

Introduction

Most solubility equilibrium investigated in this course involve *ionic* compounds as opposed to molecular compounds. Remember that the way that we consider the solubility of ionic compounds is opposite to the way in which we study precipitation reactions in which the product is a solid. Consider the net ionic equation for the formation of the silver chromate precipitate:



By contrast, solubility equilibrium reactions are written from the perspective of the solid reactant dissolving into ions



The equilibrium constant for solubility equilibria is called the solubility product constant, or K_{sp} .

The solubility expression for Rx 2 above is: $K_{sp} = [\text{Ag}^+]^2 [\text{CrO}_4^{2-}]$

Note that the solid is not included in such an expression because the ionic solid's concentration does not change no matter how much solid is present.

PreLAD: *To be done BEFORE class. Show your work on this page. Circle your final answer.*

1. Write a balanced equation in the space below that would represent the solubility equilibrium for calcium hydroxide. Then write the equilibrium expression. Calculate the molar mass of calcium hydroxide. (
2. Make a data/results table on a new sheet on your Google Data Table. Set up **four** separate sections on your data table, one section each of the four methods (and method 4 is actually two tables). Do NOT make separate sheets. Think carefully about how to arrange them so they will print on one single sheet. You are expected to embed formulas for any calculations.

PreLAD calculations – similar to LAD calculations.

3. Write a balanced chemical equation in the space below that would represent the solubility equilibrium for metal(II) hydroxide, $\text{M}(\text{OH})_2$. Then write the equilibrium expression.
4. The pH of a saturated solution of saturated metal(II) hydroxide was determined to be 11.93, calculate the K_{sp} . (Method 1)
 - a. Calculate the $[\text{OH}^-]$ that is present in the solution.
 - b. Calculate the $[\text{M}^{2+}]$ that is present in the solution.
 - c. Calculate the K_{sp} .
5. After measuring out 25.0 ml of saturated metal(II) hydroxide solution into a beaker, then drying up the water, the mass of the residue remaining was 0.0139 g. (The molar mass of **metal**, $M = 98 \text{ g/mol}$) (Method 2)
 - a. Calculate the $[\text{M}^{2+}]$ that is present in the solution.
 - b. Calculate the $[\text{OH}^-]$ that is present in the solution.
 - c. Calculate the K_{sp} .

6. It took 8.43 ml of 0.020 M HCl to neutralize 25.0 ml of a saturated metal(II) hydroxide solution. (Method 4)
- Calculate the $[\text{OH}^-]$ that is present in the solution.
 - Calculate the $[\text{M}^{2+}]$ that is present in the solution.
 - Calculate the K_{sp} .
7. Calculate the mass of calcium nitrate tetrahydrate and calculate the mass of sodium hydroxide needed to make a 100.0 ml solution of each salt to produce 0.10-molar solutions. (Info needed for Method 4)

Procedure Overview

In this lab, the solubility constant of calcium hydroxide will be determined by several different methods and the results will be compared to promote discussion about which method was the most effective.

- In the *first method*, the pH of a saturated solution will be measured with a pH probe.
- In the *second method*, a known volume of a saturated solution of calcium hydroxide is put into a dry pre-massed beaker, and the water is driven off in the oven.
- In the *third method*, a volume of the saturated calcium hydroxide solution will be titrated to neutralization with a standardized concentration of HCl.
- In the *fourth method*, solutions of calcium nitrate and sodium hydroxide of known concentrations will be combined to observe a precipitate.

Materials

Procedure Method 1 & 2

- flask **with cover** with saturated calcium hydroxide solution
 - ★ KEEP THE COVER ON THE SATURATED SOL'N TO AVOID ANY SOLID FORMING
- 25 ml pipet for $\text{Ca}(\text{OH})_2$ solution with red pump
- 2× 50 ml beakers for procedure 2

Procedure Method 3

- Tap water squirt bottle.
- 250 ml flask with stopper for 0.0300 M HCl
- dropper bottles with bromothymol blue indicator
- 2× burets with stands for acid solution
- 2× stirring plates with stirring bars
- 2× 50 ml flasks for titrations

AT Center LAB Bench

- Analytic balances (4 places after the decimal)
- Extra 0.020 M HCl
- Extra saturated $\text{Ca}(\text{OH})_2$
- pH probe

