

**VII. Observations & Data Tables:** After the procedure, setup your data tables before you get to lab to record raw data and accompanying observations for each step of the procedure. You should include enough detail so that another person could use your notebook to perform a lab and he/she would not encounter any unexpected results. ***It is most important that data and observations be recorded directly in the notebook immediately at the time of measurement.***

**VIII. Results:** Start writing results on a new page. Return to using the full width of the notebook (only procedure and observations are written in two column format). All calculations go in this section, including calculation of percent relative average deviation, or theoretical and percent yield. Show all work for your calculations. This section should always include a boxed final table that summarizes all the pertinent results of the experiment, e.g. the values you are expected to find in the experiment.

**IX. Discussion and Conclusions:** First answer the question: “Did you accomplish the goal of the experiment?” The discussion is a succinct analysis of the meaning of your results and will often be guided by questions/statements provided by the instructor. When possible, compare results to literature values. Answer any assigned questions in this section.



• All experimental data, except instrument output, should be recorded in indelible ink.

## MAINTAINING A LAB NOTEBOOK

A record of all experiments you perform in the laboratory will be kept in a notebook that is bound and has pre-printed page numbers with either carbon copy or carbonless copy pages. This laboratory notebook is as important as the actual experiments you perform and constitutes a permanent record of your experimentation. Therefore, all entries are to be made in ink, and mistakes are to be crossed out with a single line (no white out, no erasures). Use the first couple of pages of your notebook as table of contents that is kept up-to-date. An experiment should always be started on a new odd numbered page of the notebook. **All work should be done in the notebook and not on separate sheets of loose paper.** You are not being graded on neatness. It's the process we are interested in. For example, additional questions that you are asked to address in the discussion should still be included in the notebook after the results section. Use professional language throughout the notebook; avoid first and second person pronouns like I, my, you, etc. Passive voice is usually used in scientific writing (e.g., "The chemicals were mixed in the beaker." instead of "We mixed the chemicals in the beaker.") You should sign and date all pages of an experiment. Each experiment should have the following format:

### I. Title

**II. Purpose:** A brief yet complete summary of the goals of the lab. In the context of these goals, briefly mention which basic techniques are to be used and the role that those techniques serve (for example, "end point determination by titration"). It takes practice to write a good purpose statement. You may want to leave a blank space and write the purpose after you completed sections III-VI, to ensure that you really understand why an experiment is being done.

**III. Balanced reaction(s):** where necessary.

### IV. References

**V. Table of reagents and products:** List all chemicals (name and formula) to be encountered in this experiment – all reactants, reagents, solvents, and products. Include molecular weights and relevant physical properties (e.g. mp, bp, density, solubility, concentration) for all entries. Note: An incredibly useful website for finding data for various compounds is [chemspider.com](http://chemspider.com). You are encouraged to use it!

compound name and structure	MW +	other properties

**VI. Procedure:** Start writing the procedure on a new page of the notebook. The stepwise listing of operations is to be written using prose to summarize the lab manual. In general, it is a good idea to leave some space between steps (to allow ample room for accompanying observations), to sketch pictures of an apparatus the first time it is used, and to write instructions in your own words, grouping various operations according to how you would perform them in lab.

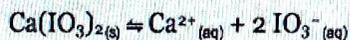
## Solubility and K<sub>sp</sub> Determination

Experimental Data	Trial 1	Trial 2	Trial 3
Molarity of sodium thiosulfate (M)	0.0351		
Volume of sodium thiosulfate			
Initial buret reading (mL)	0.03	0.02	0.00
Final buret reading (mL)	15.48	16.37	18.96
Net volume of sodium thiosulfate (mL)			

## SOLUBILITY AND $K_{sp}$ DETERMINATION

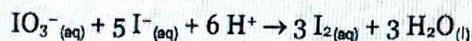
### INTRODUCTION

Calcium iodate is an ionic compound that is only slightly soluble in water. In aqueous solution, an equilibrium forms between the solid salt and its ions:

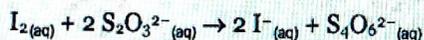


The solubility of calcium iodate can be determined by measuring the concentration of either the calcium ion or the iodate ion in a saturated solution. In this experiment the concentration of the iodate ion will be determined.

This analysis involves two reactions. First, the saturated solution of calcium iodate is acidified and reacted with excess potassium iodide, converting all the iodate ions into molecular iodine.



The molecular iodine formed is then titrated with standardized sodium thiosulfate.



The titration uses as indicators, the brown color of the molecular iodine (the iodate and iodide ions are colorless) and the dark blue color of an iodine-starch complex, (seen in the chemical kinetics experiment).

### PROCEDURE

1. Each group will need one buret and a pipet pump. In this experiment you will need a clean, dry shell vial, a clean, dry 10 mL graduated cylinder, three clean and dry filter funnels, and seven clean, 100 to 250 mL beakers (they don't need to be the same size). Four of the beakers must be dry, the other three can be wet. If your group does not have these available, clean them and put them in the oven now (remove any plastic parts from the graduated cylinders BEFORE putting them in the oven).
2. Label three beakers (the ones that can be wet) A-1, B-1, and C-1. Put about 50 mL of distilled water into each beaker. Bring a clean, dry shell vial to your instructor to obtain about 4.5 g of calcium iodate. Using the balances in the lab (NOT the analytical balances), weigh out approximately 1.0 g of calcium iodate and place it in beaker A-1. Again using the balances in the lab, weigh out approximately 1.5 g of calcium iodate and place it in beaker B-1. Weigh out approximately 2.0 g of calcium iodate and put it in beaker C-1.
3. Stir the contents of each beaker with a separate clean stir rod. Allow the solutions to sit for at least 20 minutes, stirring every few minutes. Calcium iodate is only slightly soluble and the saturated solution forms slowly. Use this time to prepare for titration (Steps 4-7). If you have put cylinders, beakers, and/or funnels into the oven, remove them now and allow them to cool.

