CHEM 140 Lab Prep Manual

Western Carolina University
Cullowhee, NC

CHEM 140 – Determining Solution Concentration Prep Sheet

1 Lab Materials

Each equipment kit (red bin) should contain the following:

Quantity	**Item**
3	50 mL beakers
2	100 mL beakers
1	500 mL beaker
1	25 mL graduated cylinder
1	100 mL graduated cylinder
1	glass stirring rod

Also place a box of disposable plastic transfer pipets at the front of the lab for students to use.

2 Chemicals

2.1 Solid NaCl Preparation

The NaCl used in this experiment is the commercially available *non-iodized* salt from a grocery store (the kind with the pour spout.) Place a container of table salt in each white chemical bin and place one bin under each hood. There should be six bins total.

2.2 NaCl Unknown Solutions

There will be 6 NaCl solutions with different concentrations that the students will use as unknowns. Each section will need approximately 40 mL of *each* unknown. Calculate the amount of each unknown you will have to make using the equation below so that there is a 25% excess.

$$\text{volume of each unknown NaCl solution} = \text{\# of sections}\left(40\frac{\text{mL}}{\text{section}}\right)(1.25)$$

Unknown solutions will have the following (w/v) concentrations: 8%, 12%, 14%, 16%, 18%, and 22%. An 8% solution will have 8 g of NaCl for every 100 mL of water. Plug the volume of the solution you need and the percent concentration into the equation below to calculate the mass of NaCl to use.

$$\text{mass of NaCl} = \text{volume of solution}\left(\frac{\text{concentration}\%}{100\text{ mL}}\right)$$

Prepare the solutions in *one* container for each unknown and label each container with the following information: Unknown # (see below), Chem 140, and the year. Place the six containers in the cabinet in front of the lab under the front lab bench.

- Unknown 140-1-1: 8%
- Unknown 140-1-2: 12%
- Unknown 140-1-3: 14%
- Unknown 140-1-4: 16%
- Unknown 140-1-5: 18%
- Unknown 140-1-6: 22%

3 Instructor's Prelaboratory Preparations

1. Sign out the computer cart from the Geosciences and Natural Resources Department and bring it to the lab.
2. Make sure the entire cart is plugged in (two cords!) and both red switches on the back have been turned on.
3. Make sure there is plenty of paper. (Go get some from NS 230 if there's not enough.)
4. Open the cart with the key: one end goes in the top lock and one end goes in the bottom lock. Then turn the handle to open.
5. Remove materials kits (red bins) from the cabinets and place them on the lab benches for the students.
6. Remove the chemical kits (white bins) from under the hoods and place them in the hood.
7. Put the unknowns on the front lab bench. (They're in the cabinet up front.)

4 Prelaboratory Lecture

1. As students enter the lab, check answers for numbers 1 and 2 on the Data Sheet for the prelab. (See the attached answer key rubrick.) Then go over the answers once everyone has been checked off.
2. Go over safety rules and procedures, indicate locations of safety equipment (fire extinguisher, eye wash station, safety shower, fire blanket), and have students sign and return safety sheets.
3. Briefly go over the premise of the laboratory, especially the concept of a calibration curve and using it to determine the physical property of an unknown.
4. Demonstrate how to dry a graduated cylinder as follows:

   (a) Roll the ENTIRE length of a stirring rod with some paper towel. (Make sure both ends of the stirring rod are covered.)
   (b) Holding the rod and the end of the paper towel simultaneously, dip the rod into the cylinder and rub it around to soak up the droplets with the paper towel.
   (c) Remove the rod and paper towel.

5. Show students how to use the computer cart.
   (a) Gently remove a laptop and record the laptop number on the data sheet.
   (b) If the laptop has a low battery, unplug the power supply cord and plug the computer in at the lab bench or the work area in the middle of the lab.
   (c) When students are finished, laptops should be returned to the cart. The computer MUST be plugged into the back of the inside of the cart to charge the battery.
   (d) Computer cords should be tucked neatly on TOP of the laptop and not hanging in front of the cart door.
   (e) Students should record their computer number on their data sheet. You will check to see that the computer has been put away properly before checking the box on the data sheet.
   (f) Please stress the importance of taking care of these computers.
6. Once all the students have some density data, use the computer workstation to demonstrate how to prepare an Excel graph. (Students can work with you on their own computers.) Be sure to demonstrate how to print the graph on one sheet of paper. Ask students to have you check their graphs BEFORE they print so they don't waste paper.
7. Students should rinse and dry all their glassware and return it to the bin. Their kit number should be recorded on their data sheet. Students should have you inspect their kit before you accept their data sheet for grading. There is a checkbox at the top of the data sheet.

5 End-of-lab Instructor Procedures

1. Check students' kits to make sure all items are accounted for and glassware is clean and dry.
2. Check to see that students have put the NaCl back in their chemical kit and that the weighing area is clean.
3. Check to see that the students' lab area is clean (no spills, no trash, etc.)
4. Check to see that the students' computer has been put away properly:

5. Collect the students’ data sheets and graphs for grading. These should not be collected unless their kit, lab area and computer have been checked.
6. If these items have not been checked off on the data sheet, the students should lose some points on their lab. (See rubrick.)

6 Data Sheet Key – *Answers and comments given in italics*

☐ Kit #________
☐ Chemical Kit #________
☐ Computer#________

Up to ten discretionary points can be deducted if students fail to clean their lab area or do not have their kits and computer checked off. You may also deduct points for failing to follow safety instructions. (No goggles, open-toed shoes, horsing around, etc.)

5 pts. 1. Mass of KCl = 5 g

5 pts. 2. Calculation:

$$\left(\frac{5\text{ g}}{25\text{ mL}}\right)\left(\frac{1\text{ mol}}{74.55\text{ g}}\right)\left(\frac{1000\text{ mL}}{1\text{ L}}\right) = 2.68M$$

Concentration = 2.68 M

5 pts. In the table below, write your data from Section 2.1. Be sure to include the correct number of significant figures in your answer. Show your calculations for each calculated mass and actual concentration below.

Calculated Mass (g)	Actual Mass (g)	Target Volume (mL)	Actual Volume (mL)	Actual Concentration (%)
7.5		30		
12		60		
4.5		30		

Table 1: Measurements for creating stock solutions.

Stock Solution Calculations

5 pts. 25% Solution:

$$30\text{ mL}\left(\frac{25\text{ g}}{100\text{ mL}}\right) = 7.5\text{ g};\quad \text{Actual Concentration} = \frac{\text{Actual Mass}}{\text{Actual Volume}}(100\%)$$

5 pts. 20% Solution:

$$60\text{ mL}\left(\frac{20\text{ g}}{100\text{ mL}}\right) = 12\text{ g}$$

$$\text{Actual Concentration} = \frac{\text{Actual Mass}}{\text{Actual Volume}}(100\%)$$

5 pts. 15% Solution:

$$30\text{ mL}\left(\frac{15\text{ g}}{100\text{ mL}}\right) = 4.5\text{ g}$$

$$\text{Actual Concentration} = \frac{\text{Actual Mass}}{\text{Actual Volume}}(100\%)$$

5 pts. In the table below, write your data from Section 2.2. Be sure to include the correct number of significant figures in your answer. Show your calculations for each calculated volume and actual concentration below.

Calculated Vol. of Stock (mL)	**Actual Volume of Stock (mL)**	**Target Total Vol. (mL)**	**Actual Total Vol. (mL)**	**Actual Concentration (%)**
25		50		
15		30		

Table 2: Measurements for creating diluted solutions.

Dilution Calculations

5 pts. 10% Solution:

$$V_2 = \frac{C_1V_1}{C_2} = \frac{(10\%)(50\text{ mL})}{(20\%)} = 25\text{ mL}$$

$$\text{Actual Concentration} = \frac{\text{(stock soln. conc.) (volume of stock)}}{\text{actual total volume}}$$

5 pts. 5% Solution:

$$\frac{(5\%)(30\text{ mL})}{(10\%)} = 15\text{ mL}$$

$$\text{Actual Concentration} = \frac{\text{(stock soln. conc.) (volume of stock)}}{\text{actual total volume}}$$

5 pts. In the table below, write your data from Section 2.2. Be sure to include the correct number of significant figures in your answer. Show your calculations for each calculated volume and actual concentration below.

Values for concentration should match those calculated for the previous tables. Densities should be calculated by dividing column 2 by column 3. If the solutions were prepared correctly, values for density should be close to those listed below.

Concentration (%)	**Mass of Sample (g)**	**Volume of Sample (mL)**	**Density (g/mL)**
Unknown #______			
Unknown #______			

Table 3: Density determinations.

Density Calculations

5 pts. 25% Solution:

5 pts. 20% Solution:

5 pts. 15% Solution:

5 pts. 10% Solution:

5 pts. 5% Solution:

5 pts. Unknown #_______:

5 pts. Unknown #_______:

Unknown Concentration Calculations

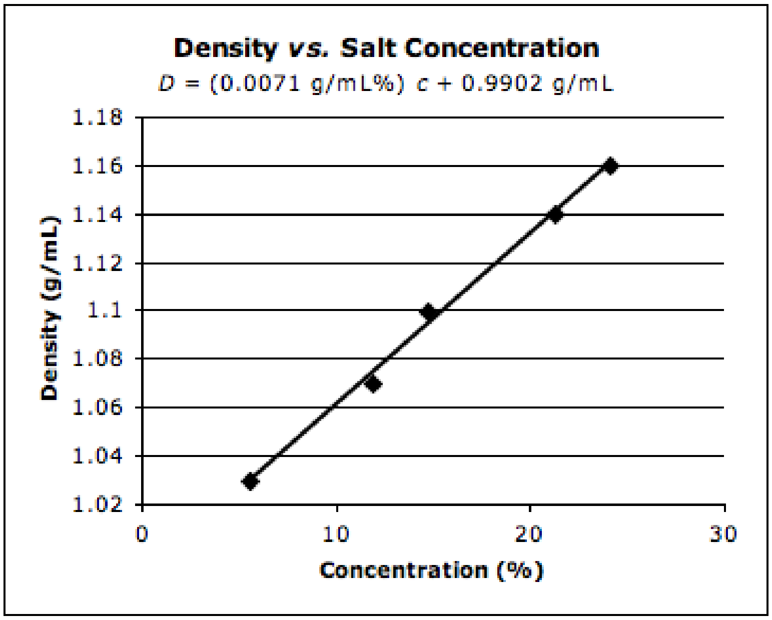


Figure 1: Density *vs.* NaCl Concentration

5 pts. Equation: *See graph above.*

5 pts. Unknown #_______: $c = \frac{D - 0.9902\,\frac{\text{g}}{}}{0.0071\,\frac{\text{g}}{\text{mL}\,\%}}$

Unknown Key:

- Unknown 1-1: 8%
- Unknown 1-2: 12%
- Unknown 1-3: 14%
- Unknown 1-4: 16%
- Unknown 1-5: 18%
- Unknown 1-6: 22%

5 pts. Unknown #_______: *Same as above.*

CHEM 140 – Heat of Solution of Solid KOH Prep Sheet

1 Lab Materials

Each equipment kit (red bin) should contain the following:

Quantity	Item
1	large Styrofoam cup with cardboard lid
1	800 mL beaker
1	Hg thermometer w/ 0.1 °C resolution
1	100 mL graduated cylinder
1	stir plate
1	small magnetic stir bar
1	ring stand with burette clamp
1	spatula

Set up an apparatus at the front of the lab for students to see as an example: Place stir bar in the cup and place the cup on a stir plate. Then, put the stir plate with cup onto the bottom of the ring stand. Use the burette clamp to suspend the thermometer in the cup so that the bottom of the thermometer is about one inch from the bottom of the cup. Also, a magnetic stir bar retriever should also be available in the laboratory.