Procedure Method 4

Plastic Pipet Packet for Ca^{2+} dilution

- 1× plastic pipet orange-labeled water
- 10 plastic pipets white-labeled Ca^{2+} 1–10
- 1× plastic pipet yellow-labeled 0.1 M OH^-

Plastic Pipet Packet for OH^- dilution

- 1× plastic pipet orange-labeled water
- 10 plastic pipets yellow-labeled OH^- 1–10
- 1× plastic pipet white-labeled 0.1 M Ca^{2+}

- Tap water squirt bottle.
- 125 ml flask for 0.10 M calcium nitrate solution
- 125 ml flask for 0.10 M NaOH solution
- Cotton swaps and tissues for cleaning well plates
- toothpicks for stirring
- dissecting microscopes

AT Center LAB Bench

- regular balances (3 places after the decimal)
- Extra saturated $\text{Ca}(\text{OH})_2$

Goggles should be worn at all times. No exceptions. Aprons are optional.

Method 1 Procedure – pH (Class Demonstration)

- A. Determine the pH of the saturated using the pH probe.
- B. This will be set up on the center lab bench.

Method 1 Processing the Data – pH

1. Convert the pH to pOH.
2. Use the formula $pOH = -\log[OH^-]$ to calculate $[OH^-]$.
3. Knowing the $[OH^-]$ and the stoichiometry of the solubility equation, calculate $[Ca^{2+}]$.
4. Calculate K_{sp} .

Method 2 Procedure – Drying and massing

- C. Mass a clean dry 50 ml beaker.
- D. Use the 25 ml glass graduated pipet to measure a known volume of saturated $Ca(OH)_2$ solution into the beaker (approximately 25 ml – but be sure and record the exact volume).
- E. Place the beaker into the oven overnight to allow the water to evaporate.
- F. Mass the beaker with the dry $Ca(OH)_2$.

Disposal and Clean Up

Leave the beaker when done on the tray on the center lab bench.

Method 2 Processing the Data – Drying and massing

5. Calculate the mass of calcium hydroxide.
6. Add the molar mass of calcium hydroxide and then calculate the moles of calcium hydroxide that was in the volume of saturated solution that you measured out.
7. Calculate the molarity of calcium hydroxide that was dissolved in the saturated solution.
8. Knowing the stoichiometry of the chemical reaction for the solubility, calculate $[Ca^{2+}]$ and $[OH^-]$.
9. Calculate K_{sp} .

Method 3 Procedure – Titration

- J. Using a pipet measure a known volume of saturated $Ca(OH)_2$ solution into the flask (at least 10 ml but less than 25 ml– be sure and record the exact volume). Drop in the magnetic stirring bar, and put in several drops of bromothymol blue. Add enough tap water to cover the stirring bar.
- K. Be sure you that your acid buret is nearly full. Record the initial volume of the HCl (Alert: remember to read the buret down - and we do not care how much HCl is actually in the buret, we simply care what the initial volume is. Titrate until the endpoint (color change) occurs. Record the final volume of the HCl.
- L. Repeat the titration at least two more times if time permits. All solutions may be poured down the drain.

Method 3 Processing the Data – Titration

12. Calculate the volume of HCl used to neutralize the $[OH^-]$.
13. Since H^+ and OH^- combine in a 1:1 ratio, use $MV = MV$ to calculate the $[OH^-]$.
14. Knowing the $[OH^-]$ and the stoichiometry of the solubility equation, calculate $[Ca^{2+}]$.
15. Calculate K_{sp} for each trial.

Disposal and Clean Up

Solutions may be poured down the sink with plenty of water. Please be sure and catch the stirring bar. Do not allow it to escape down the drain. Rinse your beakers and flasks with plenty of water, no need to dry them.

