, an alternative way to collect data is to record the times necessary to produce 2, 3, 4, and so on cm$^3$ of oxygen. Students must work in pairs; one to do the timing and the other to record the volume of oxygen gas. We want data points at the beginning that lie on a straight line and thus it is not necessary to wait for the reaction to finish.

- The initial rate can be found by drawing the tangent to the volume-time curve at $t = 0$. However, this method may not work in this investigation because the first and second data points are usually unreliable due to difficulties collecting the gases. Students should try to focus on the initial segment of the volume-time curve and then draw a best straight line.

- A wide-necked 50 cm$^3$ conical flask is recommended to allow for placement of a small weighing bottle inside it. Check whether such a type of flask is available in the lab. Conical flasks larger than 100 cm$^3$ are not recommended because the total volume of catalase and hydrogen peroxide is just about 25 cm$^3$. 


Sample Data and Results

The following table shows the initial rates of the catalase-catalyzed reaction at five different temperatures.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Initial rate (cm³/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>0.46</td>
</tr>
<tr>
<td>25</td>
<td>0.49</td>
</tr>
<tr>
<td>30</td>
<td>0.63</td>
</tr>
<tr>
<td>35</td>
<td>0.64</td>
</tr>
<tr>
<td>37</td>
<td>0.73</td>
</tr>
</tbody>
</table>

Figure 3 demonstrates how the initial rate at 25 °C can be calculated from the slope of the straight line drawn through the plotted points and expressed as cm³ of oxygen evolved per second. Students may use the Excel program to draw the straight line on the plot.

Although students used a crude sample of lettuce catalase and collected oxygen over water, the points in the Arrhenius plot (Figure 4) lay quite satisfactorily on a straight line (R² = 0.94). Based on this sample Arrhenius plot, the activation energy for the catalase-catalyzed reaction is 19 kJ/mol. The teacher should avoid comparing students’ results with the literature values because the nature of catalase affects the Eₐ values. In this guided inquiry, the values obtained by students usually range from 15 – 20 kJ/mol.
The following table shows the initial rates of the iodide-catalyzed reaction at five different temperatures:

**Table 2. Initial rates of the iodide-catalyzed reaction**

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Initial rate (cm³/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>31</td>
<td>0.058</td>
</tr>
<tr>
<td>35</td>
<td>0.088</td>
</tr>
<tr>
<td>41</td>
<td>0.11</td>
</tr>
<tr>
<td>46</td>
<td>0.15</td>
</tr>
<tr>
<td>50</td>
<td>0.24</td>
</tr>
</tbody>
</table>

Students drew the straight lines based on those data points below 6 cm³ of oxygen (Figure 5). They found that the activation energy is 55 kJ/mol (Figure 6, R² = 0.97). In this guided inquiry, the values obtained by a class of Secondary 6-7 students usually range from 54 – 58 kJ/mol.
A low value of activation energy implies high efficiency of molecular collisions to form products. The factor, $e^{-E_a/RT}$, in the Arrhenius equation is the proportion of molecules with energy equal to or greater than the activation energy. Since the activation energy for the uncatalyzed decomposition of hydrogen peroxide is 75 kJ/mol (Moelwyn-Hughes, 1971), the proportion at 25 °C is equal to $e^{-75000/(8.314 \times 298)} = 6.9 \times 10^{-14}$. The relative rates of three reactions are shown in Table 3. Lowering the activation energy from 75 to 55 kJ/mol increases the rate by a factor of $10^9$, but catalase is even more efficient than iodide by a factor of $10^6$.

Table 3. Relative rates of decomposition of H$_2$O$_2$ at 25 °C

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>$E_a$ (kJ/mol)</th>
<th>Proportion of molecules with energy $\geq E_a$</th>
<th>Relative rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>None</td>
<td>75</td>
<td>$6.9 \times 10^{-14}$</td>
<td>1.0</td>
</tr>
<tr>
<td>Iodide</td>
<td>55</td>
<td>$2.3 \times 10^{-10}$</td>
<td>$3.3 \times 10^3$</td>
</tr>
<tr>
<td>Crude lettuce catalase</td>
<td>19</td>
<td>$4.7 \times 10^{-4}$</td>
<td>$6.8 \times 10^5$</td>
</tr>
</tbody>
</table>

Possible sources of error include:
1. Some oxygen gas was lost due to solubility in water in the trough.
2. Some oxygen gas dissolved in the reaction mixture and the amount of dissolved oxygen varied with temperature.
3. The crude extract of lettuce, from which we obtained catalase, may have contained other substances that can react with hydrogen peroxide to produce gases.
4. It was difficult to measure accurately the bottom of the meniscus of water inside the measuring cylinder.
5. For each trial, maintaining a constant temperature of the reaction mixture was difficult.
6. The lettuce catalase deteriorated in air.

References


What are the Concentrations of H$_3$PO$_4$ and H$_2$PO$_4^-$ in a Sample?

Phosphoric acid, H$_3$PO$_4$, is the only inorganic acid widely used in cola-flavoured soft drinks such as Coca-Cola® and Pepsi-Cola®. It can stimulate the flow of saliva in our mouth and thus provides thirst-quenching properties. Phosphoric acid not only gives an acidic taste but also acts as a buffer to control acidity. Furthermore, it helps to prevent discoloration and decay of the soft drink.

However, research has shown that children who drink at least 1.5 L per week (approximately 4.2 cans per week) of cola drinks may have a health problem called hypocalcemia (i.e., the concentration of calcium ions in blood is below normal). In the UK, for example, the use of phosphoric acid is controlled to a maximum of 700 mg/L in soft drinks by law.

In solution, phosphoric acid is a triprotic acid. Its dissociation takes place in three stages:

\[
\begin{align*}
H_3PO_4 & \rightleftharpoons H^+ + H_2PO_4^- & K_{a1} &= 7.5 \times 10^{-3} \\
H_2PO_4^- & \rightleftharpoons H^+ + HPO_4^{2-} & K_{a2} &= 6.2 \times 10^{-8} \\
HPO_4^{2-} & \rightleftharpoons H^+ + PO_4^{3-} & K_{a3} &= 4.8 \times 10^{-13}
\end{align*}
\]

Imagine that you work as a chemist in a cola drink company. You want to determine the concentrations of H$_3$PO$_4$ molecules and H$_2$PO$_4^-$ ions in a sample of commercial solution of phosphoric acid by titration. Unfortunately, your pH meter has been damaged. So, you want to use acid-base indicators instead. Submit your plan by (date).

You will be presenting on ____________ (date) in front of a group of chemists. You will have 10 minutes to present your plan, followed by 10 minutes in which you will be expected to respond to queries. Your presentation needs to answer the following questions:

- Which acid-base indicators are appropriate for the titration? Why?
- How can you find out the concentrations of H$_3$PO$_4$ molecules and H$_2$PO$_4^-$ ions?
- Will your proposed procedure be feasible and safe?

NOTE: After reviewing your experimental design, the teacher will discuss any safety precautions that are specific to your design. Obtain teacher approval before beginning any lab work.
**Assessment Criteria for Planning the Phosphoric Acid Investigation**

Names of Students: ___________________________
______________________________
______________________________
______________________________

Date: ___________________________

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. The choice of indicators is explained clearly.</td>
<td>____</td>
<td>____</td>
<td>____</td>
</tr>
<tr>
<td>2. Calculations of the concentrations of H$_3$PO$_4$ molecules and H$_2$PO$_4^-$ ions in the sample are shown correctly.</td>
<td>____</td>
<td>____</td>
<td>____</td>
</tr>
<tr>
<td>3. Suitable choice of chemicals and apparatus.</td>
<td>____</td>
<td>____</td>
<td>____</td>
</tr>
<tr>
<td>4. Chemicals and apparatus are easily available.</td>
<td>____</td>
<td>____</td>
<td>____</td>
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<tr>
<td>5. Measurement errors are minimized by appropriate procedures or apparatus.</td>
<td>____</td>
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</tr>
<tr>
<td>6. Specific hazardous chemicals are identified.</td>
<td>____</td>
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</tr>
<tr>
<td>7. Steps are included to reduce risks.</td>
<td>____</td>
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</tr>
<tr>
<td>8. No invalid assumptions are made.</td>
<td>____</td>
<td>____</td>
<td>____</td>
</tr>
<tr>
<td>9. The methods are clear enough to be followed by other students.</td>
<td>____</td>
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</tr>
<tr>
<td>10. Reagents that need accurate measurement are identified.</td>
<td>____</td>
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<td>____</td>
</tr>
<tr>
<td>11. Lab trials are stated.</td>
<td>____</td>
<td>____</td>
<td>____</td>
</tr>
<tr>
<td>12. Repeats are stated.</td>
<td>____</td>
<td>____</td>
<td>____</td>
</tr>
<tr>
<td>13. Chemistry vocabulary is used correctly.</td>
<td>____</td>
<td>____</td>
<td>____</td>
</tr>
<tr>
<td>14. Limitations of the experimental design are described.</td>
<td>____</td>
<td>____</td>
<td>____</td>
</tr>
</tbody>
</table>

**TOTAL: ____  ____  ____**
Curriculum Links

- Polyprotic acids and their salts
- Acid-base titration
- pH values at equivalence points
- Selection of acid-base indicators

Background Information

1. Phosphoric acid is a triprotic acid (three ionizable hydrogens). It plays an important part in the formulation of cola-flavoured soft drinks (Ashurst, 1998). This inquiry-based experiment is suitable for S6-7 chemistry students. Phosphoric acid may not be found on the label of a can of Coca-Cola®. They specify “Acidity Regulator (E338)”, which is actually phosphoric acid.

2. Research has revealed that consumption of soft drinks with phosphoric acid is a risk factor for the development of hypocalcemia (Fernando, Martha & Evangelina, 1999).