2 Chemicals

The only chemical used in this experiment is solid potassium hydroxide (KOH). Each section will use approximately 324 g of KOH divided into 6 containers. To determine how much KOH to prepare in each container, multiply the number of sections by 324 g, then divide by 6 containers. Finally multiply by 1.2 to give a 20% excess.

$$\text{Amount of KOH in each container} = \frac{(\#\text{of sections})\,(324\text{ g})\,(1.2)}{6}$$

If necessary, use smaller containers, but have a larger stock container available under the front lab bench for instructors to use for refilling.

3 Waste Handling

This experiment generates a lot of aqueous waste. In each hood, provide a 2-4 L waste container labeled "Chem 140 KOH(aq) Waste" with the date. At the end of each day, collect the waste, neutralize it with small aliquots of HCl until pH reaches 7. Once neutralized, the solution can be poured down the drain. The waste should be approximately 0.8 M KOH. So for 1 L of waste, you will need approximately 67 mL of concentrated HCl.

4 Instructor's Prelaboratory Preparations

1. Sign out the computer cart from the Geosciences and Natural Resources Department and bring it to the lab.
2. Make sure the entire cart is plugged in (two cords!) and both red switches on the back have been turned on.
3. Make sure there is plenty of paper. (Go get some from NS 230 if there's not enough.)
4. Open the cart with the key: one end goes in the top lock and one end goes in the bottom lock. Then turn the handle to open.
5. Remove materials kits (red bins) from the cabinets and place them on the lab benches for the students.
6. Remove the chemical kits (white bins) from under the hoods and place them in the hood.
7. Check that there is enough KOH for each group. Refill containers if necessary from the stock container under the front lab bench.

5 Prelaboratory Lecture

1. As students enter the lab, collect the prelaboratory assignment.
2. Briefly go over the premise of the laboratory, especially the concepts of heat of solution, heat exchange, q and ΔH.
3. Remind students of how to use the computer cart.
4. Demonstrate how to use weighing paper.
5. Discuss the hazards of working with KOH. Point out what to do in case of emergency. Remind students of the location of the eye wash and safety shower.
6. Stress the importance of being careful with the mercury thermometers.
7. Once all the students have completed the part to study the effect of the mass of water, use the computer workstation to demonstrate how to prepare an Excel graph with three plots on one graph. (Students can work with you on their own computers.) Make sure students email their graphs to themselves so they'll have it when they go home.
8. Students must have you check that their computer was put away properly. Check the box on their data sheet.

9. Students should rinse and dry all their glassware and return it to the bin. Their kit number should be recorded on their data sheet. Students should have you inspect their kit before you accept their data sheet for grading. There is a checkbox at the top of the data sheet.

6 End-of-lab Instructor Procedures

1. Check students' kits to make sure all items are accounted for and glassware is clean and dry.
2. Check to see that students have put the KOH back in their chemical kit and that the weighing area is clean.
3. Check to see that the students' lab area is clean (no spills, no trash, etc.)
4. Check to see that the students' computer has been put away properly.
5. Clean up the balance areas.
6. Return the computer cart.

Name: *Answer Key*

Answers and comments given in italics

☐ Kit #__________
☐ Chemical Kit #__________
☐ Computer#__________

Up to ten discretionary points can be deducted if students fail to clean their lab area or do not have their kits and computer checked off. You may also deduct points for failing to follow safety instructions. (No goggles, open-toed shoes, horsing around, etc.)

7 Pre-lab Answers

2 pts. 1. When more water is used while keeping the mass of KOH constant, ΔT should decrease since there are more water molecules to absorb the heat released by the dissolution.

2 pts. 2. When more water is used while keeping the mass of KOH constant, ΔH should remain constant. ΔH is an *intensive* property meaning it doesn't depend on how much KOH is present.

2 pts. 3. When more KOH is used while keeping the mass of water constant, ΔT should increase. More KOH means more ionic bonds broken and therefore more heat released.

2 pts. 4. When more KOH is used while keeping the mass of water constant, ΔH should remain constant because ΔH is an *intensive* property. It doesn't depend on how much KOH is used because it's a molar quantity.

2 pts. *Students should have their answers typed.*

8 Data Tables

6 pts. *Each sheet should show the mass of KOH and the volume of H_2O used with the proper number of decimal places and units. Volume should have one decimal place. Temperatures should be given with one decimal place.*

9 Graphs

4 pts. - Each graph should have three plots with a sigmoidal shape.

2 pts. - Each graph should have axis labels with proper units (time in minutes or seconds for the x-axis and temperature in °C for the y-axis.

2 pts. - Each graph should have a title at the top.

2 pts. - The legend should clearly indicate which plot represents which solution.

4 pts. - Each graph should have a figure caption that reads something like this... Figure x: Temperature *vs.* time data for determining the effect of the mass of (KOH or water) on ΔT. Squares represent using 50 mL of water; circles represent... etc.

10 Sample Calculations

2 pts. $T_f =$*highest temperature from Data Table 1*

2 pts. $T_i =$*temperature before KOH was added*

2 pts. $\Delta T = T_f - T_i$

2 pts. Mass of water =*Volume of water* $\left(\frac{1.00\ g}{mL}\right) =$ *Volume of water from Data Table 1 in grams*

2 pts. $c = 4.184\ J/molK$

4 pts. $q = (\text{Mass of water})\,(4.184\ J/molK)\,(\Delta T)$

2 pts. Mass of KOH= *Value from Data Table 1*

2 pts. Number of moles of KOH =*Mass of KOH*$\left(\frac{1\ mole\ KOH}{56.108\ g}\right)$

2 pts. $\Delta H = \frac{q}{Number\ of\ moles\ of\ KOH}$

11 Data Summary Tables

There should be two tables that look like the table below.

2 pts. - Each value in the table should have the right number of significant figures.

2 pts. - Each value in the table should have units.

4 pts. - Each table should have a caption to indicate that it corresponds to either the effect of the mass of KOH or the effect of the mass of water.

2 pts. - Each table caption should be numbered (e.g. Table 1: ...).

12 Results

Effect of Water Mass

Table 4: Effect of _________ Mass

	Solution 1	**Solution 2**	**Solution 3**
T_f			
T_i			
ΔT			
Mass of Water			
q			
Mass of KOH			
n			
ΔH			

2 pts. - Figure 1 shows time vs. temperature data for experiments performed to study the effect of water mass on ΔT. As you can see in the graph temperature increases sharply after the addition of KOH for all three solutions.

3 pts. - The solution with the largest increase in temperature corresponds to the solution with the least amount of water.

Effect of KOH Mass

2 pts. - Figure 2 shows time vs. temperature data for experiments performed to study the effect of KOH mass on ΔT. As you can see in the graph temperature increases sharply after the addition of KOH for all three solutions.

3 pts. - The solution with the largest increase in temperature corresponds to the solution with the largest amount of KOH.

13 Discussion

Effect of Water Mass

5 pts. - ΔT decreases as the mass of water increases. The amount of heat liberated is dictated by the amount of KOH which is constant, but there are more molecules of water as the water mass increases, so since the heat is distributed over more water molecules, the temperature doesn't increase as much.

5 pts. - q stays constant as the mass of water increases because the increase in water mass is canceled by the decrease in ΔT in the equation $q = mc\Delta T$.

5 pts. - ΔH stays constant as the mass of water increases because q and the amount of KOH are constant. ($\Delta H = q/n$ is constant.)

Effect of KOH Mass

5 pts. - ΔT increases as the mass of KOH increases. More K-OH bonds are broken so more heat is liberated. Since the mass of water is constant, each water molecule receives more heat with additional KOH and the temperature increases even more.

5 pts. - q increases as the mass of KOH increases for the same reason that ΔT increases. (More K-OH bonds are broken.)

5 pts. - ΔH stays constant as the mass of KOH increases. Although q increases, n increases proportionally so the ratio q/n stays the same.

CHEM 140 – Hess's Law

1 Lab Materials

Each equipment kit (red bin) should contain the following:

Quantity	Item
2	large Styrofoam cup with cardboard lid
1	500 mL beaker
1	Hg thermometer w/ 0.1 °C resolution
1	100 mL graduated cylinder
1	stir plate
1	glass stirring rod
1	small magnetic stir bar
1	ring stand with burette clamp
1	spatula
1	red and/or blue litmus paper

Set up an apparatus at the front of the lab for students to see as an example: Place stir bar in the cup and place the cup on a stir plate. Then, put the stir plate with cup onto the bottom of the ring stand. Use the burette clamp to suspend the thermometer in the cup so that the bottom of the thermometer is about one inch from the bottom of the cup. Also, a magnetic stir bar retriever should also be available in the laboratory.

2 Chemicals

2.1 Solid Potassium Hydroxide

Each section will use approximately 120 g of KOH divided into 6 containers. To determine how much KOH to prepare in each container, multiply the number of sections by 120 g, then divide by 6 containers. Finally multiply by 1.2 to give a 20% excess.

$$\text{Amount of KOH in each container} = \frac{(\#\text{of sections})\,(120\text{ g})\,(1.2)}{6}$$

If necessary, use smaller containers, but have a larger stock container available under the front lab bench for instructors to use for refilling.

2.2 1 M Potassium Hydroxide Solution

Each section will require approximately 1200 mL of 1 M KOH solution divided into six plastic containers. To determine the total volume of KOH solution to prepare in each container, multiply the number of sections by 1.2 L, then divide by 6 containers. Finally, multiply by 1.2 to give a 20% excess.

$$\text{Liters of KOH solution in each container} = \frac{(\text{\# of sections})\,(1.2\text{ L})\,(1.2)}{6}$$

To make the desired concentration add the solid KOH to a large beaker (large enough to accommodate the final total volume). Add about 80-90% of the DI water, and a stir bar. Stir the solution until the KOH has dissolved completely. The solution will be quite hot, so wait until it cools back to room temperature before proceeding. Carefully, add the solution to a volumetric flask of the appropriate volume (six times the volume from the above calculation). Rinse the beaker with several aliquots of DI water to make sure all the KOH gets into the flask, then fill the flask to the mark with DI water. Finally, distribute the solution to the six containers. The mass of KOH to use is determined using the following formula.

$$\text{Grams of KOH solid to use} = \text{Volume from the above calcuation}\times 6\times\left(1\frac{\text{mol}}{\text{L}}\right)\left(\frac{1\text{g}}{56.11\text{mol}}\right)$$

2.3 1.5 M Hydrochloric Acid Solution

Each section will require approximately 3 L of 1.5 M HCl solution divided into six plastic containers. To determine the total volume of HCl solution to prepare in each container, multiply the number of sections by 3 L, then divide by 6 containers. Finally, multiply by 1.2 to give a 20% excess.

$$\text{Liters of HCl solution in each container} = \frac{(\text{\# of sections})\,(3\text{ L})\,(1.2)}{6}$$

To make the desired concentration add tabout 50% of the DI water, and a stir bar to a large beaker (large enough to accommodate the final total volume). Carefully add the required volume of concentrated HCl (see calculation below). Stir the solution until the HCl has mixed completely and the solution has cooled back to room temperature. Carefully, add the solution to a volumetric flask of the appropriate volume (six times the volume from the above calculation). Rinse the beaker with several aliquots of DI water to make sure all the HCl gets into the flask, then fill the flask to the mark with DI water. Finally, distribute the solution to the six containers. The volume of concentrated HCl to use is determined using the following formula.

$$\text{Liters of conc. HCl to use} = \text{Volume from the above calcuation}\times 6\times\left(1.5\frac{\text{mol}}{\text{L}}\right)\left(\frac{1\text{L}}{12.1\text{mol}}\right)$$

3 Waste Handling

This experiment generates a lot of aqueous waste. In each hood, provide a 2-4 L waste container labeled “Chem 140 HCl(aq) Waste” with the date. At the end of each day, collect the waste, neutralize it with small aliquots of NaOH or KOH until pH reaches 7. Once neutralized, the solution can be poured down the drain. The waste should be approximately 0.8 M HCl. So for 1 L of waste, you will need approximately 45 g of base.

