Experiment: Solubility Product of Calcium Iodate

Introduction: The equilibrium process in this experiment is a saturated aqueous solution of calcium iodate, Ca(IO$_3$)$_2$. The relevant solubility equation and solubility product expression are both shown below:

$$\text{Ca(IO}_3\text{)}_2(s) \leftrightarrow \text{Ca}^{2+}(aq) + 2\text{IO}_3^-(aq) \quad K_{sp} = [\text{Ca}^{2+}] [\text{IO}_3^-]^2$$

For a saturated solution of calcium iodate, if you can determine either the molar concentration of calcium ion, or the molar concentration iodate ion, the solubility product constant can be found. In other words, if the calcium ion concentration in today’s experiment was found to be 0.1 M, you could immediately say the concentration of iodate ion must be twice that value, or 0.2 M, according to the stoichiometry of the solubility equation given above. The solubility product constant could then be found with simple arithmetic. In this experiment, the iodate ion concentration of a saturated calcium iodate solution will be found via a redox titration with sodium thiosulfate, Na$_2$S$_2$O$_3$.

The concentration of iodate ion (IO$_3^-$) will be determined by titration with a standardized sodium thiosulfate (Na$_2$S$_2$O$_3$) solution in the presence of potassium iodide (KI). Starch will be used as an indicator, and a sharp blue-to-clear transition will mark the equivalence point. The relevant reaction equations are summarized as follows.

$$\text{IO}_3^-(aq) + 5\text{I}^-(aq) + 6\text{H}_3\text{O}^+(aq) \rightarrow 3\text{I}_2(aq) + 9\text{H}_2\text{O}(l)$$

This step, which occurs after adding both solid KI, and aqueous acid, to aliquots of saturated iodate solutions, has the net effect of converting iodate ions to aqueous iodine. Thiosulfate ion then reacts with aqueous iodine according to:

$$\text{I}_2(aq) + 2\text{S}_2\text{O}_3^{2-}(aq) \rightarrow 2\text{I}^-(aq) + \text{S}_4\text{O}_6^{2-}(aq)$$

The net titration reaction can be obtained by combining the two reactions above, then balancing for mass and charge:

$$\text{IO}_3^-(aq) + 6\text{S}_2\text{O}_3^{2-}(aq) + 6\text{H}_3\text{O}^+(aq) \rightarrow \text{I}^-(aq) + 3\text{S}_4\text{O}_6^{2-}(aq) + 9\text{H}_2\text{O}(l)$$

It is important to notice the molar ratio of iodate ion to thiosulfate ion in the above complete, balanced titration reaction. Aliquots from prepared, saturated solutions of calcium iodate in
water, with and without additional calcium ion, will be titrated according to the reaction chemistry of the equations outlined above.

**Materials:**
1. 2 - 250 ml beaker
2. 4 - 150 ml or 250 ml Erlynmeyer flasks
3. 1 - 10.0 ml pipet and pipet bulb
4. 1 - 50.0 ml Buret
5. 2 - 10 ml graduated cylinder (1 for solutions and 1 for KI solid)
6. Glass funnel
7. Stirring plate
8. Chemicals:
   - Ca(IO₃)₂, Calcium iodate
   - 0.05M Na₂S₂O₃, sodium thiosulfate, standardized.
   - 0.100 M KIO₃ potassium iodate solution (for standardization only)
   - solid potassium iodide
   - 2.0 M hydrochloric acid (Caution a strong acid)
   - 0.20 % starch solution
   - 0.1M calcium nitrate

**Procedure:** Download the spreadsheet to record all of your data (see Excel File)
1. Produce a Calcium iodate precipitate in water and in 0.1M calcium nitrate.
2. Use a standardized Sodium thiosulfate solution to determine the concentration of iodate in the saturated solution of Calcium iodate (solution 1) and in the calcium nitrate (solution 2).
3. Calculate the Ksp of Calcium iodate in each solution.

**Experimental Notes**
- It is important to handle the saturated iodate solution with great care, so as not to allow any of the solid calcium iodate into your titrations. This will obviously lead to a higher value for concentration of iodate than is actually present in the saturated solutions.
- Titrated solutions, and any left-over thiosulfate or iodate solutions, are safe to dilute and rinse down the sink.
- It is important for the Erlenmeyer flasks and other glassware you'll use to be as clean as possible when performing these redox titrations. Please take a moment to at least give them a thorough rinsing—they need not be completely dry before proceeding.

**Part 1. Saturated calcium iodate Ca(IO₃)₂ solution preparations:**
Work with another lab group to prepare Solution 1 and Solution 2. Once the solutions have stirred for 30 minutes, share the filtered solutions.

1. **Solution 1:** Obtain 0.50 gm of Ca(IO₃)₂ and transfer it into a 250 ml beaker. Add between 100 and 175 ml of water to the beaker, record your volume of water added. Place the solution on a stirring plate and mix for 30 minutes. Some of the white precipitate should remain visible.
2. **Solution 2**: Obtain 0.50 gm of Ca(IO₃)₂ and transfer it into a 250 ml beaker. Add between 100 and 175 ml of 0.1M calcium nitrate to the beaker, record the volume of calcium nitrate added. Place the solution on a stirring plate and mix for 30 minutes. Some of the white precipitate should remain visible.

3. While you are waiting, proceed to Part 2.

4. Filter the mixture using a Buchner funnel. The flask used to collect the filtrate should be as close to dry as possible. Do not pre-wet the filter paper. Filter the remaining precipitate and collect the filtrate. Do not rinse all the precipitate on to the filter paper. This will dilute your saturated solutions. Discard the solids on the filter paper and any remaining solids in the beaker. Keep these as your saturated solutions of calcium iodate. Label the flasks as solution 1 or solution 2.

**Part 2. Solubility Product of Ca(IO₃)₂**

1. Obtain a 50 ml buret and rinse it with water to ensure it is clean. Now rinse it with three 3.0 ml portions of the standardized Na₂S₂O₃ solution. Fill the buret with Na₂S₂O₃ solution.

2. Complete Part 1 and use the saturated solution of Calcium iodate (solution 1) in the following steps.

3. Pipet 10.0 mL of solution 1 into three Erlynmeyer flasks. To each flask add 25 ml of water, 0.25 g of solid KI and 1.0 ml of 2 M HCl, swirl to mix.

4. Place the flask on a piece of white paper under the filled buret. Add 2.0 ml of 0.20 % starch solution.

5. Record the initial buret reading. Add Na₂S₂O₃ in 1 ml increments until the solution becomes colorless. Record the final buret reading.

6. Refill the buret and repeat steps 3 thru 5 two more times for a total of 3 trials.

**Part 3. Common Ion Effect**

Duplicate the procedure in Part 2 above with 10.00 mL samples of the saturated solution of calcium iodate (solution 2) prepared in 0.1M calcium nitrate solution.

Remember that according to Le Chatelier’s principle, addition of either Ca²⁺ or 2IO₃⁻ should depress the solubility of calcium iodate.

**Part 4. Calculations**

1. Calculate the solubility of Ca(IO₃)₂ in water and in 0.1M Ca(NO₃)₂ solution

2. Calculate the value of Ksp for Ca(IO₃)₂ in each solution.

3. Calculate the average value of Ksp and the percent error from the value in the Handbook of Chemistry and Physics (CRC).