Determination of the Solubility Product:

Purpose: To experimentally determine the K_{sp} of an ionic compound.

In CHM 111, we classified ionic compounds as soluble or insoluble. In reality, most insoluble ionic compounds dissolve (and ionize) a little in water and are really slightly soluble or sparingly soluble.

The reaction for the dissolution of a binary ionic compound in water is the reverse of the reaction for the precipitation. The equilibrium constant for the dissolution of a sparingly soluble ionic compound is called the solubility product (K_{sp}) .

 $MX(s) \rightleftharpoons M^{+}(aq) + X^{-}(aq) \qquad \qquad Where K_{eq} = K_{sp} = [M^{+}(aq)][X^{-}(aq)].$

The square brackets [] imply molar concentration.

Remember that the solid MX(s) is not included in the equation for the equilibrium constant.

This equation works well for ideal solutions (usually low concentrations.) K_{sp} depends on temperature with the solubility increasing as the temperature increases (think about sugar in water) for most reactions. There are some ionic compounds whose solubility decreases as the temperature goes up (exothermic reactions.)

There are often kinetic limits on how fast these reactions occur, so sufficient needs to be allotted for the reaction to come to equilibrium.

We will be looking at the formation of lead(II) iodide to determine the solubility product for PbI₂.

 $PbI_2 \rightleftharpoons Pb^{2+}(aq) + 2 I^{-}(aq)$ $K_{sp} = [Pb^{2+}(aq)][I^{-}(aq)]^2$

As with any equilibrium constant, the Ksp will vary with temperature. We will hold the temperature constant in this experiment. In order to get a precipitate, solutions with variable concentrations of Pb^{2+} and I^- will be mixed. If the product of the concentrations of the ions is greater than the solubility product, a precipitate will form.

The reaction quotient $Q = [Pb^{2+}(aq)]_i [I^{-}(aq)]_i^2$, where i refers to the concentration of the ion after mixing, can be calculated. The formation of solid crystals of Pbl₂ will occur when the value of "Q" exceeds the solubility product value of lead iodide (i.e., when Q > Ksp). Similarly, precipitation will not occur when the value of Ksp > Q. Thus, in this experimental procedure, if some sample mixtures form Pbl₂ crystals and other solutions do not, the value of the solubility product constant lies between Q values with precipitates and Q values without precipitates.

Chemicals: Lead(II) nitrate and KI.

0.010 M Pb²⁺solution is prepared by dissolving 3.312 grams of Pb(NO₃)₂ in 1.00 liter of deionized water, while a solution of 0.010 M I⁻ is prepared by dissolving 1.660 grams of KI in 1.00 liter of deionized water.

Materials needed:

Test tube rack nine clean, dry test-tubes (15 x 125 mm)3 10.0 ml pipets 1 pipettor

Reagent bottles of 0.010 M lead(II) nitrate (aq)and 0.010 M KI(aq) and a bottle of deionized water

- 1. Clean and dry 7 medium size test tubes. Label (with pencil) 1 7.
- 2. Obtain reagent bottles of 0.010 M lead(II) nitrate (aq)and 0.010 M KI(aq) and a bottle of deionized water. Prepare a pipet for each bottle. To prepare, rinse the pipet 3 times with deionized water and then twice with a small amount of the reagent solution. Place the pipet in the reagent bottle for use in the experiment.
- **3.** For runs 1 7, pipet the correct volume of each solution into the test tubes. Start by adding the water! **Stir when you are done adding all the** reactants!!!
- 4. Allow the test tubes to sit for at least 30 minutes. Stir vigorously every few minutes. Record your results after 30 minutes.
- 5. Dispose of all the chemical waste in the supplied waste bottle under the hood.

The total volume for each solution is **10.0 ml**. Note the values for Q decrease from test tube 1 to test tube 9. While you are waiting for the reactions to reach equilibrium (this will take about 30 minutes), calculate Q for reach reaction. **The calculations for test tube 1 are below.**

Test tube #	V of DI H ₂ O	V of 0.010M Pb ²⁺ (aq)	V of 0.010M I [–] (aq)	[Pb ²⁺ (aq)] _i	[I [–] (aq)] _i	$Q = [Pb^{2+}(aq)]_i([I^{-}(aq)]_i)^2$
1	1.00 ml	4.00 ml	5.00 ml	$V_1M_1=V_2M_2$ (4.00 ml)(.010M)/(10.0 mL) = M_2 M_2= 0.004M	$V_1M_1=V_2M_2$ (5.00 ml)(.010M)/(10.0 mL) = M ₂ M ₂ = 0.005M	Q =(0.004M)(0.005M) ² Q = 1.0x10 ⁻⁷

After 30 minutes look for signs of a precipitate in the solution. A golden colored cloudiness indicates that the lead(II) iodide has precipitated. Mixtures of higher Q values will contain shiny, golden crystals of lead iodide, while the tubes of lower Q values will have no solid.

CHM112 Lab – Solubility – Grading Rubric

Criteria	Points possible	Points earned							
Lab Performance									
Proper safety procedures followed and waste disposed of correctly. Work space and glassware cleaned up. Participated actively in performing the experiment.	3								
Lab Report									
Experiment performed correctly	4								
Qs correctly calculated	5								
1. K _{sp} estimated	3								
2. Percent error	3								
3. Calculation	2								
Total	20								

Subject to additional penalties at the discretion of the instructor.

Data Table for Solubility lab

Test tube #	V of DI H ₂ O	V of Pb ²⁺ (aq)	V of I⁻(aq)	[Pb ²⁺ (aq)] _i	[I [–] (aq)] _i	Q = [Pb ²⁺ (aq)] _i ([I ⁻ (aq)] _i) ²	Pbl ₂ ppt? Appearance of solution in test tube.)
1	1.00 ml	4.00 ml	5.00 ml	$V_1M_1=V_2M_2$ [Pb ²⁺ (aq)] _i = .004M	V ₁ M ₁ =V ₂ M ₂ [I ⁻ (aq)] _i = 0.005M	$Q = (.004M)(.005M)^2$ $Q = 1.0x10^{-7}$	
2	2.00 ml	3.00 ml	5.00 ml				
3	3.00 ml	2.00 ml	5.00 ml				
4	4.00 ml	1.00 ml	5.00 ml				
5	5.50 ml	2.50 ml	2.00 ml				
6	6.30 ml	1.20 ml	2.50 ml				
7	6.75 ml	1.25 ml	2.00 ml				

1. Looking at the Q values determine the approximate K_{sp} for PbI₂. $Q_{minimum}$ with PbI₂ (ppt) > K_{sp} > $Q_{maximum}$ without PbI₂(ppt).

From your data: $K_{sp} \cong$ _____

2. Look up the <u>actual value</u> for K_{sp} and calculate the percent error. How close are you to the actual solubility product? What could you have done to improve your results?

3. Using your approximate value of K_{sp} , calculate the solubility of Pbl₂ in a 0.0100 M solution of Nal(aq).