4 Instructor's Prelaboratory Preparations

1. Sign out the computer cart from the Geosciences and Natural Resources Department and bring it to the lab.
2. Make sure the entire cart is plugged in (two cords!) and both red switches on the back have been turned on.
3. Open the cart with the key: one end goes in the top lock and one end goes in the bottom lock. Then turn the handle to open.
4. Remove materials kits (red bins) from the cabinets and place them on the lab benches for the students.
5. Remove the chemical kits (white bins) from under the hoods and place them in the hood.
6. Check that there is enough KOH, KOH solution and HCl solution for each group. Refill containers if necessary from the stock container under the front lab bench.

5 Prelaboratory Lecture

1. As students enter the lab, collect the prelaboratory assignment.
2. Briefly go over the premise of the laboratory, especially the concepts Hess's law.
3. Remind students of how to use the computer cart.
4. Demonstrate how to use litmus paper. Students should dip a stirring rod into their solution and lightly touch it to the litmus paper to observe the color. (Red paper remains red when dipped in acid and turns blue when dipped in base. Blue paper turns red when dipped in acid and remains blue when dipped in base.)
5. Discuss the hazards of working with KOH and HCl. Point out what to do in case of emergency. Remind students of the location of the eye wash and safety shower.
6. Stress the importance of being careful with the mercury thermometers.
7. Once all the students have completed the part to study the effect of the mass of water, use the computer workstation to demonstrate how to prepare an Excel graph with three plots on one graph. (Students can work with you on their own computers.) Make sure students email their graphs to themselves so they'll have it when they go home.
8. Students must have you check that their computer was put away properly. Check the box on their prelab they turned in.

9. Students should rinse and dry all their glassware and return it to the bin. Their kit number should be recorded on their data sheet. Students should have you inspect their kit before you accept their data sheet for grading. There is a checkbox at the top of the prelab. Students should also wipe down their benchtop with a wet paper towel and dry it thoroughly.

6 End-of-lab Instructor Procedures

1. Check students' kits to make sure all items are accounted for and glassware is clean and dry.
2. Check to see that students have put the KOH, KOH solution and HCl solution back in their chemical kit and that the weighing area is clean.
3. Check to see that the students' lab area is clean (no spills, no trash, etc.)
4. Check to see that the students' computers have been put away properly.
5. Clean up the balance areas.
6. Return the computer cart.

Answers and comments given in italics

☐ Kit #__________
☐ Chemical Kit #__________
☐ Computer#__________

Up to ten discretionary points can be deducted if students fail to clean their lab area or do not have their kits and computer checked off. You may also deduct points for failing to follow safety instructions. (No goggles, open-toed shoes, horsing around, etc.)

7 Prelaboratory Assignment

5 pts. 1. Using Hess's law, combine Reaction 1 and Reaction 2 to give $\text{KOH(aq)} + \text{HCl(aq)} \longrightarrow \text{H}_2\text{O(l)} + \text{KCl(aq)}$.

$$\begin{array}{l} \text{KOH(aq)} \longrightarrow \text{KOH(s)} \\ \underline{\text{KOH(s)} + \text{HCl(aq)} \longrightarrow \text{H}_2\text{O(l)} + \text{KCl(aq)}} \\ \text{KOH(aq)} + \text{HCl(aq)} \longrightarrow \text{H}_2\text{O(l)} + \text{KCl(aq)} \end{array}$$

5 pts. 2. How should ΔH_{sol} and ΔH_{neut} be combined to give the change in enthalpy for Reaction 3, ΔH?

$$\Delta H = -\Delta H_{sol} + \Delta H_{neut}$$

3. In the previous experiment, you determined values for ΔH_{sol} when 2, 4, and 6 grams of KOH was added to 100 mL of H_2O. Record the average of these three ΔH_{sol} values on your data sheet. (You should also write this value down on a separate piece of paper since you'll need it for your calculations.)

Students should record their average results from the previous experiment. No points are earned here.

8 Data Tables

No points are earned for the data tables. However, all of the following data should be included.

For each trial:

- *Mass of KOH (s) or Volume of KOH (aq)*
- *Volume of HCl (aq)*
- *litmus paper color*
- *Acid or base final solution*

All solutions should be acidic after the reaction is complete. Therefore all litmus tests should be red.

9 Graphs

2 pts. - Each graph should have two plots with a sigmoidal shape.

2 pts. - Each graph should have axis labels with proper units (time in minutes or seconds for the x-axis and temperature in °C for the y-axis.

2 pts. - Each graph should have a title at the top.

2 pts. - The legend should clearly indicate which plot represents which trial.

2 pts. - Each graph should have a figure caption that reads something like this... Figure x: Temperature *vs.* time data for determining enthalpy for Reaction 2. Squares represent using trial 1 and... etc.

9.1 Sample Calculations

Sample calculations should be shown for trial 1 for BOTH reactions.

1 pt. Total volume of solution = *From Data Table*

1 pt. Total mass of water =*From Data Table*

1 pt. T_f =*highest temperature from Data Table*

1 pt. T_i =*temperature before KOH was added*

1 pt. $\Delta T = T_f - T_i$

1 pt. q =*Mass of water* $\times$ 4.184 J mol^{-1} °C^{-1} $\times \Delta T$

1 pt. Mass of KOH = (for Reaction 2 only) - *From Data Table*

1 pt. Volume of KOH solution = (for Reaction 3 only) - *From Data Table*

1 pt. Molarity of KOH solution = (for Reaction 3 only) *1 M*

Moles of KOH used =

1 pt. *For solid KOH:* $n = \frac{\textit{Mass of KOH}}{56.11 g/mol}$

1 pt. *For aqueous KOH:* $n = (\textit{Volume of KOH soln in L})\,(1\ mol/L)$

1 pt. Volume of HCl solution =*From Data Table*

1 pt. Molarity of HCl solution = *1.5 M*

1 pt. Moles of HCl used =$n = (\text{Volume of HCl soln in L})\,(1.5\ mol/L)$

1 pt. Limiting Reagent = *KOH for all reactions*

1 pt. Does your litmus test support your result? Explain why. *All litmus tests should result in red litmus paper, meaning the resulting solution is acidic. That means all the base has been consumed, so the base (KOH) is the limiting reagent.*

1 pt. Number of moles of limiting reagent = *Should equal the number of moles of KOH*

1 pt. $\Delta H = q/n$

1 pt. Hess's Law Calculation: $\Delta H = -\Delta H_{sol} + \Delta H_{neut}$ where ΔH_{sol} is the averaged value from the previous experiment and ΔH_{neut} is the averaged value from Reaction 2.

1 pt. Percent Difference Calculation:

$$\text{Percent difference} = \frac{|\text{calculated value} - \text{experimental value}|}{\text{experimental value}} \times 100\%$$

where the calculated value is from the Hess's Law calculation above and the experimental value is the averaged value from Reaction 3.

10 Data Summary Tables

There should be two tables that look like the table below.

4 pts.

- Each value in the table should have the right number of significant figures.

Table 5: Data Summary Table

	Reaction 2		**Reaction 3**	
	Trial 1	**Trial 2**	**Trial 1**	**Trial 2**
Total volume of soln, mL				
Total mass of water, g				
T_f**, °C**				
T_i**, °C**				
ΔT**, °C**				
q**, J**				
Mass of KOH, g			▨	▨
Volume of KOH soln, L	▨	▨		
Molarity of KOH soln, mol/L	▨	▨		
Moles of KOH used, mol				
Volume of HCl soln, L				
Molarity of HCl soln, mol/L				
Moles of HCl used, mol				
Limiting Reagent				
n**, mol**				
ΔH**, J**				
Average ΔH**, J/mol**				

4 pts. • Each value in the table should correspond to the units listed in the left-most column.

• Each table should have a caption to indicate that it corresponds to either reaction 2 or reaction 3. 8 pts.

4 pts. • The table caption should be numbered (e.g. Table 1: ...).

11 Results

8 pts. • Figure 1 shows time *vs.* temperature data for Reaction 2: $KOH(s)+HCl(aq) \longrightarrow H_2O(l)+KCl(aq)$. As you can see in the graph temperature increases sharply after the addition of KOH for both trials then leveled off.

8 pts. • Figure 2 shows a similar behavior for time vs. temperature data for Reaction 3: $KOH(aq) + HCl(aq) \longrightarrow H_2O(l) + KCl(aq)$.

• The temperature increase for Reaction 3 was not as large as the increase for Reaction 2. 4 pts.

12 Discussion

5 pts.
- Restate the average experimental values of ΔH_{neut} and ΔH.

5 pts.
- Qualitative assessment: ΔH_{neut} is larger than ΔH.

5 pts.
- Explanation: More heat is released in Reaction 3 since the bonds being broken are ionic. In reaction 2, the reactants are already dissolved so the ionic bonds are not there to be broken. Since ionic bonds are high in energy (strong), a lot of heat is released when they're broken.

5 pts.
- Comment on any difference between the calculated and experimental values. Must have percent difference value.

CHEM 140 – Specific Heat of Metals Prep Sheet

1 Lab Materials

Each lab station will require a hot plate, a ring stand and a buret clamp. Additionally, each materials kit will require the following:

Quantity	**Item**
1	styrofoam cup
1	Hg thermometer with 0.1 °Cresolution
1-2	metal samples
1	tongs
1	600 mL or 800 mL beaker

2 Prelaboratory Lecture

1. As students enter the lab, check the definitions on their Data Sheet. Then, review the definitions as a class.
2. The purpose of this experiment is to determine the specific heat capacities of some metal substances and use the specific heat to identify the metal.
3. Review the theory of calorimetry and how heat will be transferred from the metal to the water. Also review the equations that will be used, but leave the students to combine Equations 2 and 3 to find c_{metal} as part of their calculations.
4. Students should have metals in their materials kits. If they only have one, they can borrow one from another group when they are finished with it.
5. Remind students that they are working with mercury thermometers again and should treat them very carefully so as not to break them.

3 End-of-lab Instructor Procedures

1. Check students' data sheets before they leave lab. If they did not clean their station and return all the items to their kits, do not check the box at the top of the data sheet.
2. Labs should be turned in at the beginning of the next lab period. They should include the following: completed data sheets, completed calculation sheet and summary table, a typed abstract, and a typed introduction.

Name: *Answer Key* Lab Section:

Partner's Name:

4 Data Sheet

☐ Kit #__________

4.1 Prelaboratory Assignment

Define the following terms:

2 pts.
- calorimeter: *A container used to study heat transfer in which the system is thermally isolated from the surroundings*

2 pts.
- heat: *The energy transferred from one object to another as the result of a temperature difference between them.*

3 pts.
- heat capacity: *The amount of heat required to raise the temperature of an object or substance by a given amount.*

3 pts.
- specific heat capacity: *The amount of heat required to raise the temperature of 1 g of a substance by 1 °C.*

2 pts.

4.2 Data for Metal 1

1. Mass of Metal: ____________________

   Metal appearance:

2. Water Mass Determination

   Trial 1:

   Mass of empty cup: ________________

   Mass of cup with water: ________________

   Mass of water in cup: ________________

   Trial 2:

   Mass of empty cup: ________________

   Mass of cup with water: ________________

   Mass of water in cup: ________________

2 pts.