4. Using one of the cooled, clean, and dry beakers, obtain about 100 mL of standardized sodium thiosulfate. Record the exact molarity of the sodium thiosulfate solution.
5. Using the clean, dry graduated cylinder that you prepared, measure out three separate samples of about 1 cm<sup>3</sup> (1 mL) each of solid KI. (A paper funnel might help you pour the KI into the cylinder without spills. Clean up any spilled KI!) Set these aside for use in step 14.
6. In each of three clean test tubes (they can be wet) put about 2.0 mL (40 drops) of 1% starch solution. You will need this indicator solution later in the titrations.
7. Clean the buret, rinse and fill it with the sodium thiosulfate solution.
8. Set up the three clean, dry funnels you have prepared using buret clamps. Place the three clean, dry beakers labeled A-2, B-2, and C-2 under these funnels. Put dry filter paper cones into each funnel.
9. After allowing the calcium iodate mixtures to come equilibrium (it takes at least 20 minutes) pour each solution through its own filter cone, catching each filtrate in its own dry beaker. **Do not add water.** Any precipitate remaining in the beakers can be discarded. It is the solution filtering into the beakers that you will be titrating and you do not want to change its concentration by adding rinse water. After the solution has filtered through, discard the filter papers and precipitates.
10. Set up three clean 125 mL titration flasks (they can be wet) labeled A-3, B-3, and C-3.
11. Clean the 10.0 mL volumetric pipet. Shake as much water as possible from the pipet and then rinse the pipet twice (each time with 1 to 2 mL) with the filtered solution from beaker A-2. Pipetting carefully (do not pipet by mouth; use a pipet pump or a bulb!), transfer exactly a 10.0 mL sample (aliquot) of the solution from beaker A-2 to flask A-3. Rinse the pipet twice with the filtered solution from beaker B-2 and transfer 10.0 mL of solution from beaker B-2 to flask B-3. Repeat the procedure for beaker C-2/flask C-3.
12. Add about 20 mL of water to each flask. (**Think** about why is it okay to add water now.)
13. Add about 8 drops of 6 M HCl to each flask.
14. Add about 1 cm<sup>3</sup> (1 mL) of solid KI to the flask that you are now ready to titrate. (As KI is added to the flask, it reacts with iodate to form brown I<sub>2</sub>.) Swirl each flask until the KI is dissolved.
15. Record the initial buret reading. Set flask A under the buret (a white piece of paper under the flask will help you see color changes).
16. Start adding sodium thiosulfate from the buret into the flask. Add about 1 mL at a time and swirl well after each addition. The sodium thiosulfate will react with the brown Iodine and will convert it to colorless iodide ions. When the color of the solution has faded to **pale yellow**, add one of the 2.0 mL aliquots of starch solution to the titration flask. The starch will react with the remaining iodine in the flask to produce a dark blue complex. (If the starch had been added at the beginning of the titration, the very large amount of iodine present would create numerous complex ions with the starch that would make it much more difficult to titrate.)

17. Continue to titrate slowly. The blue-black color will start to fade. The endpoint is when one drop of sodium thiosulfate causes the solution to become colorless. (Note: It is more difficult to titrate from a colored to a colorless solution than vice versa.)
18. At the endpoint, record the final buret reading and calculate the volume of sodium thiosulfate used.
19. Refill the buret and repeat steps 14 - 18 for flask B and then again for flask C.
20. Calculate the molarity of the iodate ion which was in the saturated solutions (in beakers A, B, and C).

Note: You must use the mole to mole relationships from both of the chemical reactions provided in the Introduction to go from moles of thiosulfate to moles of iodate.

21. Determine the average molarity of the iodate ion in the saturated solutions.
22. Calculate the molar solubility, the solubility in g/100 mL, and the solubility product constant,  $K_{sp}$ , for calcium iodate. Include appropriate units.

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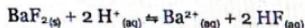
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### QUESTIONS FOR SOLUBILITY...

- How do the molarities of the iodate ion in each of the saturated solutions compare? Should they be the same? Explain.
- How would adding water in step 9 to wash the solid calcium iodate precipitate onto the filter paper change the  $K_{sp}$  value which was determined experimentally? Would the calculated value for the constant be higher, lower, or unchanged if extra water had been used in this step? Explain.
- In step 12, extra water is added to the titration flask. This added water does **not** alter the value obtained for the  $K_{sp}$ . Explain why.
- Should a precipitate form when 25.00 mL of 0.0230 M silver nitrate is added to 15.00 mL of 0.1005 M potassium acetate?
- If a 1.00 M potassium chloride solution is added dropwise (no significant volume change) to a solution containing both 0.010 M silver nitrate and 0.020 M mercury(I) nitrate, which insoluble chloride starts to precipitate first?

What percent of the cation that precipitated first remains in the solution just as the other cation reaches its saturation point with the chloride?

- What is the molar solubility of barium fluoride in a solution that contains 2.50 M acetic acid and 3.25 M sodium acetate? Hint: Combine two equilibria reactions to determine the  $K_c$  for:



and then solve for the molar solubility using the approximation method. Be sure to validate! Think about what the total concentration of fluoride ion must be, both as the free  $\text{F}^-$  and as  $\text{HF}$ .