3. This investigation is an excellent opportunity for S6-7 students to apply what they have learned about acid-base equilibrium. Their challenge is to propose two acid-base indicators for titration so that the concentrations of both $H_3PO_4$ molecules and $H_2PO_4^-$ ions can be determined accurately. In typical cookbook-style labs, decisions such as the choice of indicators and the steps of data analysis are made for students by the textbook writers or instructor.

4. Phosphoric acid is a stronger acid than ethanoic acid but weaker than hydrochloric acid and sulphuric acid. Different textbook writers often cite different $K_a$ values. According to Wilbraham, Staley, Matta and Waterman (2005), the $K_a$ values at 25 °C are

   $$
   H_3PO_4 \rightleftharpoons H^+ + H_2PO_4^- \quad K_{a1} = 7.5 \times 10^{-3}
   $$

   $$
   H_2PO_4^- \rightleftharpoons H^+ + HPO_4^{2-} \quad K_{a2} = 6.2 \times 10^{-8}
   $$

   $$
   HPO_4^{2-} \rightleftharpoons H^+ + PO_4^{3-} \quad K_{a3} = 4.8 \times 10^{-13}
   $$

5. The concentration of phosphoric acid in a real sample of cola drink such as Coca-Cola® and Pepsi-Cola® can be determined if there are enough colorimeters for student use in your lab. Students have to make absorbance measurements at 400 nm of the yellow chromophore formed when phosphate reacts with ammonium molybdate and ammonium metavanadate. For example, Murphy (1983) reported that the concentration of phosphoric acid in a decarbonated sample of Pepsi-Cola® is 0.00506 M. Other colour reagents are also available for colorimetric analysis (Lozano-Calero, Martin-Palomeque & Madueno-Loriguillo, 1996a; Rodgers & Koether, 2005).

6. If your school has adequate number of pH meters or data loggers for group experiments, students may monitor pH values as they titrate Coca-Cola® or Pepsi-Cola® with NaOH solution. See Gipps (2001) for information about the use of data loggers. Note that acid-base indicators cannot be used to determine the concentration of phosphoric acid in cola drinks because the intense brown colour of the drinks will obscure the colour change. Our trials found that it is very difficult to decolourize Coca-Cola® or Pepsi-Cola® using activated charcoal. Unfortunately, clear colas such as Crystal Pepsi® are not available in the Hong Kong market.

7. However, we do not want Secondary 6-7 students to use pH meter (see the investigative
problem) because they may determine the pH of the cola drink and then simply find out the concentration of \( \text{H}_3\text{PO}_4 \) by calculation using the first dissociation constant. Thus, the investigation would become less challenging and interesting. Below is a sample calculation:

According to Murphy (1983), the pH of undiluted decarbonated Pepsi-Cola\(^\circ\) is 2.46, so
\[
\text{pH} = 2.46 = -\log[\text{H}^+]
\]

\[
[\text{H}^+] = 3.47 \times 10^{-3} \text{ M}
\]

The first ionization step is \( \text{H}_3\text{PO}_4 \rightleftharpoons \text{H}^+ + \text{H}_2\text{PO}_4^- \).

\( K_{a1} = 7.5 \times 10^{-3} \). Let \( c \) be the concentration (mol/L) of phosphoric acid. Substitution into the expression for \( K_{a1} \) gives

\[
K_{a1} = \frac{[\text{H}^+][\text{H}_2\text{PO}_4^-]}{[\text{H}_3\text{PO}_4]} = \frac{(3.47 \times 10^{-3})(3.47 \times 10^{-3})}{(c - 3.47 \times 10^{-3})} = 7.5 \times 10^{-3}
\]

\[c = 5.07 \times 10^{-3}\]

8. Phosphoric acid in colas can be separated by using an anionic exchange resin (Lozano-Calero, Martin-Palomeque & Madueno-Loriguillo, 1996b). Bello and Gonzalez (1996) used nonsuppressed ion chromatography to determine the phosphate contents in seven cola beverages.

9. \( \text{H}_2\text{PO}_4^- \) and \( \text{HPO}_4^{2-} \) ions possess both acidic and basic properties. That is, they are amphoteric. In titrating \( \text{H}_3\text{PO}_4 \) with NaOH, a number of “buffer situations” are created (Marcus, Sienko & Plane, 1999). The “maximum buffer” point occurs when the pH of the solution equals the pK\(_a\) (i.e., 2.12, 7.21, and 12.32).

10. To reduce risks, use low concentrations of reagents for titration (e.g., 0.04 M NaOH and 0.03 M \( \text{H}_3\text{PO}_4 \)). Do NOT buy phosphoric acid crystals because they are difficult to handle. Phosphoric acid is commercially available in aqueous solutions of concentrations 75% – 90%. For this investigation, the teacher may prepare a \( \text{H}_3\text{PO}_4 \) sample by diluting a commercial solution of phosphoric acid and then add NaOH to increase the concentration of \( \text{H}_2\text{PO}_4^- \) ions in the prepared sample.

**Students’ Misconceptions and Difficulties**

1. Some students have the misconceptions that only \( \text{H}_3\text{PO}_4 \) molecules are present in the sample since phosphoric acid is a weak acid. In fact, at any given pH, \( \text{H}_3\text{PO}_4, \text{H}_2\text{PO}_4^-, \text{HPO}_4^{2-}, \) and \( \text{PO}_4^{3-} \) all exist together in equilibrium. The pH determines the concentration of each species.

2. Students generally fail to understand that two distinct end points can be observed in a titration of phosphoric acid since the first and second dissociation constants for phosphoric acid are about a factor of \( 10^5 \) apart. In other words, the second neutralization reaction will not occur until the first neutralization reaction is completed. In general, if the difference between successive ionization constants is at least \( 10^4 \), each proton can be
differentiated in a titration, that is, each is titrated separately to give stepwise pH breaks in the titration curve (Christian, 2004). The ionization of the third proton for H$_3$PO$_4$ is too small to show a pH break in the titration curve.

3. To select the appropriate indicators, students have to calculate the theoretical values for the pH of the solution at the first and second equivalence points. For the first equivalence point, the total [H$^+$] is equal to the [H$^+$] produced by the ionization of H$_2$PO$_4^-$ ions and H$_2$O minus the [H$^+$] consumed in the formation of H$_3$PO$_4$. For the second equivalence point, the total [H$^+$] is equal to the [H$^+$] produced by the ionization of HPO$_4^{2-}$ ions and H$_2$O minus the [H$^+$] consumed in the formation of H$_2$PO$_4^-$ ions. However, these calculations are beyond Secondary 6-7 students. See Fischer and Peters (1968) for details.

4. For Secondary 6-7 students, an approximate method of calculating the pH values at the first and second equivalence points is acceptable. The first approximate method of calculating the pH at the first equivalence point is to take an average of the pK$_{a1}$ and pK$_{a2}$ values (Christian, 2004; Fischer & Peters, 1968; Marcus, Sienko & Plane, 1999). Similarly, the pH at the second equivalence point is approximately equal to the average of pK$_{a2}$ and pK$_{a3}$ values. The pH values are shown below.

At the first equivalence point, \[ pH = \frac{1}{2}(pK_{a1} + pK_{a2}) = \frac{1}{2}(2.1 + 7.2) = 4.7 \]

At the second equivalence point, \[ pH = \frac{1}{2}(pK_{a2} + pK_{a3}) = \frac{1}{2}(7.2 + 12.3) = 9.8 \]

5. The second approximate method is to simplify the calculations by assuming that the pH at the first equivalence point is mainly determined by the ionization of H$_2$PO$_4^-$ ions (i.e., H$_2$PO$_4^-$ $\rightleftharpoons$ H$^+$ + HPO$_4^{2-}$) while the pH at the second equivalence point is mainly governed by the proton transfer from water to HPO$_4^{2-}$ ions (i.e., HPO$_4^{2-}$ + H$_2$O $\rightleftharpoons$ H$_2$PO$_4^-$ + OH$^-$). Below is an example.

Assume that we titrate 25.0 cm$^3$ of 0.0300 M H$_3$PO$_4$ with 0.0400 M NaOH. The first equivalence point is reached when we have added 18.75 cm$^3$ of NaOH(aq). The concentration of H$_2$PO$_4^-$ ions will be 0.0171 M. For the following equilibrium, K$_{a2}$ = 6.2 x 10$^{-8}$.

H$_2$PO$_4^-$ $\rightleftharpoons$ H$^+$ + HPO$_4^{2-}$

Let y be the concentration of hydrogen ions. Substitution into the expression for K$_{a2}$ gives

\[ K_{a2} = \frac{[H^+][HPO_4^{2-}]}{[H_2PO_4^-]} = \frac{(y)(y)}{(0.0171 - y)} = 6.2x10^{-8} \]

\[ y = 3.26x10^{-5} \]

\[ pH = -\log y = -\log(3.26x10^{-5}) = 4.49 \]

The second equivalence point is reached if we have added a total of 2 x 18.75 = 37.50 cm$^3$ of 0.0400 M NaOH. The concentration of HPO$_4^{2-}$ ions will be 0.0120 M. Consider the equilibrium HPO$_4^{2-}$ + H$_2$O $\rightleftharpoons$ H$_2$PO$_4^-$ + OH$^-$.
\[ K_b = \frac{K_w}{K_{a2}} = \frac{1.0 \times 10^{-14}}{6.2 \times 10^{-8}} = 1.61 \times 10^{-7} \]

Let \( z \) be the concentration of hydroxide ions. Substitution into the expression for \( K_b \) gives

\[ K_b = \frac{(z)(z)}{0.0120} = 1.61 \times 10^{-7} \]

\[ z = 4.4 \times 10^{-7} \]

\[ pOH = -\log[OH^-] = -\log 4.4 \times 10^{-7} = 4.4 \]

\[ pH = 14.0 - 4.4 = 9.6 \]

6. Some students have difficulties selecting the appropriate indicators based on the theoretical pH values at the first and second equivalence points. Information about acid-base indicators can be obtained from textbooks or the Internet (e.g., http://ifs.massey.ac.nz/resources/chemistry/dissociation/indicators.htm). Ideally, an indicator should change colour at the equivalence point in a titration. But in practice, this is unnecessary. An indicator beginning and ending its colour change anywhere on the rapid-rise portion of the titration curve will be fine. Thus, methyl yellow (N, N-dimethyl-p-phenylazoaniline or p-dimethylaminoazobenzene) is suitable for the titration of phosphoric acid to the first stage because it changes from red to yellow at a pH of 2.8 to 4.4. Methyl orange may also be used. For the second stage, o-cresolphthalein may be used as indicator as it changes from colourless to violet-red at a pH of 8.2 to 9.8. Phenolphthalein can also act as indicator.