4.3 Data for Metal 2

1. Mass of Metal: ________________

   Metal appearance:

2. Water Mass Determination

   Trial 1:

   Mass of empty cup: ________________

   Mass of cup with water: ________________

   Mass of water in cup: ________________

Trial 2:

Mass of empty cup: ____________________

Mass of cup with water: ____________________

Mass of water in cup: ____________________

Metal 1, Trial 1		**Metal 1, Trial 2**	
time (min:sec)	**Temp. (°C)**	**time (min:sec)**	**Temp. (°C)**

Metal 2, Trial 1		**Metal 2, Trial 2**	
time (min:sec)	**Temp. (°C)**	**time (min:sec)**	**Temp. (°C)**

5 Calculations and Results

Use your data from trial 1 of metal 1 to calculate the answers below. Then, apply these calculations to all four of your trials, and fill in the summary chart on the next page.

5.1 Determining ΔT

1 pt. 1. T_i(water) is the the initial temperature of the cool water just before the hot metal was added: T_i(water)= ________________

1 pt. 2. T_f(water) is the highest temperature the cool water reached before leveling off or beginning to fall back down: T_f(water)= ________________

1 pt. 3. ΔT(water)= $\underline{T_f\text{(water) - }T_i\text{(water)}}$

1 pt. 4. T_i(metal) is the initial temperature of the hot metal. Even though this was not measured, you should know what this is: T_i(metal)= ________________

1 pt. 5. T_f(metal) is the final temperature of the metal. Even though this was not measured directly, you should know what this is: T_f(metal)= ________________

1 pt. 6. ΔT(metal)= $\underline{T_f\text{(metal) - }T_i\text{(metal)}}$ *Note: Should be negative!*

5.2 Determining c for the metal

5 pts. 1. Combine Equations 2 and 3 to write an expression containing the masses, specific heat capacities and changes in temperature for the metal and the water. (Use subscripts to indicate which substance the variable applies to. For example: write m_{metal} for the mass of the metal. You should only have variables in your equation at this point.)

$$m_{metal} \times c_{metal} \times \Delta T(\text{metal}) = m_{water} \times c_{water} \times \Delta T(\text{water}) \quad (1)$$

5 pts. 2. Solve your equation from the previous problem for the specific heat capacity of the metal.

$$c_{metal} = \frac{m_{water} \times c_{water} \times \Delta T(\text{water})}{m_{metal} \times \Delta T(\text{metal})} \quad (2)$$

5 pts. 3. Using your data for the mass of water, the mass of the metal, and the changes in temperature you determined in Section 7.1, calculate c_{metal}. (Use $c_{H_2O} = 4.184$ J mol^{-1} °C $^{-1}$.)

In testing this lab, I believe the metals used are brass (labeled brass), bronze (looks like copper, labeled CO.) and zinc (the silver one). The values I determined for the specific heats are given below. Also, compare student results to the values in the table.

Brass: $c = 0.299$ J mol^{-1} °C^{-1}

Bronze: $c = 0.284$ J mol^{-1} °C^{-1}

Zinc: $c = 0.309$ J mol^{-1} °C^{-1}

10 pts. 4. Enter your results for the four trials in the table below.

	Metal 1		**Metal 2**	
	Trial 1	**Trial 2**	**Trial 1**	**Trial 2**
T_i**(water)**				
T_f**(water)**				
ΔT**(water)**				
T_i**(metal)**				
T_f**(metal)**				
ΔT**(metal)**				
m_{water}				
m_{metal}				
c_{metal}				
Average c_{metal}				

5. Use the table below to identify your metals.

Metal	c, **J g^{-1} °C^{-1}**
Aluminum	0.897
Brass	0.300
Bronze	0.284
Copper	0.385
Gold	0.129
Iron	0.449
Lead	0.130
Magnesium	1.023
Nickel	0.444
Silver	0.235
Zinc	0.388

5 pts. Metal 1 = ____________________

5 pts. Metal 2 = ____________________

6. For each metal, calculate the percent differences between your values of c_{metal} and the values listed in the table.

$$\text{Percent Difference} = \frac{|c_{experimental} - c_{known}|}{c_{known}} \times 100\%$$

5.3 Abstract

The abstract should include the following in paragraph form:

5 pts. • the *scientific* purpose of the experiment: *"The purpose of this experiment was to determine the specific heat capacity of two unknown metals, and use known values of heat capacities to identify the metals."*

5 pts. • the final results: *"The specific heat capacity for metal 1 was determined to be ______ and was identified as ______. The specific heat capacity for metal 2 was determined to be and was identified as ______."*

5 pts. • any comparisons to known or literature values. *"For metal 1, c had a ______% error when compared to the literature value of ______. For metal 2, c had a ______% error when compared to the literature value of ______."*

5.4 Introduction

The introduction should include the following in paragraph form.

4 pts. - Restate the goal. *See the first bullet for the abstract.*

4 pts. - Give a brief synopsis of how the goal was accomplished: *Specific heat was determined using a calorimetry experiment.*

4 pts. - Explain how you did your calculations, making sure to include the equations given in this handout, and identifying any variables in your equations: *Students must include Equations 2 and 3 from the lab handout as well as the answers to Questions 1 and 2 in Section 5.2.*

4 pts. - You should define any relevant terms required in your explanation: *Students should include all the definitions from their prelaboratory assignment.*

CHEM 140 – Aspirin Purification and Analysis Prep Sheet

1 Materials

The following materials should be provided in each equipment kit.

Table 6: Materials for each equipment kit.

Quantity	Item
2	500 mL beakers
1	hot plate
3	test tubes
1	spatula
1	test tube holder
1	glass stirring rod
1	Bunsen burner
1	hose
1	striker
1	ruler
1	50 mL beaker
1	glass spot plate
1	tweezers
1	400 mL beaker
1	watch glass to cover beaker

Also provide the following items at the front of the lab:

- glass Pasteur pipets (for the acetone)
- 2 UV lamps
- pre-cut TLC plates (PE SIL/G UV)
- open-ended capillary tubes

Two waste containers should be provided in the hood. One should be labeled "CHEM 140 – TLC Solvent: Ethyl acetate, cyclohexane, acetic acid," and the other should be labeled "CHEM 140 – Ethanol Supernant: Ethanol and water." Be sure to include your initials and the date (mm/dd/yyyy) on the labels as well.

2 Chemicals

The following chemicals will be required for each *section*.

Table 7: Chemicals for each section.

Quantity	Chemical
75 mL	95% ethanol
	ice
75 mL	acetone
150 mL	TLC solvent (200:100:1 ethyl acetate:cyclohexane:acetic acid)

Multiply the amounts listed above by the total number of sections. Then divide the total amount required into six containers, one for each hood.

3 Prelaboratory Instructions

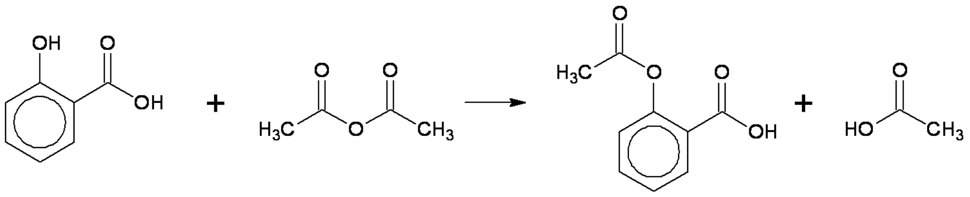
1. Demonstrate the use of a Büchner funnel apparatus for filtering.
2. Demonstrate how to make a spotting capillary.
3. Stress that no flammable liquids (like TLC solvent) should be used while Bunsen burners are lit. Consider having students make capillaries at the very beginning of the lab.
4. Students can use half of one TLC plate for their analysis. Then, if they need to repeat the TLC analysis, they can flip the plate upside-down.
5. If spots are too large, too dark, or leave streaks, the student has applied too much material to the plate and the analysis should be repeated. Either don't hold the capillary onto the plate for so long, or don't put so much solid on the spot plate so the applied solution is more dilute.
6. Use the short wavelength setting on the UV lamps.

4 Answer Key

4.1 Prelaboratory Assignment

10 pts. Week 1:

Students should show the reaction with carbons and hydrogens drawn explicitly. (I can't make the drawing on my home computer, so I've just reproduced the reaction scheme from the handout. But obviously, the C's and H's have been omitted.)



salicylic acid $C_7H_6O_3$

acetic anhydride $C_4H_6O_3$

acetylsalicylic acid (aspirin) $C_9H_8O_4$

acetic acid $C_2H_4O_2$

Week 2:

5 pts. 1. What is the purpose of recrystallization?

Recrystallization is used to purify a sample containing impurities.

5 pts. 2. What is the purpose of thin-layer chromatography?

TLC is used to assess the components of a mixture.

4.2 Observations

(*Please note that I did not get a chance to test this lab. So I'm basing these observations off of what I think was supposed to happen. -Carmen*)

Week 1:

16 pts. 1. Mass of salicylic acid for test 1 = ____________ g
Mass of salicylic acid for test 2 = ____________ g
Mass of salicylic acid for test 3 = ____________ g
Mass of salicylic acid for test 4 = ____________ g

4 pts. 2. Some of the salicylic acid dissolves when 2 mL of acetic anhydride was added.

2 pts. 3. No change was observed when the catalysts (sulfuric acid, acetic acid, hydrochloric acid) were added.

5 pts. 4. As the reaction sat in the ice bath, white, snow-like crystals appeared.

16 pts. 5. Mass of filter paper and watch glass for test 1 = ______________ g
Mass of filter paper and watch glass for test 2 = ______________ g
Mass of filter paper and watch glass for test 3 = ______________ g
Mass of filter paper and watch glass for test 4 = ______________ g

Week 2:

16 pts. 1. Mass of filter paper, watch glass and product for test 1 = ______________ g

Mass of filter paper, watch glass and product for test 2 = ______________ g

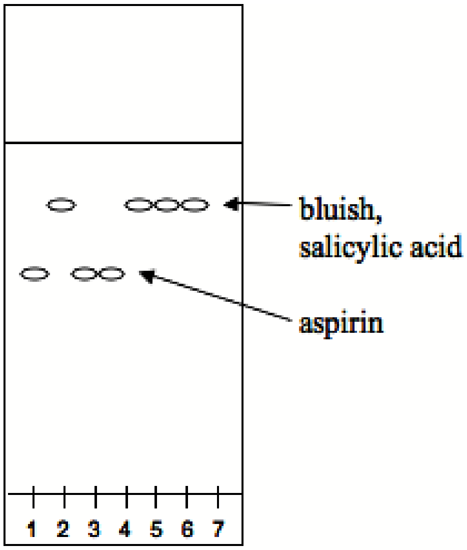
Mass of filter paper, watch glass and product for test 3 = ______________ g

Mass of filter paper, watch glass and product for test 4 = ______________ g

5 pts. 2. The white crystals from the previous week dissolve in the hot ethanol when the warm water is added to it.

5 pts. 3. Snow-like crystals form once the solution sits in the ice-water bath.

10 pts. 4. TLC was performed. After developing with a UV lamp, the plate looks like the sketch below. (The figure is not drawn to scale, but students' sketches should be.)



4.3 Data Analysis Questions

1. Theoretical yield is the amount of product you should create according to the stoichiometry of a reaction based on the amount of limiting reagent you started with. The percent yield is the percentage of product you actually made compared to the theoretical yield given that some product may be lost to side reactions. Show handwritten sample calculations for each question below:

10 pts. (a) For each of the four aspirin synthesis procedures, calculate the theoretical yield

for aspirin using salicylic acid as the limiting reagent.

