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# Solution Stoichiometry

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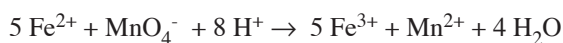
## INTRODUCTION

Stoichiometry is the calculation of the quantities of substances involved in chemical reactions. In solution stoichiometry, volumes of solutions are measured and quantities calculated using the molarities of the solutions.

Chemical analysis often uses the accurately known molarity of a solution to determine the quantity of a substance present in a sample. The solute in the solution reacts with the substance in the sample, and as the solution is added to the sample, the reaction is observed. The process of adding one reactant to another while monitoring the completeness of the reaction is called *titration*. At the *equivalence point* of the titration, the amount of solute added precisely matches the amount of substance present. To know when the equivalence point is reached, some observable phenomenon must occur. This is called the *endpoint* of the titration.

You will do two titrations in this experiment, one with an redox reaction, the other with an acid-base reaction.

The redox titration will use a solution of potassium permanganate,  $\text{KMnO}_4$ , to find the percent of iron in an iron compound. The net ionic reaction that takes place is (all ions are aqueous, water is liquid):

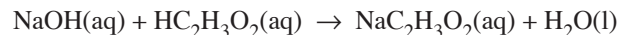


The endpoint of the reaction is clearly observable since the  $\text{MnO}_4^-$  ion is intensely purple, while the other ions in the reaction are only faintly colored. As the  $\text{MnO}_4^-$  ion solution is added to the iron compound to be analyzed, the  $\text{MnO}_4^-$  reacts with the  $\text{Fe}^{2+}$  ions in the sample, decolorizing in the process. When there is no more  $\text{Fe}^{2+}$  present, additional  $\text{MnO}_4^-$  added causes the solution to color. At the moment this begins to happen, further addition of the  $\text{MnO}_4^-$  solution is stopped. The volume added is recorded.

In analytical work, molarities are usually measured to 3 or 4 significant digits. A solution with an accurately measured molarity is called a *standardized* solution.

Given the molarity of the  $\text{MnO}_4^-$  solution and the mole relationships in the chemical reaction, the number of moles of Fe and the mass of the Fe can be determined. The mass of Fe divided by the mass of the sample, times 100, gives the % of Fe in the sample.

The acid-base titration will use a standardized solution of NaOH to find the percentage of acetic acid in a sample of vinegar. The reaction is:



After enough NaOH is added to react with all of the acetic acid present in the vinegar, addition of more NaOH will cause the solution to turn basic.

To be able to observe when this has happened, a small amount of the dye phenolphthalein is added at the beginning of the titration. Phenolphthalein is colorless in acid solutions, vivid pink in basic solutions. It is one of many dyes (called acid-base indicators) that change color depending on the acid-base level of the solution.

As soon as the color of the vinegar solution begins to turn pink, addition of NaOH is stopped. Knowing the volume and the strength of the NaOH solution will enable you to find the moles, and hence the mass, of the acetic acid present.

In this experiment, you will use pre-standardized solutions and simple techniques to get an introduction to solution stoichiometry. Titrations in Chem 101B are more sophisticated. Burets are used to measure volumes, and you will learn how to standardize solutions.

## EQUIPMENT

### Special supplies:

- one stir bar.

### From the wall cabinet:

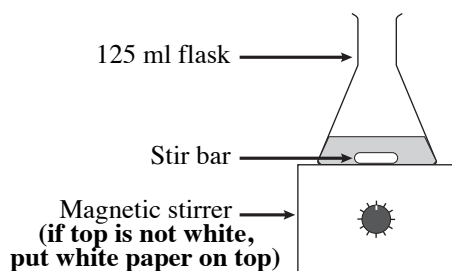
- one magnetic stirrer.

### From your locker:

- one 125 ml flask.
- one 10 ml graduated cylinder.
- one transfer pipet.
- two 50 ml plastic beakers.
- one 100 ml beaker for solution disposal

## EXPERIMENT

For parts a and b, you will add standardized solutions to samples contained in a flask set on a magnetic stirrer:



The stirrer should be set to give a slight vortex in the solution. Do not set the spin speed too high, or splashing will stick droplets of the solution onto the sides of the flask. If this happens, use the deionized water bottle to rinse the droplets back down into the body of the solution.

### Procedures to be used in parts a) and b):

In each part, you will measure the volume of standardized solution used by adding the solution from a graduated cylinder using a transfer pipet. To avoid contaminating standardized solutions, the following procedures are used.

Potassium permanganate solutions must not be disposed in the sink. Sodium hydroxide solutions can go in the sink.

- 1) Bring 20 ml of the reagent to your work place as directed in the first sentence of part a) and part b).
- 2) Rinse the transfer pipet with deionized water.
- 3) Draw up a small amount of standardized solution into the transfer pipet, rotate to rinse all of the inner surface, then discard the solution into the disposal beaker for part a), into the sink for part b). Repeat.
- 4) Rinse the graduated cylinder with deionized water and allow it to drain.
- 5) Add 2 ml of the standardized solution, and while rotating the cylinder, allow the solution to rinse the walls of the cylinder as you pour the solution out into the disposal beaker (for part a). Repeat.
- 6) Fill the cylinder to above the 10 ml mark with the standardized solution. Use the transfer pipet to get the volume right at the 10.0 ml mark, discarding excess into the disposal beaker (part a). Record 10.0 ml as the initial volume on the data sheet.