7. A lot of students believe that they must use the two indicators (e.g., methyl yellow and o-cresolphthalein) in a single titration. Consequently, they find it difficult to observe the colour change at the second end point.

8. Few students include lab trials in their experimental plans to estimate the amounts of chemicals to be used.
Sample Procedure

**Materials (per group)**

- Safety goggles (1 pair per student)
- Rubber gloves (1 pair per student)
- Apron or lab coat (1 per student)
- Phosphoric acid of “unknown” molarity (Pipette 2.0 cm$^3$ of 85% H$_3$PO$_4$ into a 1-L volumetric flask. Add 75.0 cm$^3$ of 0.040 M NaOH. Mix and bring to volume with deionized water)
- 0.0400 M sodium hydroxide (standardized)
- 0.1% methyl yellow indicator (Dissolve 0.1 g of methyl yellow in a mixture of 90 cm$^3$ absolute ethanol and 10 cm$^3$ water)
- 0.04% o-cresolphthalein indicator (Dissolve 0.04 g of o-cresolphthaein in 100 cm$^3$ of absolute ethanol)
- Deionized or distilled water
- 25-cm$^3$ bulb pipette
- Pipette filler
- Wash bottle
- 250-cm$^3$ beakers (2)
- 250-cm$^3$ conical flasks (3)
- Burette
- Small funnel for filling burette
- White tile
- Clamp stands, bosses and clamps

**Experimental Details**

(A) Titration using methyl yellow indicator

1. Pipette 25.0 cm$^3$ of the sample phosphoric acid solution into a clean 250-cm$^3$ conical flask.
2. Add 2 drops of methyl yellow indicator into the flask and mix the contents well. The phosphoric acid solution should turn red.
3. Titrate the phosphoric acid solution with 0.0400 M NaOH solution. Look through the solution on to the white tile. The end point has been reached when the solution turns from red to yellow. Record the volume of NaOH solution used in an appropriate format.
4. Repeat the titration to obtain consistent results.
(B) Titration using o-cresolphthalein

1. Pipette 25.0 cm$^3$ of the sample phosphoric acid solution into a clean 250-cm$^3$ conical flask.

2. Add 5 drops of o-cresolphthalein indicator into the flask and mix the contents well. The phosphoric acid solution should remain colourless.

3. Titrate the phosphoric acid solution with 0.0400 M NaOH solution. Look through the solution on to the white tile. The end point has been reached when the solution turns from colourless to violet-red. Record the volume of NaOH solution used in an appropriate format.

4. Repeat the titration to obtain consistent results.

NOTES:

- Let V$_{eq1}$ be the volume of NaOH added using methyl yellow indicator and V$_{eq2}$ be the volume of NaOH using o-cresolphthalein indicator. To check students’ understanding of the chemical principle of the method, you may ask students whether it is possible that V$_{eq2}$ is less than 2 x V$_{eq1}$ in this phosphoric acid investigation.

- Students are generally weak in titration techniques. To get consistent results, they must master the skills in the use of pipette and burette. For example, check whether your students know how to add less than one full drop from a burette.

**Sample Data and Results**

Concentration of the NaOH solution used = 0.0381 M

<table>
<thead>
<tr>
<th>Titration results using methyl yellow indicator:</th>
<th>1 (Trial)</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final burette reading (cm$^3$)</td>
<td>21.25</td>
<td>25.30</td>
<td>21.80</td>
</tr>
<tr>
<td>Initial burette reading (cm$^3$)</td>
<td>4.60</td>
<td>8.70</td>
<td>5.20</td>
</tr>
<tr>
<td>Volume of NaOH added (cm$^3$)</td>
<td>16.65</td>
<td>16.60</td>
<td>16.60</td>
</tr>
</tbody>
</table>

Average titre = 16.60 cm$^3$

<table>
<thead>
<tr>
<th>Titration results using o-cresolphthalein indicator:</th>
<th>1 (Trial)</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final burette reading (cm$^3$)</td>
<td>46.35</td>
<td>37.81</td>
<td>38.55</td>
</tr>
<tr>
<td>Initial burette reading (cm$^3$)</td>
<td>10.25</td>
<td>2.35</td>
<td>3.10</td>
</tr>
<tr>
<td>Volume of NaOH added (cm$^3$)</td>
<td>36.10</td>
<td>35.46</td>
<td>35.45</td>
</tr>
</tbody>
</table>

Average titre = (35.46 + 35.45)/2 = 35.46 cm$^3$
For methyl yellow indicator, the neutralization reaction that takes place during titration is

\[ \text{H}_3\text{PO}_4 + \text{NaOH} \rightarrow \text{NaH}_2\text{PO}_4 + \text{H}_2\text{O} \]

Thus, the concentration of \( \text{H}_3\text{PO}_4 \) in the sample = \( \frac{0.0381 \times 16.60}{25.0} \) = 0.0253\( M \)

For o-cresolphthalein indicator, the neutralization reaction that takes place during titration is

\[ \text{NaH}_2\text{PO}_4 + \text{NaOH} \rightarrow \text{Na}_2\text{HPO}_4 + \text{H}_2\text{O} \]

Therefore, the concentration of \( \text{H}_2\text{PO}_4^- \) in the sample is

\[ \frac{0.0381 \times (35.46 - 2 \times 16.60)}{25.0} = 0.00344\ M \]

Possible sources of error include:

- During the titration, it was difficult to decide exactly when the two indicators changed colour.
- Glassware such as pipette, conical flask and burette may not have been cleaned thoroughly. For example, drops of liquid were left hanging from the walls of the burette.
- We may not have handled the pipette properly. We are not sure whether we used exactly 25.0 cm\(^3\) of the phosphoric acid sample in each titration.

References


**How Much Ethanol Does Red Wine Contain?**

Imagine that the Consumer Council has received a number of complaints about fake red wines. Your group is employed by the Consumer Council as analytical chemists to test wine authenticity.

Ethanol content in red wines can be determined by physical and chemical methods. You will be provided with a bottle of red wine for checking brand authenticity. Your task is to plan and carry out an investigation to determine its ethanol content based on the chemical properties of ethanol molecules. Submit the plan as group work by _____________ (date).

Your group will be presenting on _____________ (date) in front of a group of chemists. You will have 10 minutes to present your plan, followed by 10 minutes in which you will be expected to respond to queries. Your presentation needs to answer the following questions:

- How can you remove the red pigments in wine?
- What reagent can be used to react with ethanol? Why?
- How can you check the accuracy of your method?
- Will your proposed procedure be feasible and safe?

NOTE: After reviewing your experimental design, the teacher will discuss any safety precautions that are specific to your design. Obtain teacher approval before beginning any lab work.
## Assessment Criteria for Planning the Red Wine Investigation

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Proper procedure has been included to remove the red pigments.</td>
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<tr>
<td>2. Proper chemical method is included to determine the amount of ethanol in wine</td>
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<tr>
<td>3. Suitable choice of chemicals and apparatus.</td>
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<tr>
<td>4. Chemicals and apparatus are easily available.</td>
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<tr>
<td>5. Measurement errors are minimized by appropriate procedures or apparatus.</td>
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<td>6. Specific hazardous chemicals are identified.</td>
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<td>7. Steps are included to reduce risks.</td>
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<td>8. No invalid assumptions are made.</td>
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<tr>
<td>9. The methods are clear enough to be followed by other students.</td>
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<tr>
<td>10. Reagents that need accurate measurement are identified.</td>
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<tr>
<td>11. Lab trials are stated.</td>
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<tr>
<td>12. Repeats are stated.</td>
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<tr>
<td>13. Chemistry vocabulary is used correctly.</td>
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<tr>
<td>14. Limitations of the experimental design are described.</td>
<td></td>
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</tbody>
</table>

TOTAL: 84
Curriculum Links

- Chemistry of alcohols
- Redox titration
- Back titration

Background Information

1. Analysis of sulphur dioxide content in white wine is a common cookbook-style lab done in Hong Kong. However, students seldom have an opportunity to plan an experiment to determine the ethanol content in red wine. This investigation is suitable for S6-7 chemistry students. The sample procedure was adapted from Denby (1998). The use of dichromate was also mentioned by Newton (2003).

2. In this investigation, red wine is better than white wine because we can provide students with an opportunity to use activated charcoal to remove colours from red wine. Adsorption of pigments by activated charcoal depends upon factors such as the origin and total adsorptive surface of charcoal (Singleton & Draper, 1962). In the sample procedure, we suggest that about 10 g of activated charcoal be added, but the teacher should test the activated charcoal available in his or her school and adjust the mass accordingly.

3. It is important to note that dichromate is very toxic with concentration of 0.2 M or more and is toxic with concentration of 0.003 M or more but less than 0.2 M. For this investigation, the use of dichromate solution with concentration greater than 0.1 M is not recommended.