A=Aspirin (Acetylsalicylic Acid); SA=Salicylic Acid

$$\text{Mass of Salicylic Acid}\left(\frac{\text{1 mole SA}}{\text{138.1 g SA}}\right)\left(\frac{\text{1 mole A}}{\text{1 mole SA}}\right)\left(\frac{\text{180.2 g A}}{\text{1 mole A}}\right)$$

$$= \text{Theoretical Yield of Aspirin}$$

10 pts. (b) Calculate the percent yield of each product.

$$\text{Percent Yield} = \frac{\text{Actual Yield}}{\text{Answer from above}} \times 100\%$$

10 pts. 2. In the recrystallization process, why do you think you used ice water?

The solubility of the product (aspirin) decreases with temperature. In order to maximize the number of crystals, the temperature of the solution should be lowered as much as possible. Ice water brings the temperature below 0 °C.

16 pts. 3. Use a ruler to measure the distance each spot and the solvent front traveled up the plate. Use these values to calculate the R_f value for each compound in each sample.

R_f (salicylic acid) $\approx$ 0.56

R_f (acetylsalicylic acid) $\approx$ 0.34

4. Spots with the same R_f values on a particular plate are likely to be identical compounds. Recall which compounds you spotted at marks 1 and 2 on the plate. Then compare the R_f values for the spots above these marks to the R_f values for the spots above marks 3-7. Identify which compound(s) are present in each sample for samples 3-7.

5 pts. *Sample 3: pure aspirin*

5 pts. *Sample 4: mostly aspirin (maybe some salicylic acid)*

5 pts. *Sample 5: mostly pure salicylic acid (maybe some aspirin)*

5 pts. *Sample 6: mostly pure salicylic acid (maybe some aspirin)*

5 pts *Sample 7: mostly pure salicylic acid (maybe some aspirin)*

10 pts. 5. The terms quantity and quality are often confused. Quantity describes how much of something you have, and quality describes how good it is. In this experiment, which procedure gave the greatest yield of product (the largest quantity)? Which procedure gave the purest aspirin (the best quality)?

Answer should match the data. Highest quantity = greatest percent yield from question 1b. Best quality = procedure 1 because it's the only one who's crude product had mostly aspirin.

10 pts. 6. Recall the purpose of recrystallization: to isolate a component in a mixture. Look at the spots above marks 3 and 4 on your TLC plate. Would you say your recrystallization worked? Provide evidence to support your answer.

The crystallization worked since the crude product (spot 4) has two spots (one belonging to the starting material, salicylic acid) and the recrystallized product (spot 3) only has one spot belonging to pure aspirin. Therefore, recrystallizing removed the salicylic acid from the mixture.

10 pts. 7. What is a catalyst? Based on your results, do all the acids work as suitable catalysts in this synthesis? Which catalyst gave you the best aspirin?

A catalyst is a compound that speeds up a reaction by reducing the activation energy for the reaction. Not all the catalysts worked well since not all reactions gave a high yield of aspirin. Sulfuric acid (from procedure 1) gave the best and purest yield.

CHEM 140 – Chemical Kinetics with the Method of Initial Rates Prep Sheet

1 Materials

Each equipment kit should contain the following:

Table 8: Materials required for each equipment kit.

Quantity	Item
1	500 mL beaker
1	250 mL beaker
1	125 mL beaker
1	10 mL graduated cylinder
1	wash bottle of DI water
1	alcohol-based 0 - 100 °C thermometer

2 Chemicals

Each *pair* of stations should have a chemicals kit in the hood that contains the chemicals in Table 9. The quantity in the table is for one *pair* of students in one section. To determine the total amount to prepare (Total Volume Required), multiply the quantity by twelve (for the twelve groups in the lab), then by the number of sections, then by 1.2 (to get a 20% excess). Round the number up to a value for which you have a volumetric flak available. Once the solution has been made, divide that total quantity into six individual containers for each chemical kit.

Table 9: Chemicals required for each pair of students.

Quantity	Concentration	Compound
27.5 mL	0.010 M	potassium iodide (KI) solution
25 mL	0.0010M	sodium thiosulfate ($Na_2S_2O_3$) solution
27.5 mL	0.040M	potassium bromate ($KBrO_3$) solution
32.5 mL	0.10M	hydrochloric acid (HCl) solution
1.5 mL		starch indicator solution (in a squirt bottle)

2.1 Making solutions from solid solutes

General procedure: The calculation to determine the mass of each solute to use has been shown below for each chemical. Once the appropriate mass has been calculated, add this mass of the solute to a large beaker with a volume greater than that of the Total Volume Required. Add most of the DI water and a stir bar. Once all the solute has dissolved and the solution has returned to room temperature, transfer the solution to a volumetric flask with a volume equal to that of the Total Volume Required. (Hold another stir bar on the outside of the beaker to keep the stir bar on the in the beaker from falling into the flask.) Rinse the beaker with DI water and add the rinse to the volumetric flask. Finally, fill the volumetric flask to the mark with DI water. Shake to mix thoroughly, and transfer to individual containers.

- Potassium Iodide

$$\text{Mass of KI} = \text{Total Volume Required} \times \left(\frac{1L}{1000mL}\right) \times \left(\frac{0.010mol}{1L}\right) \times \left(\frac{166.003g}{1mol}\right)$$

- Sodium Thiosulfate

$$\text{Mass of } \mathrm{Na_2S_2O_3} = \text{Total Volume Required} \times \left(\frac{1L}{1000mL}\right) \times \left(\frac{0.0010mol}{1L}\right) \times \left(\frac{248.17g}{1mol}\right)$$

- Potassium Bromate

$$\text{Mass of } \mathrm{KBrO_3} = \text{Total Volume Required} \times \left(\frac{1L}{1000mL}\right) \times \left(\frac{0.040mol}{1L}\right) \times \left(\frac{167.001g}{1mol}\right)$$

2.2 Making solutions from concentrated stock solutions

General procedure: The calculation to determine the volume of the concentrated solution to use has been shown below for each chemical. Once the appropriate volume has been calculated, follow the procedure for solid solutes, but use a volumetric pipet to add the correct volume of the concentrated solution to the beaker. (If you are adding more than 50 mL of the concentrated solution, you can use a graduated cylinder, but be as accurate as possible when measuring the correct volume.)

- Hydrochloric Acid

$$\text{Volume of concentrated HCl} = \text{Total Volume Required} \times \left(\frac{0.10M}{12.1M}\right)$$

3 Prelaboratory Lecture

1. Go over the prelaboratory assignment (see rubric). Explain how the method of initial rates gives the order of the reaction with respect to each reactant and the overall reaction order.

2. Explain the concept of a rate law. (You can use the example from the handout.)
3. Go over the basic reaction and the need for the addition of the clock reaction.
4. You may want to go over the stoichiometric relation between the two reactions to show how the amount of $Na_2S_2O_3$ that's added dictates the amount of BrO_3^- that's consumed. Although, this is part of their calculation.
5. Briefly describe the procedure, stressing the need to rinse the cylinder between the addition of each reagent. Also, if there is a lot of water remaining in the cylinder, this will affect the initial concentrations of the reactants and also the rates.
6. Explain that students will collect data for the first four reactions, then do all their calculations. Once their calculations are complete, they will try Reaction 5 to test their predicted reaction time.

4 Post-laboratory Procedures

1. Check each student's Data Sheet if they have cleaned their workspace and put their chemicals away.
2. Make sure students know what they must do for the laboratory assignment. This experiment requires a full write-up, and the expectations for what they should include in each section (abstract, introduction, results and discussion) is provided at the end of the handout (before the data sheet). They should also use their old lab handouts as a guide.

Prelaboratory Assigment

2 pt. 1. Order with respect to reactant A: 1

Explanation: *As the concentration of A is doubled from trial 1 to trial 2, the rate doubles.*

2 pt. 2. Order with respect to reactant B: 0

Explanation: *As the concentration of B is doubled from trial 2 to trial 3, the rate remains the same.*

4 pts. 3. Order with respect to reactant C: 3

Explanation: *As the concentration of C is doubled from trial 4 to trial 1, the rate increases by a factor of 8.* ($2^3 = 8$)

2 pts. 4. Overall Reaction Order: 4

Explanation: $1 + 0 + 3 = 4$

Partner's Name: ______________________________

5 Data Sheet

☐ Equipment Kit #__________

☐ Chemical Kit #__________

1 pt. Reaction 1

Temperature: ______________

Reaction time (in seconds): $\sim$ 234 s

1 pt. Reaction 2

Temperature: ______________

Reaction time (in seconds): $\sim$ 114 s

1 pt. Reaction 3

Temperature: ______________

Reaction time (in seconds): $\sim$ 111 s

1 pt. Reaction 4

Temperature: ______________

Reaction time (in seconds): $\sim$ 54 s

1 pt. If the temperature varied for each reaction, report the average temperature here: ______________

6 Calculations

6.1 Reactant Concentration

For each reaction, you must determine the concentration of the three reactants BrO_3^-, I^-, and H^+ initially present in the reaction beaker. Since each reactant was diluted (remember that other solutions were added in!), the concentration is calculated using the dilution equation

$$c_1V_1 = c_2V_2 \tag{3}$$

where c_1 and c_2 are the concentrations of the substance before and after it was diluted, and V_1 and V_2 are the volumes of the substance before and after it was diluted. For example, in *Reaction 2*, 5 mL of 0.010 M KI was used. The total volume of the solution (after all the reactants from Beaker A and Beaker B were added together) was 25 mL. So the concentration of KI (and therefore I^-) is 0.002 M as shown below.

$$c_2 = \frac{c_1V_1}{V_2} \tag{4}$$

$$[\mathrm{I}^-] = [\mathrm{KI}] = \frac{(0.010\ M)(5\ \mathrm{mL})}{25\ \mathrm{mL}} = 0.002\ M \tag{5}$$

In the space below, show a sample calculation of each initial ion concentration for *Reaction 1*. Then, fill in Table 10 with the the initial concentrations for all the ions in all the reactions. (The rate will be added to the table in the next section.)

Reaction 1

1 pt. $[BrO_3^-]$:

$$[\mathrm{BrO_3^-}] = [\mathrm{KBrO_3}] = \frac{(0.040M)(5mL)}{25mL} = 0.008M$$

1 pt. $[I^-]$:

$$[\mathrm{I}^-] = [\mathrm{KI}] = \frac{(0.010M)(5mL)}{25mL} = 0.002M$$

1 pt. $[H^+]$:

$$[\mathrm{H}^+] = [\mathrm{HCl}] = \frac{(0.10M)(5mL)}{25mL} = 0.02M$$

6 pts. Table

Reaction	$[BrO_3^-]$	$[I^-]$	$[H^+]$	**Rate**
1	0.008 M	0.002 M	0.02 M	$1.42 \times 10^{-7} Ms^{-1}$
2	0.016 M	0.002 M	0.02 M	$2.92 \times 10^{-7} Ms^{-1}$
3	0.008 M	0.004 M	0.02 M	$3.00 \times 10^{-7} Ms^{-1}$
4	0.008 M	0.002 M	0.04 M	$6.17 \times 10^{-7} Ms^{-1}$

Table 10: Initial concentrations of BrO_3^-, I^-, and H^+, and the rates of Reactions 1-4.

6.2 Reaction Rate

The rate of each reaction is calculated by dividing the change in BrO_3^- concentration when the indicator turns blue by the time it took for the color change to occur. To determine how much BrO_3^- was consumed, we have to examine the amount of $S_2O_3^{2-}$ that was used since it is this species that controls when the solution turns blue.