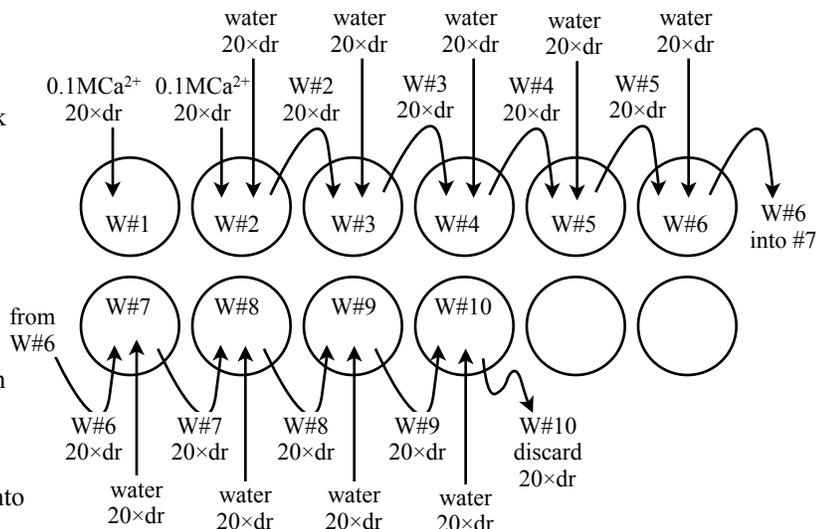
Method 4 Procedure - Successive Dilution**Procedure - Goggles should be worn at all times. No exceptions. Aprons are optional.**

- G. One group at the bench will prepare 100 ml of 0.10-molar calcium nitrate solution for both groups to use. You may use tap water to fill the flask $\frac{3}{4}$ full – swirl to dissolve the salt, then top off to the 100 ml mark using the squirt bottle. Be sure and hold the cover on and invert several times to completely mix the solution.
- H. The other group will prepare 100 ml of 0.10-molar sodium hydroxide solution for both groups to use. You may use tap water to fill the flask $\frac{3}{4}$ full – swirl to dissolve the salt, then top off to the 100 ml mark using the squirt bottle. Be sure and hold the cover on and invert several times to completely mix the solution.
- I. Clean your micro well-plate by moistening a Q-tip and wiping out two horizontal rows of 10 adjacent wells of both groups
- J. One lab group will receive a bag of pipets labelled Ca^{2+} dilution: (1) orange pipet for water, (2) a set of white pipets, one labeled 0.10 M Ca^{2+} and the remaining white pipets labeled 2 through 10, and (3) one yellow pipet labeled 0.10 M OH^- .
- K. The other lab group will receive a different bag of pipets labelled OH^- dilution: (1) orange pipet for water, (2) a set of yellow pipets, one labeled 0.10 M OH^- and the remaining yellow pipets labeled 2 through 10, and (3) one white pipet labeled 0.10 M Ca^{2+} .
- L. Use the labeled piece of paper #'s 1–10 to place under the well plate to help keep track of which solution is which.

One Group will Prepare a Series of Diluted Ca^{2+} Solutions (Other group should turn to next page.)

- M. One lab group will prepare the OH^- dilutions while the other group is preparing the Ca^{2+} dilutions. Using the #1 white pipet labeled 0.10 M Ca^{2+} , put 20 drops of the stock 0.10 M calcium nitrate into well #1 and 20 drops of the stock 0.10 M calcium nitrate into well #2 in the same row. Using the orange-labeled water pipet place 20 drops of water into each of the wells #2 – #10.
- N. Using a clean toothpick, mix the solution in well #2 thoroughly. Using white pipet #2- Ca^{2+} , suck up solution from well #2 and put 20 drops into well #3, returning any remaining solution from the pipet back into well #2.

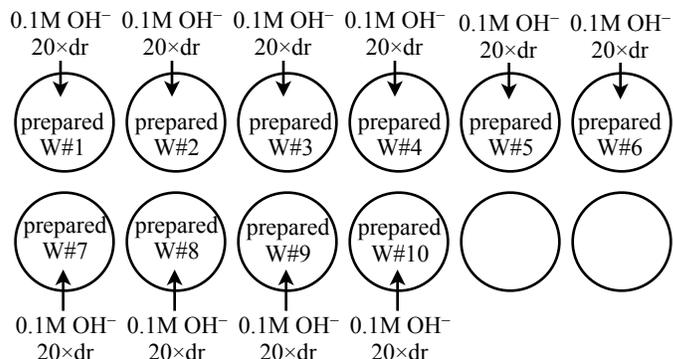
Note the number on the pipet indicates the solution that the pipet can touch, taking solution from that well # and placing into the next well # in the row. Preparing these solutions will take steady hands, close attention to detail, and careful focus.