4. For wines with ethanol content more than 20% v/v, they should be diluted appropriately. Note that % v/v is the ratio of the volume of ethanol present to the total volume of the wine.

5. The teacher should NOT allow students to oxidize the wine sample with acidified permanganate in a closed container because gases will be evolved. Never allow students to mix permanganate with concentrated sulphuric acid (see Education Department, 2002, p. 91).

6. For wines with ethanol content less than 10% v/v, about 3 cm³ of wine may be added to the 250-cm³ volumetric flask. But carbonated beverages such as beer and sparkling wines are not recommended for this investigation because it is hard to obtain accurate results.

7. The potassium dichromate solution used in this investigation contains concentrated sulphuric acid which is corrosive. The teacher should instruct students to wear safety goggles, rubber gloves and apron while performing lab work.

8. N-phenylanthranilic acid is one of the common indicators used in dichromate-iron(II) titrations (Kolthoff & Belcher, 1957; Sriramam, 1973; Syrokomskii & Stiepin, 1936). The end point is fairly sharp and the colour change can be seen more clearly if the
solutions are dilute. Other indicators are also available (Denby, 1998; Harris, 2003; Kolthoff & Belcher, 1957; Zoecklein et al., 1995).

9. Iron (II) can reduce dichromate. An acidic solution of iron(II) ions is fairly stable and atmospheric oxidation is not very rapid. But a freshly prepared iron(II) solution works best.

10. As an extension, you may ask students to compare physical and chemical methods for determining ethanol content in wines (e.g., hydrometric analysis vs titrimetric analysis using acidified dichromate). For an example of physical method, see Richardson and Chasteen (2003). Note that complete separation of an ethanol-water mixture is impossible because the liquids will form an azeotrope.

11. In the investigation, students are required to suggest how they can check the accuracy of their methods. Accuracy cannot be determined based on the % v/v of ethanol shown on the label as the wine may be a fake. If there is no time constraint, students may be allowed to conduct a physical analysis of the same red wine for comparison or to test the accuracy of their chemical method by using a known concentration of ethanol solution. Our sample procedure will tend to overestimate the % v/v of ethanol because there are other reducing agents (e.g., sulphur dioxide and ascorbic acid) in red wine but our simple procedure does not take them into account. The error will become even greater if the oxidation period is longer than 24 hours.

Students’ Misconceptions and Difficulties

1. Permanganate is a more powerful oxidizing agent than dichromate. Also, permanganate is not as toxic as dichromate. Some students suggest that permanganate can be used to oxidize ethanol in wines and the end point in titrations can be taken as the first permanent disappearance of pink colour in the solution. However, permanganate is not suitable for this investigation because the reaction between ethanol and permanganate is non-stoichiometric due to competing side-reactions. For example, partial oxidation of ethanoic acid by permanganate to form carbon dioxide may occur (Sharp, 1961). Permanganate can also react with phenolic compounds (not the simple hydroxybenzene), sugars, and tartaric acid in wines (Zoecklein et al., 1995).

2. Many students have the misconception that acidified dichromate cannot oxidize ethanol to form ethanoic acid unless the reaction mixture is heated under reflux. Our trials indicated that the oxidation of ethanol by acidified dichromate at room temperature is almost completed after 24 hours. To save lab time, the oxidation of ethanol by acidified dichromate may be speeded up by warming the reaction mixture in a water bath at 60-65 °C for 30 minutes CAUTION: Do NOT warm the mixture in a closed system. Then, cool down the mixture to room temperature and carry out the back titration. But our trials found that this procedure tend to underestimate the ethanol content due to loss of ethanol by evaporation.

3. Some students may suggest that the unreacted dichromate ions can react with an excess of iodide ions to produce iodine. Then, standard sodium thiosulphate solution may be used to titrate the iodine using starch as indicator. However, our trials found that it is
difficult to obtain accurate results, probably due to the low solubility of iodine in the solution.

4. Some students propose that the unreacted dichromate ions can be back titrated with iron(II) solution. But they do not know why lab manuals recommend the use of iron(II) ammonium sulphate rather than iron(II) sulphate. Actually, iron(II) ammonium sulphate is better than iron(II) sulphate because the double salt has higher purity (Kolthoff & Belcher, 1957).
Sample Procedure

IMPORTANT: This sample procedure is for teacher information only. It should not be given to students as a cookbook-style experiment. Risk assessments should be done in advance by the teacher. Obtain MSDS information on all hazardous chemicals involved. Label chemicals with the appropriate safety hazard warning labels.

Materials (per group)

- Safety goggles (1 pair per student)
- Gloves (1 pair per student)
- Apron or lab coat (1 per student)
- Red wine (about 50 cm³, 10 – 15% v/v ethanol works best)
- 100 cm³ of 0.1000 M potassium dichromate solution
- 200 cm³ of 0.0300 M iron(II) ammonium sulphate solution
- N-phenylanthranilic acid indicator
- Deionized water
- 250-cm³ volumetric flask
- 25-cm³ bulb pipette
- 10-cm³ bulb or graduated pipette
- 5-cm³ graduated pipette
- 100-cm³ measuring cylinder
- Pipette filler
- Wash bottle
- 100-cm³ conical flasks (3)
- Burette
- Filter funnel
- Small funnel for filling burette
- White tile
- Stand and clamp
- 100-cm³ beakers (3)
- Droppers
- Spatulas
- Access to activated charcoal powder and filter paper (grade 1 or 2)

Experimental Details

(A) Pre-lab to be done by technician

1. Preparation of 0.1000 M potassium dichromate solution: In a 2-L volumetric flask, dissolve 58.8368 g of analytical reagent grade K₂Cr₂O₇ in approximately 1,000 cm³ of deionized water. Carefully add 320 cm³ of concentrated (18 M) H₂SO₄. Mix, cool, and bring to volume with deionized water. CAUTION: potassium dichromate solution is toxic and concentrated H₂SO₄ is corrosive.

2. Preparation of 0.0300 M iron(II) ammonium sulphate solution: In a 2-L volumetric flask, dissolve 23.5206 g of analytical reagent grade FeSO₄(NH₄)₂SO₄·6H₂O in approximately
1,500 cm$^3$ of deionized water. Carefully add 50 cm$^3$ of concentrated (18 M) H$_2$SO$_4$. Mix, cool, and bring to volume with deionized water. **CAUTION:** concentrated H$_2$SO$_4$ is corrosive.

3. Preparation of N-phenylantranilic acid as redox indicator: Dissolve 0.1 g of N-phenylantranilic acid in 5 cm$^3$ of 0.1 M sodium hydroxide solution and make up to 100 cm$^3$ with deionized water. This may produce a murky white suspension. Transfer to convenient-sized dropper bottles.

(B) Procedures to be followed by students

**Day 1: Decolourization of red wine and oxidation of ethanol**

1. Pour about 30 cm$^3$ of red wine sample into a 100-cm$^3$ beaker and add about 10 g of activated charcoal powder into it.
2. Mix and stir the mixture for about 10 minutes.
3. Filter the mixture using a funnel and grade 2 filter paper (if you don’t have grade 2 filter paper, you may set up the filtration system by putting two sheets of grade 1 filter paper together). Collect about 5 cm$^3$ of the colourless or pale pink filtrate. Repeat the filtration process if the filtrate still contains charcoal.
4. When decolourisation is complete, pipette 2.0 cm$^3$ of the filtrate into a 250-cm$^3$ volumetric flask and add about 100 cm$^3$ of deionized water. Then using another pipette, carefully add 50.0 cm$^3$ of 0.1000 M acidified potassium dichromate solution into the flask (CAUTION: The acidified potassium dichromate solution contains corrosive concentrated H$_2$SO$_4$ and dichromate is toxic). Bring to volume with deionized water.
5. Press the stopper in position and invert the volumetric flask several times to mix the contents well. Leave overnight for the oxidation of the ethanol in the flask to proceed to completion.

**Day 2: Titration**

1. Pipette 10.0 cm$^3$ of the ethanol/dichromate solution into a clean 100-cm$^3$ conical flask.
2. Add 5 drops of N-phenylantranilic acid into the flask as indicator and mix the contents well. The mixture is purple-brown in colour.
3. Titrate the mixture with 0.0300 M iron(II) ammonium sulphate solution. The end point has been reached when the mixture just turns from purple-brown to green.
   **NOTE:** Dispose of your titrated solution as directed by your teacher.
4. Record the results in an appropriate format.
5. Repeat the titration to obtain consistent results.
6. Record the average titre.
Sample Data and Results

Red wine sample = Cavalier Rouge 2004 (with 11.5% v/v of ethanol written on the bottle, HK$30).