Calculate the initial concentration of $S_2O_3^{2-}$ as you did for BrO_3^-, I^-, and H^+ below. (Note: it is the same for every reaction!)

1 pt. $[S_2O_3^{2-}]$:

$$[S_2O_3^{2-}] = [Na_2S_2O_3] = \frac{(0.0010M)(5mL)}{25mL} = 0.0002M$$

2 pts. We will now use stoichiometry to determine how much BrO_3^- this corresponds to. Fill in the stoichiometric coefficients below to find out how many BrO_3^- ions are consumed for every 1 $S_2O_3^{2-}$ ion.

$$1\ S_2O_3^{2-} \left(\frac{\boxed{1}\quad I_2}{\boxed{2}\ S_2O_3^{2-}} \right) \left(\frac{\boxed{1}\ BrO_3^-}{\boxed{3}\quad I_2} \right) = \frac{1}{6}\ BrO_3^-$$

Finally, multiply your result by the concentration of $S_2O_3^{2-}$ that you calculated to get the change in bromate concentration, $\Delta[BrO_3^-]$.

1 pt. $\Delta[BrO_3^-]$:

$$\Delta[BrO_3^-] = \frac{1}{6} \times 0.0002M = 3.33 \times 10^{-}5M$$

The rate of reaction is $\Delta[\mathrm{BrO_3^-}]/\Delta\mathrm{time}$. Divide $\Delta[\mathrm{BrO_3^-}]$ by the time each reaction took to turn blue to calculate the rate of each reaction, and record your answers in Table 10. (Don't forget your units!)

6.3 Reaction Order

The order of the reaction with respect to each reactant is found by analyzing the data in Table 10 in a manner similar to that described in the Introduction. The orders for this reaction should be small whole numbers. Determine the reaction order with respect to each reactant below, and explain your rationale. Also, calculate the overall reaction order.

1 pt Order with respect to BrO_3^-: 1

Explanation: *As the concentration of BrO_3^- is doubled from reaction 1 to reaction 2, the rate doubles.*

1 pt. Order with respect to I^-: 1

Explanation: *As the concentration of I^- is doubled from reaction 1 to reaction 3, the rate doubles.*

1 pt. Order with respect to H^+: 2

Explanation: *As the concentration of H^+ is doubled from reaction 1 to reaction 4, the rate is quadrupled.*

1 pt. Overall Reaction Order: 4

Explanation: $1 + 1 + 2 = 4$

6.4 Rate Law

Now that you know the order of the reaction with respect to each reactant, write the rate law for the reaction in the space below. (Use Equation **??** as a guide where x, y, and z are the orders of the reaction.)

1 pt. Rate Law:

$$Rate = k[\mathrm{BrO_3^-}][\mathrm{I^-}][\mathrm{H^+}]^2$$

6.5 Rate Constant

1 pt. The rate constant is determined by rearranging the rate law to solve for k. Show your algebra below:

$$k = \frac{Rate}{[\mathrm{BrO_3^-}][\mathrm{I^-}][\mathrm{H^+}]^2}$$

1 pt. Using your equation above, calculate the rate constant for *Reaction 1* by plugging in the rate and initial concentrations from Table 10. (Don't forget to keep track of the units!)

$$k = \frac{1.42 \times 10^{-7} M s^{-1}}{(0.008M)(0.002M)(0.02M)^2} = 22.3M^{-3}s^{-1}$$

Now, calculate k for the remaining 3 reactions and record your results in Table 11. (Don't forget your units!) Then, determine the average rate constant of all four reactions.

Reaction	**Rate Constant**
1	$22.18M^{-3}s^{-1}$
2	$22.81M^{-3}s^{-1}$
3	$23.44M^{-3}s^{-1}$
4	$24.10M^{-3}s^{-1}$
Average	$23.13M^{-3}s^{-1}$

Table 11: Rate constants for Reactions 1-4.

6.6 Theoretical Reaction Time for Reaction 5

Calculate the initial concentrations of BrO_3^-, I^-, and H^+ for Reaction 5 given the volumes and concentrations in Table **??**.

$[BrO_3^-]$:

$$[\mathrm{BrO_3^-}] = [\mathrm{KBrO_3}] = \frac{(0.040M)(2.5mL)}{25mL} = 0.004M$$

$[I^-]$:

$$[\mathrm{I^-}] = [\mathrm{KI}] = \frac{(0.010M)(2.5mL)}{25mL} = 0.001M$$

$[H^+]$:

$$[\mathrm{H^+}] = [\mathrm{HCl}] = \frac{(0.10M)(7.5mL)}{25mL} = 0.03M$$

1 pt Now, calculate the theoretical rate of the reaction by plugging these values into your rate law from Section 6.4. (Use your averaged rate constant from Table 11 for k.):

$$Rate = 23.13M^{-3}s^{-1}(0.004M)(0.001M)(0.03M)^2 = 8.33 \times 10^{-8}Ms^{-1}$$

1 pt. In the space below, rearrange the equation $Rate = \Delta[\mathrm{BrO_3^-}]/\Delta\mathrm{time}$, to solve for Δtime:

$$\Delta\mathrm{time} = \frac{\Delta[\mathrm{BrO_3^-}]}{Rate}$$

1 pt. Finally, plug in the value you used for $\Delta[\mathrm{BrO_3^-}]$ from Section 6.2 and the theoretical rate you calculated above to calculate Δtime:

$$\Delta\mathrm{time} = \frac{3.33 \times 10^{-5}M}{8.33 \times 10^{-8}Ms^{-1}} = 400s$$

Theoretically, this is the amount of time it will take for Reaction 5 to turn blue. Now, test your result as directed in Section **??**.

1 pt. Reaction time (in seconds): ______________

1 pt. Calculate the percent error for your result using Equation **??**:

$$\text{Percent Error} = \frac{|t_{\mathrm{theor}} - t_{\mathrm{meas}}|}{t_{\mathrm{theor}}} \times 100\%$$

Abstract Usually, the abstract is only about 5 lines long, although the exact length is not critical. Be very specific and use short sentences. (Don't try to make it too "flower-y.") The abstract should include the following in paragraph form:

- 3 pts. State the *scientific* purpose of the experiment.
- 3 pts. State the reaction you studied.
- 4 pts. State the final result (the rate law with the rate constant). Make sure your rate constant has the proper units!

Introduction The introduction is written in paragraph form and gives the background information about your experiment.

- 3 pts. Restate the goal.
- 4 pts. Give a brief synopsis of how the goal was accomplished. (Don't give the whole procedure with volumes and concentrations of solutions, just indicate what method was used, initial rates in this case, and explain what that means.)
- 4 pts. Explain, in paragraph form, how you did your calculations, making sure to include the equations given in this handout, and identify any variables in your equations.
- 4 pts. You should define any relevant terms required in your explanation. (Rate, rate law, rate constant, etc.)
- Be concise and "to the point." Don't try to make your writing too "flower-y."

Results Your Results section should contain a data summary table and a description of your data in paragraph form. You should use complete sentences that are grammatically correct and check your spelling.

- 8 pts. Generate tables in a Word document to summarize the calculated results in Tables 10 and 11. Be sure your table is labeled correctly and has an appropriate caption.
- 7 pts. Your paragraph can simply be 1-2 sentences to reference the table in this section. You should also describe the data. (Did the reaction rate increase or decrease as the concentration of the different reactants changed? Was the change significant? Did the rate double? triple? stay the same?)

Discussion In paragraph form, discuss how you analyzed your data. Include the following:

- 5 pts. Explain how you determined the order of the reaction with respect to each reactant and the overall order of the reaction. (This should be similar to your explanations on page 50).
- 5 pts. Explain how you calculated k, and give your averaged result.
- 5 pts. State the determined rate law.
- 5 pts. Discuss how you calculated the theoretical time Reaction 5 would take, and give the percent error for your experimental value.

CHEM 140 – The Relationship Between Kinetics and Equilibrium Prep Sheet

1 Laboratory Preparation

There are no chemicals or materials to prepare for this exercise. Students will need cards marked "P" on one side and "R" on the other. Cards should be bundled into sets of 18 with a rubber band with one set for each group.

2 Prelaboratory Instructions

1. Retrieve the computer cart.
2. Students may not have covered much about equilibrium at this stage, so some review of formulas may be necessary.
3. It is important to stress the idea that once equilibrium is reached, the rates of the forward and reverse reactions are equal. Students often have the misconception that the reactions cease to occur at this point.
4. You can do Case 1(a) together as a class. It is very short, but it will be useful in helping students to understand how to determine the number of cards to flip over.
5. It may be difficult for students to arrive at the equation $K = \frac{k_f}{k_r}$ on their own, but it is important for them to think about this and take a crack at deriving it independently. Try not to just give the answer away.
6. It's best if students plot as they go, but if this is not possible, they can plot at the end of the card-flipping exercise or they can plot at home. Make sure they understand how to plot *two* series on one graph!

3 Postlaboratory Instructions

Please make sure the students return the computers to the cart properly. Then return the cart to where it belongs.

Partner's Name:

Each question is worth 2 points (22 questions, 44 points total). Each table is worth 5 points (6 tables, 30 points). Each graph is worth 5 points (5 graphs, 25 points). (Of course, this only adds up to 99 points. Just give the student one point for free.)

4 Data Sheet

Table 12: Equilibrium: Case 1(a), $k_f = k_r = 0.5$

Time	**[R]**	**[P]**	**Q**	**Forward Rate** $= k_f$**[R]**	**Reverse Rate** $= k_r$**[P]**	**Net Rate** $= k_f$**[R]**$-k_r$**[P]**
1	16	0	0	8	0	8
2	8	8	1	4	4	0
3						
4						
5						

1. Calculate the equilibrium constant using $K = [\mathrm{P}]_{eq}/[\mathrm{R}]_{eq}$.

$$K = \frac{8}{8} = 1$$

2. What happens to the rate of the forward reaction as you approach equilibrium?

   It decreases.

3. What happens to the rate of the reverse reaction as you approach equilibrium?

   It increases.

4. What observation can you make about the rate of both forward and reverse reactions at equilibrium?

   They are equal.

5. If the rate of the forward and reverse reactions were doubled, what would happen to the equilibrium constant?

   It would remain the same.

Table 13: Equilibrium: Case 1(b), $k_f = k_r = 0.25$

Time	**[R]**	**[P]**	**Q**	**Forward Rate** $= k_f$**[R]**	**Reverse Rate** $= k_r$**[P]**	**Net Rate** $= k_f$**[R]**$-k_r$**[P]**
1	16	0	0	4	0	4
2	12	4	0.33	3	1	2
3	10	6	0.60	2	1	1
4	9	7	0.78	2	1	1
5	8	8	1	2	2	0

6. Calculate the equilibrium constant for the reaction using $K = [\mathrm{P}]_{eq}/[\mathrm{R}]_{eq}$.

$$K = \frac{8}{8} = 1$$

7. Which reaction reached equilibrium the fastest, (a) or (b)?

   (a) is faster because it took less iterations to reach equilibrium.

8. Is it possible to predict the rate of a reaction if you know its equilibrium constant? Explain.

   No. Even though K was the same for the last two reactions, the reactions reached equilibrium at different rates.