- O. Using a different clean toothpick, mix the solution in well #3 thoroughly. Using white pipet #3- Ca^{2+} , continue the serial dilution by sucking up some of solution #3, and putting 20 drops into well #4 and returning any remaining drops of solution #3 back into well #3.
- P. Repeat this successive (aka serial) dilution technique using the appropriately labeled pipet by mixing with a clean toothpick each time and then moving 20 drops of the previous solution into the 20 drops of water in each well down the row until you get to well #10. Mix each solution with a clean toothpick, then discard 20 drops of the solution from well #10 (You can discard directly into the sink.)

Combine the series of diluted Ca^{2+} solutions with 0.10 M OH^-

- Q. Using the appropriate yellow-labeled pipet, put 20 drops of 0.10 M sodium hydroxide into each of the 10 wells that contain the prepared Ca^{2+} dilution solutions. Tip the well plates gently back and forth to mix the solutions. Do this several times over the course of the next minute or two.
- R. Allow a minute or two for any precipitates to form. Sometimes the precipitates can be hard to detect. Look at the well plate under the dissecting scope to look for solid $\text{Ca}(\text{OH})_2$ crystals forming on the surface. They will look like ice on the surface of the solution. Experiment with the light from above or below. If you are having difficulty focusing on the surface of the solutions, please call the teacher over to help.



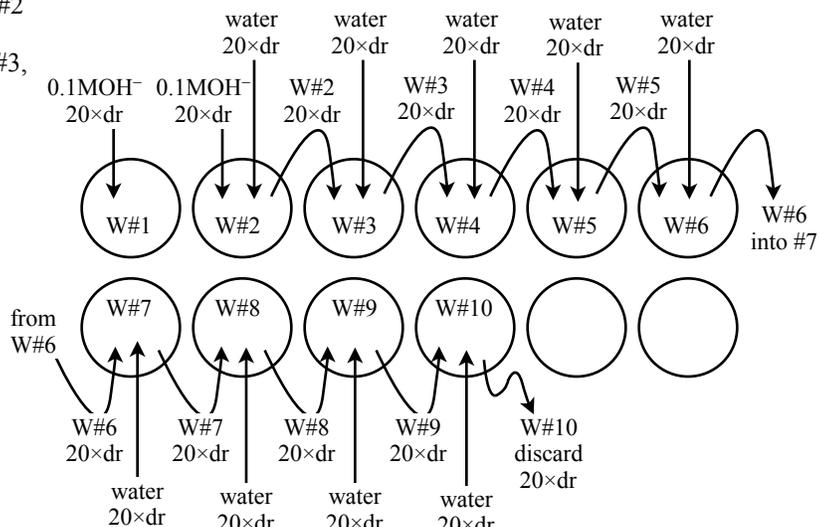
(Be sure and read the final direction on the next page.)

Determine the last solution with a precipitate and the first solution without a precipitate and **BOLD** those two rows in your Google data table. While under the scope, show your well plates to the teacher for precipitation confirmation.

Other Group will Prepare a Series of Diluted OH^- Solutions

- S. You will share your stock solutions, and one lab group will prepare the Ca^{2+} dilutions while the other group is preparing the OH^- dilutions. Using the #1 yellow pipet labeled 0.10 M- OH^- , put 20 drops of stock 0.10 M sodium hydroxide solution into well #1 and well #2. Using the orange water-labeled pipet place 20 drops of water into each of the wells #2 – #10.
- T. Using a clean toothpick, mix the solution in well #2 thoroughly. Using yellow pipet #2 OH^- , suck up solution from well #2 and put 20 drops into well #3, returning any remaining solution from the pipet back into well #2.

Note the number on the pipet indicates the solution that the pipet can touch, taking solution from that well # and placing into the next well # in the row. Preparing these solutions will take steady hands, close attention to detail, and careful focus.