Concentration of the potassium dichromate solution = 0.1000 M

Concentration of the iron(II) ammonium sulphate solution = 0.0300 M

Titration Results:

<table>
<thead>
<tr>
<th></th>
<th>1 (Trial)</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final burette reading (cm$^3$)</td>
<td>20.10</td>
<td>36.80</td>
<td>42.70</td>
</tr>
<tr>
<td>Initial burette reading (cm$^3$)</td>
<td>3.30</td>
<td>20.10</td>
<td>26.00</td>
</tr>
<tr>
<td>Volume of iron(II) ammonium sulphate added (cm$^3$)</td>
<td>16.80</td>
<td>16.70</td>
<td>16.70</td>
</tr>
</tbody>
</table>

Average titre = 16.70 cm$^3$

Iron (II) reduces dichromate to form chromium (III):

$$6 \text{Fe}^{2+} + \text{Cr}_2\text{O}_7^{2-} + 14 \text{H}^+ \rightarrow 6 \text{Fe}^{3+} + 2 \text{Cr}^{3+} + 7 \text{H}_2\text{O}$$

Number of moles of Fe$^{2+}$ used in the titration = $0.0300 \times (16.70/1000) = 5.01 \times 10^{-4}$ mol

Number of moles of Cr$_2$O$_7^{2-}$ reacted with Fe$^{2+}$ in titration = $5.01 \times 10^{-4} \times \frac{1}{6} = 8.35 \times 10^{-5}$ mol

Number of moles of Cr$_2$O$_7^{2-}$ reacted with Fe$^{2+}$ in the 250 cm$^3$ volumetric flask

$= 8.35 \times 10^{-5} \times (250/10)$

$= 2.09 \times 10^{-3}$ mol

Number of moles of Cr$_2$O$_7^{2-}$ in the 250 cm$^3$ volumetric flask

$= 0.1000 \times \frac{50.0}{1000}$

$= 5.00 \times 10^{-3}$ mol

Number of moles of Cr$_2$O$_7^{2-}$ reacted with ethanol in the 250 cm$^3$ volumetric flask

$= 5.00 \times 10^{-3} - 2.09 \times 10^{-3}$

$= 2.91 \times 10^{-3}$ mol

Dichromate oxidizes ethanol to form ethanoic acid:

$$2 \text{Cr}_2\text{O}_7^{2-} + 3 \text{C}_2\text{H}_5\text{OH} + 16 \text{H}^+ \rightarrow 4 \text{Cr}^{3+} + 3 \text{CH}_3\text{COOH} + 11 \text{H}_2\text{O}$$

The number of moles of ethanol in the original 2.0 cm$^3$ wine sample

$= 2.91 \times 10^{-3} \times \frac{3}{2}$

$= 4.37 \times 10^{-3}$ mol

Molar mass of ethanol = 46.062 g mol$^{-1}$
Mass of ethanol in the red wine $= 4.37 \times 10^{-3} \times 46.062$
$= 0.201\, \text{g}$

Density of ethanol at $20^\circ\text{C} = 0.798\, \text{g cm}^{-3}$

Volume of ethanol in the original $2.0\, \text{cm}^3$ wine sample $= 0.201 / 0.798 = 0.252\, \text{cm}^3$

Thus, the volume-to-volume percentage of ethanol in the red wine “Cavalier Rouge 2004”
$= \frac{0.252\, \text{cm}^3}{2.0\, \text{cm}^3} \times 100\% = 12.6\%$.

From the above calculations, the titration results give a slightly higher percentage of ethanol content than the value (11.5%) written on the label. Students may suggest the following major sources of error:

- The red wine may have contained other reducing agents such as sulphur dioxide, ascorbic acid and phenols. So, less dichromate was available for back titration.
- The oxidation of ethanol by dichromate ions was not complete.
- It was difficult to determine when the indicator changed colour.

References
Education Department (2002). *Safety in science laboratories*. Hong Kong: Printing Department.
What is the Mass of Lithium in a Sample?

Lithium metal has been widely used to make batteries in recent years. In addition to being lighter than common dry cells, lithium batteries produce a higher voltage. They are used to power electrical appliances such as mobile phones, cameras and computers. Lithium batteries are the best alternative to nickel-cadmium rechargeable batteries because they contain fewer toxic materials and do not have a memory effect that results in loss of capacity.

Your task:

Imagine that you work as a chemist in a lithium battery company. Your boss has asked you to test a sample of lithium. Lithium reacts with water vigorously according to the following equation:

\[ 2 \text{Li}(s) + 2 \text{H}_2\text{O}(l) \rightarrow 2 \text{LiOH}(aq) + \text{H}_2(g) \]

Using this chemical reaction, plan TWO different methods to determine the percentage by mass of lithium in the sample. Submit your plan by (date).

Your group will be presenting on (date) in front of the company representatives. You will have 10 minutes to present your plan, followed by 10 minutes in which you will be expected to respond to queries. Your presentation needs to answer the following questions:

- What are your two methods?
- How do you calculate the percentage by mass of lithium?
- Which method do you think is more accurate? Why?
- Will the proposed methods be feasible and safe?

NOTE: After reviewing your procedures, the teacher will discuss any safety precautions that are specific to your experiments. Obtain teacher approval before beginning the experiments.
**Assessment Criteria for Planning the Lithium Investigation**

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
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<tr>
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<td>4. Justification for the better method is convincing.</td>
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<tr>
<td>5. Suitable choice of chemicals and apparatus.</td>
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<tr>
<td>6. Chemicals and apparatus are easily available.</td>
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<td>7. Measurement errors are minimized by appropriate procedures or apparatus.</td>
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<td>9. Steps are included to reduce risks.</td>
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<td>10. No invalid assumptions are made.</td>
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<td>12. Lab trials are stated.</td>
<td>___________</td>
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<td>14. Controls on variables are clearly stated.</td>
<td>___________</td>
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<td>___________</td>
</tr>
<tr>
<td>15. Chemistry vocabulary is used correctly.</td>
<td>___________</td>
<td>___________</td>
<td>___________</td>
</tr>
</tbody>
</table>

TOTAL: ___________  ___________  ___________
Curriculum Links

- Reaction of alkali metals with water
- The ideal gas equation
- Partial vapour pressure
- Acid-base titration

Background Information

1. Chemistry students seldom have an opportunity to compare the accuracy of two different methods when they plan scientific investigations. The purpose of this guided inquiry is to determine the mass of lithium in a sample using two methods. This inquiry was adapted from an experiment used by the Oxford, Cambridge and RSA Examinations in the UK. We require students to plan the inquiry and make their measurements accurately.

2. Allow your students enough time to decide on the methods and write up their plans. The assessment criteria for the planning component should be distributed to students together with the problem. You may adapt the criteria to meet your needs. Note that the criteria should not be too specific to give students a clue to the answer (e.g., suitable choice of an indicator for titration, a proper method to collect hydrogen gas).

3. Lithium metal is very hard to cut. Therefore, granules rather than sticks or wires should be purchased from a chemical supplier. Lithium is a silver-white metal which floats on water (Stwertka, 1998).

4. Actually, modern lithium batteries use lithium ions to make electrodes (Gomez-Romero & Casan-Pastor, 1996; Treptow, 2003; Vincent & Scrosati, 1997), but this will not adversely affect Secondary 6 students to plan the chemistry investigation. Lithium also has other high-tech applications. See Ennis (2006) for details.

5. This investigation provides Secondary 6 students an opportunity to apply the ideal gas equation \( PV = nRT \). Thus, you should not allow students to assume that the molar volume of gases is 24 dm\(^3\), irrespective of the actual lab temperature and pressure.

6. In addition, this investigation is a very good lab exercise for students to apply the titration techniques that they have learned in Secondary 4.

7. The international standard unit of pressure is Pascal (Pa). 1 Pascal = 1 newton/metre\(^2\). The unit used by the Hong Kong Observatory is hecto-Pascal (hPa).

8. Textbook writers often use other units to quantify pressure. Note that 1 atmosphere = 1.01325 \( \times 10^5 \) Pascals = 760 mm mercury = 760 torr = 14.70 pounds per square inch.

9. Store lithium by immersion in kerosene or mineral oil in a sealed glass container that is itself placed in an unbreakable, leakproof outer container. Store the containers in a cool, dry, well-ventilated, and locked location that is NOT protected by a water sprinkling system. Destroy unwanted lithium by immersing quantities no larger than 10 g in isopropyl alcohol containing no more than 10% water. For more information about the hazardous properties of lithium, see Young (2005).
10. This investigation can be used to consolidate the concept of partial pressure. If the hydrogen gas collected is moist, then students have to consider the vapour pressure of water. Below is a table showing the vapour pressure of water at different temperatures.

<table>
<thead>
<tr>
<th>Temp (°C)</th>
<th>VP (mmHg)</th>
<th>Temp (°C)</th>
<th>VP (mmHg)</th>
<th>Temp (°C)</th>
<th>VP (mmHg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>9.8</td>
<td>21</td>
<td>18.7</td>
<td>31</td>
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<td>12</td>
<td>10.5</td>
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<td>19.8</td>
<td>32</td>
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<tr>
<td>13</td>
<td>11.2</td>
<td>23</td>
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<td>33</td>
<td>37.7</td>
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<tr>
<td>14</td>
<td>12.0</td>
<td>24</td>
<td>22.4</td>
<td>34</td>
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<tr>
<td>15</td>
<td>12.8</td>
<td>25</td>
<td>23.8</td>
<td>35</td>
<td>42.2</td>
</tr>
<tr>
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<td>13.6</td>
<td>26</td>
<td>25.2</td>
<td>36</td>
<td>44.6</td>
</tr>
<tr>
<td>17</td>
<td>14.5</td>
<td>27</td>
<td>26.7</td>
<td>37</td>
<td>47.1</td>
</tr>
<tr>
<td>18</td>
<td>15.5</td>
<td>28</td>
<td>28.3</td>
<td>38</td>
<td>49.7</td>
</tr>
<tr>
<td>19</td>
<td>16.5</td>
<td>29</td>
<td>30.0</td>
<td>39</td>
<td>52.4</td>
</tr>
<tr>
<td>20</td>
<td>17.5</td>
<td>30</td>
<td>31.8</td>
<td>40</td>
<td>55.3</td>
</tr>
</tbody>
</table>


**Students’ Misconceptions and Difficulties**

1. Because kerosene or oil does not react with water, some students have the misconception that it is not necessary to remove the kerosene or oil when weighing the lithium granules.

2. Students have to think of a reliable way to determine the volume of hydrogen gas formed from the reaction. But some students believe that they need to collect pure hydrogen gas.

3. A lot of students add lithium to water directly in a flask and then determine the volume of hydrogen gas evolved. They assume that no hydrogen gas or air will be lost by closing the flask with a stopper quickly.

4. The amount of water must be in excess. But some students are not sure how to estimate the appropriate amount of water to be added to lithium.