### Part a) Iron sample analysis:

Bring about 20 ml of the standardized  $\text{KMnO}_4$  solution to your lab space in a 50 ml plastic beaker. This is enough solution for rinsing and reacting. Prepare the graduated cylinder and transfer pipet as noted. Record the molarity of the solution in the data table. Fold a piece of weighing paper in half, and then open it up. This puts a crease in the paper that will make pouring the solid out of the paper easier. Place the paper on the milligram balance, cover with the draft shield, and zero the balance. Weigh a 0.16 to 0.18 g sample of the iron compound onto this piece of zeroed-out weighing paper. Record the mass to the nearest 0.001 g. Transfer into a clean (not necessarily dry) 125 ml flask. Add 50 ml of deionized water, using the mark on the flask to monitor the volume. If any of the iron sample is stuck on the side of the flask, tip the flask to get it into the liquid. Add 3 ml of 1 M  $\text{H}_2\text{SO}_4$  solution to provide the  $\text{H}^+$  noted in the balanced equation on the page 27. Turn the stirrer on. When the crystals are dissolved, begin adding the permanganate solution drop by drop. Note how the color disappears. As you near the end point, the rate of color disappearance slows. Try to stop the addition of the permanganate solution when there are just 1 or 2 extra drops, as evidenced by the permanent faint purple caused by the excess unreacted  $\text{MnO}_4^-$  ions. Squirt any unused solution from the transfer pipet back into the graduated cylinder, read the volume of liquid in the cylinder, and record to the nearest 0.1 ml as the final volume in the data table. Pour any remaining permanganate solution into the beaker marked "Permanganate Waste" located in the fume hood. Pour the contents of the flask into the sink. Rinse out the flask, cylinder, and pipet with deionized water. Do not dump the stir bar down the drain.

### Part b) Vinegar analysis

Bring about 20 ml of the standardized  $\text{NaOH}$  solution to your lab space in a 50 ml plastic beaker. Prepare the graduated cylinder and transfer pipet as noted, and fill the graduated cylinder to the 10.0 mark. Record the molarity of the solution in the data table. Weigh about 0.8 grams of vinegar into the 125 ml flask. (Zero out the flask weight, then add vinegar until about 0.8 gram is on the display.) Record the mass of the vinegar to the nearest 0.001 g. Add 50 ml of deionized water, using the mark on the flask to monitor the volume. Add 2 drops of phenolphthalein solution. Turn the stirrer on. Add the  $\text{NaOH}$  solution drop by drop. Try to stop the addition of the hydroxide solution when there are just 1 or 2 extra drops, as evidenced by the permanent faint pink caused by the excess unreacted  $\text{OH}^-$  ions interacting with the phenolphthalein. Squirt any unused solution from the transfer pipet back into the graduated cylinder, read the volume of liquid in the cylinder, and record to the nearest 0.1 ml as the final volume in the data table. Dispose of these solutions in the sink. Clean up the equipment, return the stir bar to the storeroom, the magnetic stirrer to the cabinet (the electrical cord should be wound around the stirrer), and your materials to the locker. Clean up the work space, then proceed with the calculations.

Use Significant Digits Properly! Show calculation setups in the spaces in the left-hand columns for each part.

DATA

PART A		PART B	
1. mass of iron compound	g	1. mass of vinegar	g
2. molarity of $\text{KMnO}_4$	M	2. molarity of NaOH	M
3. initial volume	ml	3. initial volume	ml
4. final volume	ml	4. final volume	ml

CALCULATIONS

5. volume of $\text{MnO}_4^-$ solution used (3 - 4)	ml	5. volume of NaOH solution used (3 - 4)	ml
6. mol of $\text{MnO}_4^-$ used ( $M \times \text{vol}$ in liters)	mol	6. mol of NaOH used ( $M \times \text{vol}$ in liters)	mol
7. reacting ratio mol $\text{Fe}^{2+}$ to mol $\text{MnO}_4^-$		7. reacting ratio, mol $\text{HC}_2\text{H}_3\text{O}_2$ to mol NaOH	
8. mol of $\text{Fe}^{2+}$ in compound sample (from 6 and 7)	mol	8. mol of $\text{HC}_2\text{H}_3\text{O}_2$ in vinegar sample (from 6 and 7)	mol
9. g of $\text{Fe}^{2+}$ in compound sample	g	9. g of $\text{HC}_2\text{H}_3\text{O}_2$ in vinegar sample	g
10. % of $\text{Fe}^{2+}$ in compound sample	%	10. % of $\text{HC}_2\text{H}_3\text{O}_2$ in vinegar sample	%

**QUESTIONS**

1. The iron compound is  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ . What is its molecular mass? (The raised dot shows that 7 waters are included in the molecular mass.) What is the % iron in this compound from the formula?
2. Use the relationship:  $\% \text{ deviation} = \frac{\% \text{ iron}_{\text{exp}} - \% \text{ iron}_{\text{formula}}}{\% \text{ iron}_{\text{formula}}} \times 100$  to calculate the % deviation of your result. Notice how many significant digits there are in the numerator after the subtraction. This will limit the digits in the %.
3. Look on the vinegar bottle label to find the % acetic acid listed. Use a similar relationship as in question 2 to find the % deviation of your result from the label.
4. The 50 ml of water added to the flask serves as the medium in which the reaction takes place. If 75 ml were added instead of 50, your results would be the same. Explain why varying this volume does not change the result.
5. What would you do to improve the accuracy of the analysis you performed today? To answer, look at the calculations. Which numbers used in the calculations have the fewest number of significant digits? What would you do to get more significant digits for these numbers? (It is a constant assumption that if you were more careful, your results would be better, so please don't use the "more careful" cliché in your answer.)