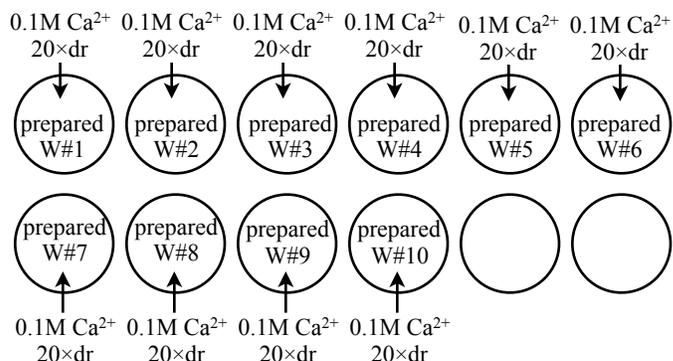
- U. Using a different clean toothpick, mix the solution in well #3 thoroughly. Using yellow pipet #3- OH^- , continue the serial dilution by sucking up some of the solution #3 and putting 20 drops of well #3 solution into #4, returning any remaining into well #3.
- V. Repeat this successive dilution technique using the appropriately labeled pipet by adding 20 drops of the previous solution into the 20 drops of water in each well down the row until you get to well #10. Mix each solution with a clean tooth pick. After mixing well #10 thoroughly discard 20 drops of the solution from well #10. (You can discard directly into the sink.)

Combine the series of diluted OH^- solutions with 0.10 M Ca^{2+}

- W. Using the white 0.01 M Ca^{2+} pipet, place 20 drops of 0.10 M calcium nitrate into each of the 10 wells prepared with diluted hydroxide solutions. Tip the well plates gently back and forth to mix the solutions. Do this several times over the course of several minutes.

- X. Allow a minute or two for any precipitates to form. Sometimes the precipitates can be hard to detect. Look at the well plate under the dissecting scope to look for solid $\text{Ca}(\text{OH})_2$ crystals forming on the surface. They will look like ice on the surface of the solution. Experiment with the light from above or below. If you are having difficulty focusing on the surface of the solutions, please call the teacher over to help.

Determine the last solution with a precipitate and the first solution without a precipitate and **BOLD** those two rows in your Google data table. While under the scope, show your well plates to the teacher for precipitation confirmation.



Disposal and Clean Up – This is very important to maintain clean well plates for next year, please be thorough.

Solutions may be poured down the sink with plenty of water. Please take the time to clean the micro well-plates thoroughly. This includes rinsing with lots of water, gently smacking the well-plate upside down to remove water, then wiping each well with a Q-tip, changing Q-tips several times.

Method 4 Processing the Data – *Successive Dilutions*

16. Calculate the K_{sp} using the concentrations of the Ca^{2+} and OH^- for only for two mixtures – the last to show a precipitate, and the first to not show a precipitate. The experimentally determined K_{sp} must be somewhere between these two K_{sp} values.

Calcium Dilution Series				
Well #	$[\text{Ca}^{2+}]$ (after diluted with water)	$[\text{Ca}^{2+}]$ (after diluted with OH^-)	$[\text{OH}^-]$ (after diluted with Ca^{2+})	Calculated K_{sp} Bold the two rows as directed in Procedure R
1	(this trial undiluted) 0.10	0.05	0.05	
2				
3				
4				
5				
6				
7				
8				
9				
10				

17. Calculate the K_{sp} using the concentrations of the Ca^{2+} and OH^- for only for two mixtures – the last to show a precipitate, and the first to not show a precipitate. The experimentally determined K_{sp} must be somewhere between these two K_{sp} values.

Hydroxide Dilution Series				
Well #	$[\text{OH}^-]$ (after diluted with water)	$[\text{Ca}^{2+}]$ (after diluted with OH^-)	$[\text{OH}^-]$ (after diluted with Ca^{2+})	Calculated K_{sp} Bold the two rows as directed in Procedure X
1	(this trial undiluted) 0.10	0.05	0.05	
2				
3				
4				
5				
6				
7				
8				
9				
10				

Post LAD Questions

1. Look up the K_{sp} for calcium hydroxide in your text. Which method in this experiment gave the most accurate value? Propose any suggestion(s) that procedure may have made that the most accurate.
2. The reaction for the dissolving of calcium hydroxide is exothermic. In *Method 1*, if the pH of the saturated solution of calcium hydroxide were measured at 35° instead of 25°C, how would you expect the pH to be larger, smaller, or remain the same *and* the resulting K_{sp} calculation to be larger, smaller, or remain the same?
3. In *Method 2*, if the beaker were wet when taking the mass of the empty beaker, how would the resulting K_{sp} be larger, smaller, or remain the same? (Follow your error analysis through each affected measurement and resulting calculation.)
4. In *Method 3*, what would be the effect on the calculated K_{sp} value if there were an air bubble in the tip of the buret when you started the titration, and this air bubble were drained during the titration? (Follow your error analysis through each affected measurement and resulting calculation.)