5. Many students want to use a syringe to collect hydrogen gas. The gas syringe method does not give accurate results because there is friction between the plunger and barrel. Displacement of water inside a test-tube is also not a good choice because the volume of gas collected cannot be directly determined.

6. Some students think of two methods to determine the concentration of LiOH solution: titration and pH meter. But class sizes are large in Hong Kong and pH meters are not easily available for group experiments in school.

7. Titration is an acceptable method, but students are generally weak in procedural understanding. For example, some students do not recognize that it is important to repeat the titration to guarantee good reliability of data.

8. Some students propose to determine the mass of hydrogen gas produced by weighing the experimental set-up before and after the reaction. However, they have difficulty finding an accurate electronic balance. Also, they do not know the mass of water carried
by the hydrogen gas.

9. Some students think that lithium sinks in water and plan to collect hydrogen gas by an inverted funnel.

10. Some students suggest that a tap funnel can be used to add water to prevent loss of hydrogen or air. However, they do not recognize that the water will displace air. As a result, they forget to subtract the volume of water added from the total volume of gases collected.

11. Some students do not understand that the air inside a reaction flask will not affect the volume of hydrogen gas.

12. If students plan to measure the volume of hydrogen gas from water displacement in a measuring cylinder, then moist hydrogen gas will be collected. Many students do not take the vapour pressure of water into account when they plan this investigation.

13. To measure the volume of hydrogen gas accurately, few students recognize that they must equate the level of the water in the measuring cylinder with the water outside the cylinder.
Sample Procedure

IMPORTANT: This sample procedure is for teacher information only. It should not be given to students as a cookbook-style experiment. Risk assessments should be done in advance by the teacher. Obtain MSDS information on all hazardous chemicals involved. Label chemicals with the appropriate safety hazard warning labels.

Materials (per group)

Safety goggles (1 pair per student)
Gloves (1 pair per student)
Apron or lab coat (1 per student)
Lithium granules (store them in kerosene or oil in a Petri dish. Place the dish next to the electronic balance)
Forceps
Filter paper to remove kerosene or oil
Weighing bottle (glue a small magnetic chip at the bottom)
Magnetic bar
250-cm³ conical flask with a stopper and bent glass tubing
Rubber delivery tube
Deep trough (e.g., a 5-L distilled water container)
Deionized or distilled water
100-cm³ measuring cylinder
250-cm³ measuring cylinder with a glass plate
stand and clamp
Thermometer
50-cm³ burette
25-cm³ pipette
Pipette filler
Small funnel for filling burette
250-cm³ conical flask (2) for titration
White tile
150 cm³ of 0.10 M HCl
Phenolphthalein indicator
Access to a balance (±0.001 g) and barometer (atmospheric pressure is also available from the Hong Kong Observatory website http://www.hko.gov.hk/)
Experimental Details

Method 1

NOTE: Because hydrogen gas is generated from the experiment, **THERE SHOULD BE NO FLAMES IN THE LABORATORY AT ANY TIME.**

1. Tare your balance to the weight of a clean and dry weighing bottle (with a small magnetic chip at the bottom). Using a piece of filter paper, remove as much oil as possible from the lithium granules. Weigh between 0.070 g and 0.100 g of lithium. Record the exact mass of lithium.

2. Pour exactly 100.0 cm$^3$ of distilled water into a 250-cm$^3$ conical flask. Carefully put the weighing bottle into the conical flask. Fill a 250-cm$^3$ measuring cylinder with tap water. Set up the apparatus as shown. Make sure that all connections are air-tight.

3. Tilt the weighing bottle with a magnet to start the reaction.

4. Collect the gas evolved.

5. Make the pressure on the gases inside the measuring cylinder equal to atmospheric pressure by raising or lowering the cylinder until the water level inside is the same as that outside. Record the final volume.

6. Keep the LiOH solution in the conical flask for Method 2. Repeat the experiment to obtain another set of data.

7. Record the laboratory temperature and atmospheric pressure.
NOTES:

- A balance with at least three decimal places is best for weighing the lithium sample. The teacher may pre-weigh 0.10 g of lithium to see approximately how many granules are needed. To minimize risks, do not use more than 0.10 g of lithium. The use of two different samples of lithium for Method 1 and Method 2 is not recommended because there may be errors in weighing the lithium.

- Ideally, the 100.0 cm$^3$ of distilled water should be neutral (i.e., pH = 7). Unfortunately, distilled or deionized water is often slightly acidic. Check the pH of distilled or deionized water in your lab beforehand. Lithium may react with strongly acidic water explosively. Also, the titration results may be affected.

- The lab technician should insert the glass tubing into the rubber stoppers rather than the students. If the 250-cm$^3$ measuring cylinder is not fully filled with tap water, students must record the initial volume before they start the reaction. Remind students to clamp the 250-cm$^3$ measuring cylinder.

Method 2

1. Pipette 25.0 cm$^3$ of the LiOH solution into a 250-cm$^3$ conical flask.
2. Add a few drops of phenolphthalein indicator.
3. Titrate with 0.10 M hydrochloric acid.
4. Record the results in an appropriate format.
5. Repeat the titration to obtain consistent results.
6. Record the average titre.

Sample Data and Results

Mass of lithium used = 0.078 g
Volume of gas collected = 112 cm$^3$
Laboratory temperature = 25 °C = 298 K
Laboratory atmospheric pressure = 1.016 x 10$^5$ Pa

Titration results:

<table>
<thead>
<tr>
<th></th>
<th>1 (Trial)</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final burette reading (cm$^3$)</td>
<td>31.15</td>
<td>23.30</td>
<td>44.85</td>
</tr>
<tr>
<td>Initial burette reading (cm$^3$)</td>
<td>9.50</td>
<td>1.80</td>
<td>23.30</td>
</tr>
<tr>
<td>Volume of acid added (cm$^3$)</td>
<td>21.65</td>
<td>21.50</td>
<td>21.55</td>
</tr>
</tbody>
</table>

Average titre = 21.53 cm$^3$
Concentration of the standardized HCl(aq) = 0.102 M
For Method 1, the mass percentage can be calculated as follows:

\[ 2 \text{Li(s)} + 2 \text{H}_2\text{O(l)} \rightarrow 2 \text{LiOH(aq)} + \text{H}_2(\text{g}) \]

From data book, vapour pressure of water at 298 K = 23.8 mmHg = 3.173 x 10^3 Pa
Partial pressure of hydrogen = 1.016 x 10^5 Pa – 3.173 x 10^3 Pa = 9.843 x 10^4 Pa

Number of moles of hydrogen = \( \frac{PV}{RT} = \frac{(9.843 \times 10^4 \text{Pa})(112 \times 10^{-6} \text{m}^3)}{(8.314 \text{Pa} \cdot \text{m}^3)/(\text{mol} \cdot \text{K})(298 \text{ K})} = 0.00445 \text{ mol} \)

Number of moles of lithium = 0.00445 mol x 2 = 0.00890 mol

Mass percentage of lithium in the sample = \( \frac{0.00890 \text{ mol} \times 6.94 \text{ g mol}^{-1}}{0.078 \text{ g}} \times 100\% = 79.2\% \)

Note:
Universal gas constant
\[ R = 8.314 \frac{\text{Pa} \cdot \text{m}^3}{\text{mol} \cdot \text{K}} = 8.314 \frac{\text{kPa} \cdot \text{dm}^{-3}}{\text{mol} \cdot \text{K}} = 8.314 \frac{\text{J}}{\text{mol} \cdot \text{K}} = 0.08206 \frac{\text{L} \cdot \text{atm}}{\text{mol} \cdot \text{K}} \]

For Method 2, the mass percentage can be calculated as follows:

The balanced equation for the titration reaction is LiOH + HCl \( \rightarrow \) LiCl + H\(_2\)O

Number of moles of HCl used in the titration = \( \frac{21.53}{1000} \text{ L} \times 0.102 \text{ M} = 0.002196 \text{ mol} \)

Number of moles of LiOH in 100 mL of solution = 0.002196 mol x 4 = 0.008784 mol

Mass percentage of lithium in the sample = \( \frac{0.008784 \text{ mol} \times 6.94 \text{ g mol}^{-1}}{0.078 \text{ g}} \times 100\% = 78.2\% \)

From the above calculations, the titration method resulted in a smaller mass percentage of lithium in the sample. Some students argued that the titration method is more accurate than the collection of hydrogen gas because titration results were very consistent. They suggested the following major sources of error:

1. It was difficult to measure accurately the bottom of the meniscus of water inside the measuring cylinder.
2. After oil was removed from lithium, some granules were oxidized in air to form lithium
oxide (Li₂O). So, less hydrogen gas was collected. However, the titration results were not affected.

3. Some hydrogen gas or air may have been leaked out of the flask.

Some students also suggested that the mass percentage of lithium in the sample was underestimated due to the following reasons:

1. We were unable to remove all the oil from the lithium granules.
2. The distilled or deionized water was not neutral. So, less HCl(aq) was required to titrate the LiOH solution.
3. The LiOH solution may have absorbed carbon dioxide from the air to form lithium carbonate. (Note: Actually, this does not affect the titration results.)

References


What is the Mass of Calcium Carbonate in Eggshells?

A hen lays about 250 eggs per year. To avoid breakage of eggs before reaching market, the eggshell needs to be as strong as possible. The main ingredient of eggshells is calcium carbonate. The other ingredients include phosphorus, magnesium, sodium, potassium, zinc, manganese, iron, copper, soluble proteins, and insoluble proteins. The strength of an eggshell depends on the size, shape and organization of the inorganic and organic ingredients in the shell. Thus, there is a possibility that a thinner eggshell may be stronger than a thicker one.