9. What is the mathematical relationship between K (the equilibrium constant) and the rate constants k_f and k_r? (Hint: Remember that at equilibrium, the rate of the forward reaction is equal to the rate of the reverse reaction, so $k_f[\text{R}] = k_r[\text{P}]$.)

$$\text{Forward Rate} = \text{Reverse Rate}$$
$$k_f[\text{R}] = k_r[\text{P}]$$
$$K = \frac{[\text{P}]}{[\text{R}]} = \frac{k_f}{k_r}$$

10. Calculate K for both reactions (a) and (b) using your equation from Question 9.

    (a) $K = \frac{0.5}{0.5} = 1$

    (b) $K = \frac{0.25}{0.25} = 1$

11. Calculate the free energy of the reaction using Equation 7. Use $R = 8.314$ J/molK and $T = 293$ K. Is the reaction endergonic or exergonic?

$$\Delta G = -RT\ln K = -(8.314\,\frac{\text{J}}{\text{mol K}})(293\text{ K})\ln(1) = 0$$

The reaction is neither endergonic nor exergonic because ΔG is neither positive nor negative.

Table 14: Equilibrium: Case 2, $k_f = 0.25 < k_r = 0.5$

Time	**[R]**	**[P]**	**Q**	**Forward Rate** $= k_f$**[R]**	**Reverse Rate** $= k_r$**[P]**	**Net Rate** $= k_f$**[R]**$-k_r$**[P]**
1	18	0	0	4	0	4
2	14	4	0.29	3	2	1
3	13	5	0.38	3	2	1
4	12	6	0.5	3	3	0
5						
6						
7						

12. Calculate the equilibrium constant for the reaction using $K = [\mathrm{P}]_{eq}/[\mathrm{R}]_{eq}$.

$$K = \frac{5}{12} = 0.5$$

13. Calculate the equilibrium constant for the reaction using your equation from Question 9.

$$K = k_f k_r = \frac{0.25}{0.5} = 0.5$$

14. Calculate the Gibbs free energy of the reaction using Equation 7 and the same values for R and T as in Question 11. Is the reaction endergonic or exergonic?

$$\Delta G = -RT \ln K = -(8.314\ \frac{\mathrm{J}}{\mathrm{mol\ K}})(293\ \mathrm{K}) \ln(0.5) = 1689\ \frac{\mathrm{J}}{\mathrm{mol}}$$

The reaction is endergonic because ΔG is positive.

15. Does a reaction with a positive ΔG have to be endo*thermic* (have a positive ΔH)? Does a reaction with a negative ΔG have to be exo*thermic* (have a negative ΔH)? Use Equation **??** to explain your answer.

    No. $\Delta G = \Delta H - t\Delta S$ *so if* ΔH *is positive,* ΔG *could still be negative if the quantity* $T\Delta S$ *is greater than the value for* ΔH.

Table 15: Equilibrium: Case 3(a), $k_f = 1/3 > k_r = 1/6$

Time	**[R]**	**[P]**	**Q**	**Forward Rate** $= k_f$**[R]**	**Reverse Rate** $= k_r$**[P]**	**Net Rate** $= k_f$**[R]**$-k_r$**[P]**
1	18	0	0	6	0	6
2	12	6	0.5	4	1	3
3	9	9	1	3	1	2
4	7	11	1.57	2	1	1
5	6	12	2	2	2	0
6						
7						
8						

16. Calculate the equilibrium constant for the reaction using $K = [\mathrm{P}]_{eq}/[\mathrm{R}]_{eq}$.

$$K = \frac{12}{6} = 2$$

17. Calculate the equilibrium constant for the reaction using your equation from Question 9.

$$K = \frac{k_f}{k_r} = \frac{1/3}{1/6} = 2$$

18. Calculate the Gibbs free energy of the reaction using Equation 7 and the same values for R and T as in Question 11. Is the reaction endergonic or exergonic?

$$\Delta G = -RT \ln K = -(8.314 \, \frac{\text{J}}{\text{mol K}})(293 \text{ K}) \ln(2) = -1689 \, \frac{\text{J}}{\text{mol}}$$

The reaction is exergonic because ΔG *is negative.*

Table 16: Equilibrium: Case 3(b), $k_f = 1/3 > k_r = 1/6$

Time	**[R]**	**[P]**	**Q**	**Forward Rate** $= k_f$**[R]**	**Reverse Rate** $= k_r$**[P]**	**Net Rate** $= k_f$**[R]**$-k_r$**[P]**
1	0	18	–	0	3	-3
2	3	15	5	1	2	-1
3	4	14	3.5	1	2	-1
4	5	13	2.6	1	2	-1
5	6	12	2	2	2	0
6						
7						
8						

19. What effect did starting the reaction with all product and no reactant have on the equilibrium?

No change. K remains the same.

Table 17: Le Châtelier's Principle: $k_f = 0.25$ and $k_r = 0.5$

	Time	**[R]**	**[P]**	**Q**	**Forward Rate** $= k_f$**[R]**	**Reverse Rate** $= k_r$**[P]**	**Net Rate** $=$ k_f**[R]**$-k_r$**[P]**
	1	18	0	0	4	0	4
	2	14	4	0.29	3	2	1
	3	13	5	0.38	3	2	1
	4	12	6	0.5	3	3	0
	5						
remove [P]	6	12	0	0	3	0	3
	7	9	3	0.33	2	1	1
	8	8	4	0.5	2	2	0
	9						
	10						
add 6 [R]	11	14	4	0.29	3	2	1
	12	13	5	0.38	3	2	1
	13	12	6	0.5	3	3	0
	14						
remove [P]	15	12	0	0	3	0	3
	16	9	3	0.33	2	1	1
	17	8	4	0.5	2	2	0
	18						
	19						
add 6 [R]	20	14	4	0.29	3	2	1
	21	13	5	0.38	3	2	1
	22	12	6	0.5	3	3	0
	23						

20. What effect does removing product from the reaction have on the equilibrium constant?

    There is no effect on K. It remains the same.

21. What effect does adding more reactant to the reaction have on the equilibrium constant?

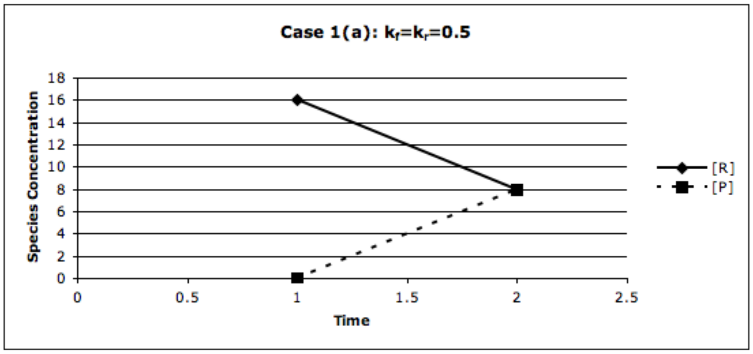
    There is no effect on K. It remains the same.

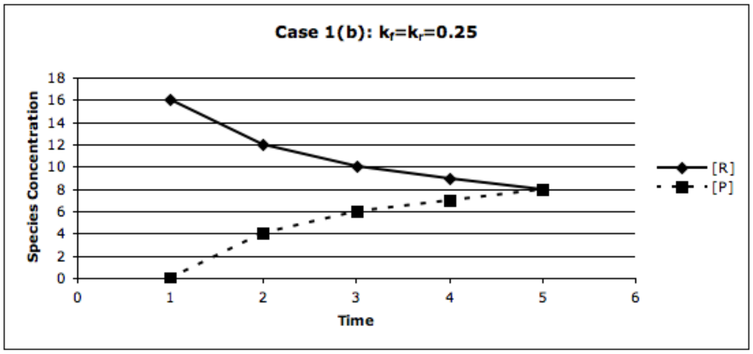
22. How could you improve the yield (amount of product produced) on a reaction that has an equilibrium constant less than one ($K < 1$)?

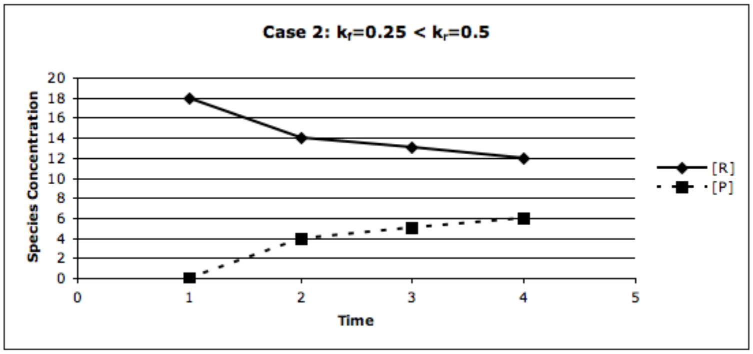
    Either remove the product that has already been produced OR add more reactant.

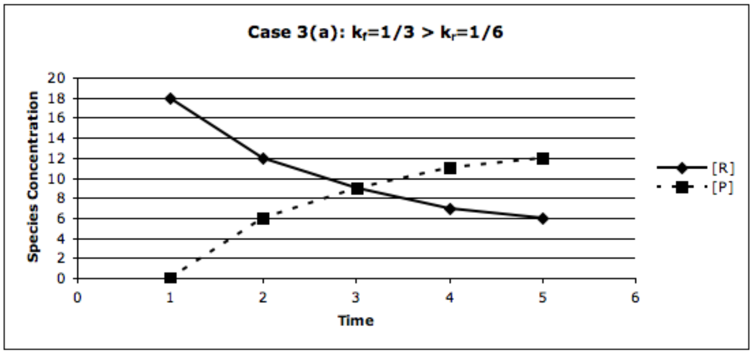
5 Graphs

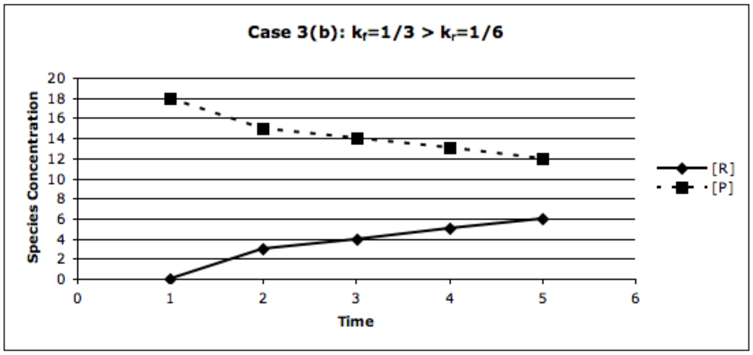
The graphs are displayed below. There are five altogether.






Case 2: k_f=0.25 < k_r=0.5
Species Concentration
0
2
4
6
8
10
12
14
16
18
20
Time
0
1
2
3
4
5
[R]
[P]


Case 3(a): k_f=1/3 > k_r=1/6
Species Concentration
0
2
4
6
8
10
12
14
16
18
20
Time
0
1
2
3
4
5
6
[R]
[P]


Case 3(b): k_f=1/3 > k_r=1/6
Species Concentration
0
2
4
6
8
10
12
14
16
18
20
Time
0
1
2
3
4
5
6
[R]
[P]

CHEM 140 – Determining the Equilibrium Constant for $I_2(aq) + I^-(aq) \rightleftharpoons I_3^-(aq)$ – Prep Sheet

1 Equipment

Each equipment kit will require the following items:

Table 18: Materials list.

Quantity	Item
1	10 mL graduated cylinder
1	50 mL graduated cylinder
1	100 mL graduated cylinder
1	250 mL Erlenmeyer flask
1	#6 rubber stopper to fit 250 mL Erlenmeyer
5	125 mL Erlenmeyer flasks
2	#5 rubber stoppers to fit 125 mL Erlenmeyers
1	50 mL buret
1	buret clamp
1	ring stand
1	funnel to fit the top of the buret

At the front of the lab you should also provide a box of plastic transfer pipets.