Imagine that you are working in the commercial egg industry. To help your boss monitor the eggshell quality, you need to determine the percentage by mass of calcium carbonate in hen eggshells. Your task is to plan and carry out TWO methods that will allow you to measure the amount of calcium carbonate in eggshells accurately. Of the two methods you designed, discuss which you think is better and the criteria you used for selecting the better method. Submit your plan by ____________ (date).

On ____________ (date), representatives of your company will give you an opportunity to share your plan. You will have 10 minutes to present your plan, followed by 10 minutes in which you will be expected to respond to queries. Your presentation needs to answer the following questions:

- What are your two methods?
- How do you calculate the percentage by mass of calcium carbonate?
- Which method do you think is better? Why?
- Will the proposed methods be feasible and safe?

NOTE: After reviewing your procedures, the teacher will discuss any safety precautions that are specific to your experimental design. Obtain teacher approval before beginning any lab work.
### Assessment Criteria for Planning the Calcium Carbonate Investigation

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<td></td>
<td></td>
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<tr>
<td>2. Labelled drawings are used to help present the methods.</td>
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<tr>
<td>3. Calculations of the percent of CaCO₃ are correct.</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>4. Justification for the better method is convincing.</td>
<td></td>
<td></td>
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<tr>
<td>5. Suitable choice of chemicals and apparatus.</td>
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<tr>
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<td>8. Specific hazardous chemicals are identified</td>
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<td>10. No invalid assumptions are made.</td>
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<td>11. Reagents that need accurate measurement are identified.</td>
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<tr>
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<tr>
<td>15. Chemistry vocabulary is used correctly.</td>
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</tbody>
</table>

**TOTAL:** 103
Curriculum Links

- Acid-based chemistry
- Analytical chemistry
- Stoichiometry
- The ideal gas equation
- Partial vapour pressure

Background Information

1. The structure and composition of eggshells are complicated (Mikšik, Charvátová, Eckhardt & Deyl, 2003; Panheleux et al., 1999). In poultry science, nutritionists and physiologists determine the calcium content of eggshells by atomic absorption spectrophotometry. Since the shell thickness and density vary at different points throughout an eggshell, they determine the calcium content in the entire eggshell.

2. Ideally, we want chemistry students to determine the percentage by mass of calcium carbonate in eggshells accurately. Unfortunately, this goal is not practical in school. Although procedures for determining the mass of calcium carbonate in eggshells are described by a number of chemistry educators (e.g., Lechtanski, 2000; Tocci & Viehland, 1996; CUHK, EMB & HKEAA, 2004), none of those procedures can produce accurate results.

3. Chemistry students are generally unaware that experimental procedures described in textbooks may have limitations. The aim of this guided inquiry is to provide S6-7 students with an opportunity to think of two different methods to determine the mass of calcium carbonate in eggshells and identify their limitations. Since student results are bound to be inaccurate, the teacher should not assess students’ performance solely based on the accuracy of their results. Instead, this guided inquiry would be more meaningful if students are required to identify the major sources of error in each method and explain their effects on the experimental results.

4. S6-7 students usually plan to determine the percentage by mass of calcium carbonate in eggshells by reacting calcium carbonate with hydrochloric acid. The equation for the chemical reaction is

\[
\text{CaCO}_3(s) + 2 \text{HCl(aq)} \rightarrow \text{CaCl}_2(aq) + \text{H}_2\text{O(l)} + \text{CO}_2(g)
\]

However, there are at least four common methods to collect and analyze data:

- Measure the mass of eggshell and hydrochloric acid before the reaction and compare that value to the final mass of the mixture. The difference in mass is equal to the mass of carbon dioxide produced. From that, students calculate the mass of calcium carbonate present in the original eggshell.

- Measure the volume of carbon dioxide gas produced by water displacement when a known mass of eggshell reacts with excess hydrochloric acid.

- Dissolve a known mass of eggshell in hydrochloric acid. Boil the mixture to remove dissolved carbon dioxide. Determine the excess amount of hydrochloric acid by back-titration with sodium hydroxide solution.

- Add excess hydrochloric acid to a known mass of eggshell. Filter the mixture and...
dry the unreacted eggshell in an oven. Weigh the unreacted eggshell. The mass of the reacted eggshell is the mass of calcium carbonate present in the eggshell.

5. We tried out the above four methods using the same eggshell. Several runs were conducted for each method. As predicted, none of these methods is the best to determine the percent by mass of calcium carbonate in eggshells because each method has its sources of error (see Table 1).

Table 1. Comparing four methods

<table>
<thead>
<tr>
<th>Method</th>
<th>% of CaCO₃ in eggshell</th>
<th>Major source of error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measurement of the mass of CO₂ produced by</td>
<td>66 – 72</td>
<td>Some carbon dioxide gas dissolved in HCl(aq). Thus, the percentage of calcium carbonate</td>
</tr>
<tr>
<td>weighing the set-up</td>
<td></td>
<td>in the eggshell was underestimated.</td>
</tr>
<tr>
<td>Measurement of the volume of CO₂ produced</td>
<td>77 – 80</td>
<td>Some carbon dioxide gas dissolved in HCl(aq) and the water in the trough. Because less</td>
</tr>
<tr>
<td>by water displacement</td>
<td></td>
<td>carbon dioxide was collected, the percentage of calcium carbonate in the eggshell was</td>
</tr>
<tr>
<td>Back titration of excess HCl(aq) by NaOH(aq)</td>
<td>70 – 73</td>
<td>underestimated.</td>
</tr>
<tr>
<td>Measurement of the mass of the unreacted</td>
<td>91 – 94</td>
<td>We assumed that all of the soluble substances in the eggshell are calcium carbonate.</td>
</tr>
<tr>
<td>eggshell</td>
<td></td>
<td>Actually, there are other soluble proteins and inorganic components. Thus, this method</td>
</tr>
<tr>
<td></td>
<td></td>
<td>may have overestimated the percentage of calcium carbonate in the eggshell.</td>
</tr>
</tbody>
</table>

The results in Table 1 indicate that the actual mass of calcium carbonate in the eggshell sample is probably 80 – 94%. These four methods assumed that all the carbonate ions in eggshells exist as calcium carbonate. But magnesium carbonate is present in eggshells. Actually, these four methods cannot directly determine the calcium content of eggshells.

6. To minimize risks, the use of hydrochloric acid with concentration less than 2 M is recommended.

7. Lechtanski (2000) reported that white eggshells contain more calcium carbonate by mass than brown eggshells.
Students’ Misconceptions and Difficulties

1. This guided inquiry can be done by titrimetry, but few S6-7 students understand why back-titration must be used. The dissolution of eggshell and the reaction between calcium carbonate and hydrochloric acid are too slow to give a sharp end point. Ethanol may be added to increase the dissolution of eggshell.

2. Some students do not recognize that excess acid must be added to the eggshell. The experimental plan must include lab trials to estimate the appropriate amount of acid to be added.

3. Many students use different samples of eggshells when performing different experiments. This should be avoided because the thickness and density of shell vary at different points throughout the eggshell, resulting in inconsistency of data. A means to minimize sampling errors should be included in the experimental plan.

4. Some students use back-titration to determine the calcium carbonate content of the eggshell. But they assume that no carbon dioxide gas will dissolve in hydrochloric acid. Students may boil the reaction mixture to reduce solubility of carbon dioxide in hydrochloric acid.

5. Those students who want to collect carbon dioxide gas by displacement of water in an inverted measuring cylinder seldom consider the lab temperature, barometric pressure and the vapour pressure of water when they calculate the number of moles of carbon dioxide gas produced.

6. Students generally add eggshells to an acid in a flask and assume that no carbon dioxide gas or air will be lost by closing the flask with a stopper quickly. S6-7 students should take the loss of gases into account when they design their investigations.

7. Some students add hydrochloric acid to a known mass of eggshell and then collect the carbon dioxide gas formed by a syringe. This method does not give accurate results because there is friction between the plunger and barrel, particularly if plastic syringes are used.

8. Students believe that they can use a syringe to collect and weigh the carbon dioxide gas. It must be remembered that there is buoyancy of the syringe and plunger when weighed in air.
Sample Procedure

In this section, we present four methods that can be completed in a single run. Remember that students are required to propose two methods only.

**IMPORTANT:** This sample procedure is for teacher information only. It should not be given to students as a cookbook-style experiment. Risk assessments should be done in advance by the teacher. Obtain MSDS information on all hazardous chemicals involved. Label chemicals with the appropriate safety hazard warning labels.

**Materials (per group)**

- Safety goggles (1 pair per student)
- Gloves (1 pair per student)
- Apron or lab coat (1 per student)
- 1.00 M hydrochloric acid (standardized)
- 1.00 M sodium hydroxide (standardized)
- Ethanol
- Phenolphthalein indicator
- Deionized or distilled water
- 250-cm³ conical flask (3)
- Weighing bottle (glue a magnetic clip at the bottom)
- Magnet bar
- 10-cm³ measuring cylinder
- 100-cm³ measuring cylinder
- 25-cm³ bulb pipette
- Pipette filler
- 50-cm³ burette
- Wash bottle
- Small funnel for filling burette
- stand and clamp
- White tile
- Trough
- Delivery tube
- Filter paper
- Büchner funnel
- Suction flask
- Suction pump
- Spatula
- Hot plate
- Access to a balance (±0.001 g), oven, oven mitts and a desiccator
Experimental Details

(A) Preparation of eggshell powder

1. Remove the white and the yolk from an egg to leave a clean shell. Immers the shell in a beaker of water for about 5 minutes. Then remove all the membranes from the inside of the shell. Wash the shell with distilled water. Place all of the shell in a beaker and dry it in an oven at 110 °C for about 20 minutes.