Also provide a large separatory funnel in a hood labeled "I_2, I^-. I_3^-, water and hexanes" and a glass waste bottle labeled "Iodine and acetone waste" for waste collection. At the end of the week, aqueous solutions (the bottom layer of the separatory funnel) can be poured down the drain with copious amounts of water. Acetone and hexanes waste can be combined and should be brought to the stockroom for disposal. Be sure the waste is labeled appropriately.

2 Chemicals

Each section will require the following chemicals.

Table 19: Chemicals list.

Quantity	**Concentration**	**Chemical**
1380 mL	0.15 M	potassium iodide (KI)
480 mL	0.04 M	iodine (I_2) in hexanes
720 mL	0.1 M	hydrochloric acid (HCl)
915 mL	0.03 M	sodium thiosulfate ($Na_2S_2O_3$)
50 mL	1%	starch solution

To determine the total volume of each solution you need to prepare, multiply the quantity above by the total number of lab sections and then by 1.2 to provide a 20% excess. Volumes should then be rounded to a volume for which there is volumetric glassware available. All solutions should be prepared in a large beaker and stirred until the solid is dissolved or the solution is completely mixed. This solution can then be transferred to a volumetric flask and brought to the mark with DI water. Finally, solutions should be divided into about six containers, one for each hood in the laboratory.

Calculations for preparing solutions

Potassium Iodide, KI

$$\text{Mass of KI} = \text{Total Volume Required in mL} \times \frac{1\text{ L}}{1000\text{ mL}} \times \frac{0.15\text{ mol}}{1\text{ L}} \times \frac{166.00\text{ g}}{1\text{mol}}$$

Iodine, I_2

$$\text{Mass of } I_2 = \text{Total Volume Required in mL} \times \frac{1\text{ L}}{1000\text{ mL}} \times \frac{0.04\text{ mol}}{1\text{ L}} \times \frac{253.8\text{ g}}{1\text{mol}}$$

This solution should be prepared using hexanes as the solvent. Prepare this solution in the hood and be sure to wear gloves and goggles.

Hydrochloric Acid, HCl

$$\text{Volume of Concentrated HCl} = \text{Total Volume Required in mL} \times \frac{0.1\text{ M}}{12.1\text{ M}}$$

As a safety precaution, add the acid to the water, not the other way around.

Sodium Thiosulfate, $Na_2S_2O_3$

$$\text{Mass of Na}_2\text{S}_2\text{O}_3 = \text{Total Volume Required in mL} \times \frac{1\text{ L}}{1000\text{ mL}} \times \frac{0.03\text{ mol}}{1\text{ L}} \times \frac{248.18\text{ g}}{1\text{mol}}$$

Note that the molecular weight above is for the hydrated salt. When in doubt, use the molecular weight on the bottle to perform your calculations.

Starch Solution

$$\text{Mass of starch} = \frac{\text{Total Volume Required in mL}}{100}$$

This solution should be heated to a gentle boil for a few minutes, then allowed to cool to room temperature before adding it to a volumetric flask.

3 Prelaboratory Instructions

1. Go over an outline of the experiment, and define vocabulary words in bold in the handout: equilibrium, equilibrium constant, titration, buret, titrant, endpoint, immiscible, distribution coefficient, decant, and separatory funnel.
2. Go over the prelab answers, pointing out how they can be used as a guide in computing the calculations at the end of the experiment.
3. Go over safety precautions:
    (a) Hydrochloric acid (HCl) will be used in this experiment. While the concentration is low, HCl is still a strong acid and will burn the skin or eat holes in your clothing. Use caution when handling this solution. Rinse your skin with copious amounts of water if it comes in contact with HCl and notify your instructor immediately. Thoroughly rinse the benchtop or floor with water should there be a small spill. For a large spill, notify your instructor immediately.
    (b) Hexanes is a volatile and flammable organic solvent. All hexanes solutions should be kept in the hood, and Erlenmeyer flasks containing hexanes should be stoppered when not in use.
    (c) Gloves and goggles should be worn at all times throughout the laboratory period.
4. Plastic transfer pipets should only be used for aqueous solutions.

4 Answer Keys

4.1 Prelaboratory Assignment

In a titration, 10.4 mL of 0.05 M $Na_2S_2O_3$ was required to turn 15 mL of an aqueous solution containing I_2, I_3^- and a starch solution from dark blue to colorless.

2 pts. 1. How many moles of $S_2O_3^{2-}$ were added?

$$10.4\text{ mL} \times \frac{1\text{ L}}{1000\text{ mL}} \times \frac{0.05\text{ mol}}{1\text{ L}} \times \frac{1\text{ mol } S_2O_3^{2-}}{1\text{ mol } Na_2S_2O_3} = 0.00052\text{ mol } S_2O_3^{2-}$$

2 pts. 2. How many moles of I_2 and I_3^- were present in the solution altogether?

$$0.00052\text{ mol } S_2O_3^{2-} \times \frac{1\text{ mol I species}}{2\text{ mol } S_2O_3^{2-}} = 0.00026\text{ mol I species}$$

3 pts. 3. What is the concentration of the total reactive iodine species? (I_2 and I_3^- together)

$$\frac{0.00026\text{ mol I species}}{15\text{ mL I solution}} \times \frac{1000\text{ mL}}{1\text{ L}} = 0.01733\text{ M}$$

3 pts. 4. From a separate titration with I_2 in hexanes, you learned that the concentration of I_2 in the water phase is 1.4×10^{-5}. What is the concentration of I_3^-?

$$0.01733\text{ M} - 1.4 \times 10^{-5}\text{ M} = 0.01732\text{ M}$$

4.2 Observations

2 pts. • The I_2 solution in hexanes is a bright pink/magenta color. The KI solution is colorless or may be a light yellow color.

2 pts. • When mixed, there are two layers with hexanes as the top layer. The whole solution appears dark brown or red, but in actuality, the hexanes layer is still magenta and the aqueous layer is a dark yellow/brown.

1 pt. • Concentration of $Na_2S_2O_3$ = ______________ M

Titration of the hexanes layer

Trial 1

1 pt. • Volume of hexanes layer = ______________ mL

2 pts. • When starch is added, both layers in the Erlenmeyer turn dark blue.

1 pt. • Initial buret volume = ______________ mL

1 pt. • Final buret volume = ______________ mL

Trial 2

1 pt. • Volume of hexanes layer = ______________ mL

1 pt. • Initial buret volume = ______________ mL

1 pt. • Final buret volume = ______________ mL

Titration of the water layer

Trial 1

1 pt. - Volume of water layer = ______________ mL

2 pts. - When starch is added the water in the Erlenmeyer turns dark blue.

1 pt. - Initial buret volume = ______________ mL

1 pt. - Final buret volume = ______________ mL

Trial 2

1 pt. - Volume of water layer = ______________ mL

1 pt. - Initial buret volume = ______________ mL

1 pt. - Final buret volume = ______________ mL

4.3 Calculations

Calculating $[I_2]_{\text{water}}$

3 pts. 1. Calculate the volume of $Na_2S_2O_3$ solution added in each hexanes titration. ($V_{\text{final}} - V_{\text{initial}}$)

Each volume is calculated by subtracting the initial buret reading from the final buret reading.

5 pts. 2. For each hexanes titration, convert the volume above to the number of moles of $S_2O_3^{2-}$.

$$n_{S_2O_3^{2-}} = \text{Volume in mL} \times \frac{1\ \text{L}}{1000\ \text{mL}} \times [\text{NaS}_2\text{O}_3] \times \frac{1\ \text{mol}\ S_2O_3^{2-}}{1\ \text{mol}\ Na_2S_2O_3}$$

5 pts. 3. For each hexanes titration, calculate the number of moles of I_2 the $S_2O_3^{2-}$ reacted with using the reaction stoichiometry.

$$n_{I_2,\text{hexanes}} = n_{S_2O_3^{2-}} \times \frac{1\ \text{mol}\ I_2}{2\ \text{mol}\ S_2O_3^{2-}}$$

5 pts. 4. Calculate the concentration (molarity) of I_2 that was in the hexanes by dividing the answers above by the volumes of hexanes that were added to the flask in liters (approx. 0.010 L).

$$[I_2]_{\text{hexanes}} = \frac{n_{I_2,\text{hexanes}}}{V_{\text{hexanes}}}$$

4 pts. 5. Average the molarities of I_2 from the hexane solutions.

Average the results from the previous question.

5 pts. 6. Use the distribution equation to calculate the concentration of I_2 in the water phase.

$$[\mathrm{I_2}]_{\text{water}} = \frac{[\mathrm{I_2}]_{\text{hexanes}}}{34}$$

Calculating $[I_3^-]$

3 pts. 7. Calculate the volume of $Na_2S_2O_3$ solution added in each water layer titration. ($V_{\text{final}} - V_{\text{initial}}$)

Each volume is calculated by subtracting the initial buret reading from the final buret reading.

5 pts. 8. For each water layer titration, convert the volume above to the number of moles of $S_2O_3^{2-}$.

$$n_{\mathrm{S_2O_3^{2-}}} = \text{Volume in mL} \times \frac{1\ \mathrm{L}}{1000\ \mathrm{mL}} \times [\mathrm{NaS_2O_3}] \times \frac{1\ \mathrm{mol\ S_2O_3^{2-}}}{1\ \mathrm{mol\ Na_2S_2O_3}}$$

5 pts. 9. For each water layer titration, calculate the total number of moles of I_2 and I_3^- (I_{total}) the $S_2O_3^{2-}$ reacted with using the reaction stoichiometry. (Recall that you can't determine the concentrations of I_2 and I_3^- independently. However, the stoichiometric ratio for $S_2O_3^{2-}$ to both reactive iodine species is still 2:1.)

$$n_{\text{I, total}} = n_{\mathrm{S_2O_3^{2-}}} \times \frac{1\ \mathrm{mol\ I_{total}}}{2\ \mathrm{mol\ S_2O_3^{2-}}}$$

5 pts. 10. Calculate the concentration (molarity) of reactive iodine species (I_2 and I_3^- together) by dividing the answers above by the volumes of water added to the Erlenmeyer in each titration (approx. 0.010 L).

$$[I]_{\text{total}} = \frac{n_{\text{I, total}}}{V_{\text{water}}}$$

4 pts. 11. Average the molarities of the total reactive iodine species.

Average the results from the previous question.

5 pts. 12. Calculate the I_3^- concentration by subtracting the concentration of I_2 in the water layer from the concentration of the total reactive iodine species.

$$[\mathrm{I}_3^-] = [I]_{\text{total}} - [\mathrm{I}_2]_{\text{water}}$$

Calculating [I⁻]

5 pts. 13. Calculate the concentration of I^- present in the water layer given that the concentration of the I_3^- is equal to the amount of I^- *consumed* in the reaction. Since the initial amount of I^- is known (this is the concentration of KI from the stock solution), the concentration of I^- at equilibrium is this amount minus the concentration of I_3^-.

$$[\mathrm{I}^-] = [\mathrm{I}^-]_o - [\mathrm{I}_3^-]$$

Calculating K

5 pts. 14. Calculate the equilibrium constant, K, from the equilibrium concentrations of I_2 (Question 6), I^- (Question 13), and I_3^- (Question 12) in the water layer at equilibrium.

$$K = \frac{[\mathrm{I}_3^-]}{[\mathrm{I}^-][\mathrm{I}_2]_{\text{water}}}$$

5 pts. 15. The literature value of the equilibrium constant for this reaction is $K = 710\ M^{-1}$. Compare your result as a percent difference using the equation below.

$$\text{Percent Difference} = \frac{|\text{Literature Value} - \text{Experimental Value}|}{\text{Literature Value}} \times 100\%$$