2. Remove the eggshell and beaker from the oven. Cool them to room temperature in a desiccator.

3. Grind the eggshell to a very fine powder using a mortar and pestle.

(B) Determination of CaCO$_3$ content in eggshell

1. Pipette 25.0 cm$^3$ of 1.00 M HCl solution into a 250-cm$^3$ conical flask.

2. Add 5 cm$^3$ of ethanol into the HCl solution.

3. Using a dry weighing bottle, weigh about 0.300 g of eggshell powder. Record the exact mass.

4. Carefully put the weighing bottle into the conical flask. Measure the total mass of the conical flask, weighing bottle, eggshell powder, ethanol, and HCl solution. Record the exact mass.
5. Setup the apparatus as shown below.

6. Tilt the weighing bottle with a magnet to start the chemical reaction.

7. Shake the flask gently and collect the gas produced.

8. When the reaction is completed, make the pressure on the gas inside the measuring cylinder equal to atmospheric pressure by raising or lowering the cylinder until the water level inside is the same as that outside. Record the final volume, the lab temperature and pressure.

9. Record the total mass of the conical flask, weighing bottle and the resulting mixture.

10. Boil the resulting mixture gently for about 5 minutes over a hot plate.

   **CAUTION:** (a) The mixture contains ethanol which is extremely flammable. Use a hot plate rather than a Bunsen burner. (b) The mixture contains hydrochloric acid. Do not boil the mixture to dryness.

11. Allow the mixture to cool to room temperature.

12. Add a few drops of phenolphthalein indicator into the mixture.

13. Titrate the mixture with 1.00 M NaOH solution until a light pink colour appears. Record the volume of NaOH solution used.

14. Dry a piece of filter paper in an oven for 20 minutes. Then measure the mass of the dried filter paper. Set up the apparatus as shown below for suction filtration. Make the filter paper wet with distilled water. Turn on the suction pump. Pour the mixture from the reaction flask into the filter paper and allow the filtrate to pass into the suction flask. Use a spatula to scrape the unreacted eggshell from the reaction flask into the filter paper.
15. Rinse the reaction flask, weighing bottle, spatula and unreacted eggshell with distilled water and allow the water to pass into the suction flask.

16. Dry the filter paper (with unreacted eggshell) in an oven at 110 °C for about 20 minutes. After drying, measure the mass of both filter paper and unreacted eggshell.

17. Repeat Steps 1-16 to obtain consistent results.

**Sample Data and Results**

Concentration of HCl = 0.964M  
Concentration of NaOH = 0.980M  
Lab temperature = 25 °C = 298 K  
Atmospheric pressure = 1.008 x 10^5 Pa (obtained from the Hong Kong Observatory website http://www.hko.gov.hk/)  
From data book, vapour pressure of water at 25 °C = 23.8 mm Hg = 3.173 x 10^3 Pa  
Partial pressure of CO₂ = 1.008 x 10^5 Pa – 3.173 x 10^3 Pa = 9.763 x 10^4 Pa  
Mass of eggshell used = 0.299 g

Tables 2-5 display one set of data and results. As explained in the background information section, none of the four methods can yield accurate results. The actual mass of calcium carbonate in this particular eggshell sample is probably 80% – 91%. Of course, students should collect more than one set of data before they draw their conclusions.
Table 2. Determining the mass of carbon dioxide gas by weighing

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of the set-up before the reaction (g)</td>
<td>151.396</td>
</tr>
<tr>
<td>Mass of the set-up after the reaction (g)</td>
<td>151.309</td>
</tr>
<tr>
<td>Mass of CO$_2$ gas produced (g)</td>
<td>0.087</td>
</tr>
<tr>
<td>Number of moles of CO$_2$ (mol)</td>
<td>0.00198</td>
</tr>
<tr>
<td>Number of moles of CaCO$_3$ (mol)</td>
<td></td>
</tr>
<tr>
<td>Mass of CaCO$_3$ (g)</td>
<td>0.198</td>
</tr>
<tr>
<td>% CaCO$_3$ by mass</td>
<td>66.2%</td>
</tr>
</tbody>
</table>

Table 3. Measuring the volume of carbon dioxide gas by water displacement

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume of CO$_2$ collected (cm$^3$)</td>
<td>61.0</td>
</tr>
<tr>
<td>Number of moles of CO$_2$ (mol)</td>
<td>0.00240</td>
</tr>
<tr>
<td>Mass of CaCO$_3$ (g)</td>
<td>0.240</td>
</tr>
<tr>
<td>% CaCO$_3$ by mass</td>
<td>80.3%</td>
</tr>
</tbody>
</table>

Table 4. Back titration with NaOH(aq)

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final burette reading (cm$^3$)</td>
<td>23.50</td>
</tr>
<tr>
<td>Initial burette reading (cm$^3$)</td>
<td>3.20</td>
</tr>
<tr>
<td>Volume of NaOH used (cm$^3$)</td>
<td>20.30</td>
</tr>
<tr>
<td>Number of moles of NaOH (mol)</td>
<td>0.0199</td>
</tr>
<tr>
<td>Number of moles of HCl added to the eggshell sample (mol)</td>
<td>0.0241</td>
</tr>
<tr>
<td>Number of moles of HCl reacted with CaCO$_3$ (mol)</td>
<td>0.0042</td>
</tr>
<tr>
<td>Number of moles of CaCO$_3$ in the eggshell sample (mol)</td>
<td>0.0021</td>
</tr>
<tr>
<td>Mass of CaCO$_3$ in the eggshell sample (g)</td>
<td>0.21</td>
</tr>
<tr>
<td>% CaCO$_3$ by mass</td>
<td>70.2%</td>
</tr>
</tbody>
</table>
Table 5. Determining the mass of the unreacted eggshell

<table>
<thead>
<tr>
<th>Mass of filter paper and unreacted eggshell (g)</th>
<th>0.586</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of filter paper (g)</td>
<td>0.560</td>
</tr>
<tr>
<td>Mass of unreacted eggshell (g)</td>
<td>0.586 – 0.560 = 0.026</td>
</tr>
<tr>
<td>Mass of CaCO₃ (g)</td>
<td>0.299 – 0.026 = 0.273</td>
</tr>
<tr>
<td>% CaCO₃ by mass</td>
<td>(\frac{0.273}{0.299} \times 100% \approx 91.3%)</td>
</tr>
</tbody>
</table>

Major sources of error have been listed in Table 1. Other possible sources of error include:

1. The distribution of calcium carbonate in an eggshell is not even. Although we had grinded the eggshell to a fine powder, we are not sure whether the sample that we used was indeed representative.
2. The eggshell powder may have absorbed atmospheric moisture before we performed the experiment.
3. The stopper was not air-tight when we collected gases by water displacement.

References


Appendix A: One possible way to overcome large class size

1. The teacher let students know the problem and assessment criteria.

2. Students plan their investigation in groups.

3. Students submit their plans.

4. The teacher evaluates students’ plans and decides which groups will do oral presentation.

5. Students present their experimental designs orally and receive feedback from classmates and the teacher.

6. During the oral presentations, the teacher facilitates students to reach a consensus on the experimental design and emphasizes how the practical work can be done safely. Students revise their procedures if necessary.

7. Students carry out experiments, collect and analyze data, and submit a lab report.
**Appendix B: Assessment criteria for oral presentation**

<table>
<thead>
<tr>
<th>Content</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Your speech was clear and focused.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2. You made your purpose clear.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3. You effectively used visual aids to present information.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4. You answered your classmates’ queries clearly.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Organization</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5. Your presentation had an effective beginning, body, and ending.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6. You had good timing.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7. Team members were all involved in presentation.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Communication Skills</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8. Your language was suitable and accurate.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>9. You maintained effective eye contact.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10. The rate at which you spoke was appropriate.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>11. Your volume and body language were appropriate.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>TOTAL:</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
# Appendix C: Assessment criteria for written lab reports

<table>
<thead>
<tr>
<th>Introduction</th>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Purpose is stated.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.</td>
<td>Clearly explains why this inquiry is important.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.</td>
<td>Clearly explains the chemical principle of the method.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.</td>
<td>Procedures are clear enough to be replicated.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5.</td>
<td>Labelled drawings are used to help presentation.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Results</th>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.</td>
<td>Summarizes data clearly using tables and/or graphs.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.</td>
<td>Appropriate units and significant figures are used.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8.</td>
<td>Calculations are correct and shown clearly.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>9.</td>
<td>Identifies trends and outlying data.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Conclusion</th>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>10.</td>
<td>Makes conclusions that are well supported by the data.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>11.</td>
<td>Uses scientific ideas to try to explain.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12.</td>
<td>Identifies major sources of error and explains their effects on results.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>13.</td>
<td>Identifies possible improvements in the experimental design.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Overall Report</th>
<th>Criteria</th>
<th>Marks Possible</th>
<th>Assessment Self</th>
<th>Assessment Teacher</th>
</tr>
</thead>
<tbody>
<tr>
<td>14.</td>
<td>The work is neat and organized.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>15.</td>
<td>Uses proper spelling and grammar.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>16.</td>
<td>Appropriately cites written and/or web-based references.</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**TOTAL:** 115
Appendix D: Student questionnaire

Please circle your answer to each question on a scale of 1 to 6, with 1 = strongly disagree and 6 = strongly agree.

<table>
<thead>
<tr>
<th></th>
<th>The catalase investigation was interesting.</th>
<th>Strongly disagree</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>The catalase investigation is useful because I now know more about the chemistry inside my body.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>I have learnt from the catalase investigation that there is often more than one way to solve a chemistry problem.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>The catalase investigation is important because I had a chance to apply chemical knowledge to solve an unfamiliar problem.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>The catalase investigation was challenging because I had to plan the procedure.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>The catalase investigation was a good way for me to practise practical skills.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>The catalase investigation was challenging because I needed to decide how the data should be presented and analyzed.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Overall, I like this kind of inquiry-based experiment more than the traditional “cookbook” laboratory work.</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td></td>
</tr>
</tbody>
</table>

Other comments (if any):