Stoichiometry of a Redox Reaction ☮ AP Chemistry

Introduction:

In general, when different chemicals are brought together, one can usually detect whether a chemical reaction has occurred or not. In redox reactions the identity of the products are less obvious. It is important for chemists to determine the products, by determining the electrons that were transferred in the reaction. One way to follow the electrons is to perform a redox titration.

In this lab we will be observing the reaction of Fe^{3+} with the compound hydroxylamine, NH_2OH. In this reaction, the Fe^{3+} is reduced to Fe^{2+}. Since the iron is being reduced, the NH_2OH must be supplying the electrons - by being oxidized.

The purpose of this experiment is to make a reasonable assignment as to what N containing compound could be formed upon oxidation of NH_2OH. In other words, your goal is to determine the question mark in the following equation.

$$NH_2OH + Fe^{3+} \text{ --------> } \underline{\mathbf{?}} + Fe^{2+}$$

The reaction will be carried out by heating a precisely known amount of hydroxylamine *in an acidic medium* with a substantial excess of iron(III)sulfate. The Fe^{2+} produced by the reaction will be determined by potassium permanganate titration. The following *unbalanced* equation will reveal more information:

$$MnO_4^- + Fe^{2+} \text{ -------> } Mn^{2+} + Fe^{3+}$$

By knowing the molarity and volume of MnO_4^- needed to complete the titration, one can figure out the moles of Fe^{2+} produced, moles of electrons transferred in the original reaction and change in oxidation state of the N in hydroxylamine which will, in the end, allow you to predict the oxidized product.

Procedure:

1.Gather materials:
- -1 hot plate
- -2 small flasks
- -1 50mL buret

2.Pipet *exactly* 10mL of hydroxylamine, NH_2OH, solution in both flasks (you are going to do two trials). Record the molarity and volume of the hydroxylamine solution used on your data table.

3. Using a graduated cyclinder, transfer about 25mL of the iron(III)sulfate solution to each flask. The molarity of the iron(III) solution is about 0.25 M.

4. Add about 20 mL of distilled water to each of the flasks.

5. On a hot plate, boil the solutions *gently* for 5 minutes. This should be sufficient to complete the oxidation of the hydroxylamine.

6. Add about 50 mL of distilled water and about 5 mL of 6M phosphoric acid (used to de-color the solution).

7. Titrate the two solutions with potassium permanganate.
   a. Clean your buret by rinsing once (or twice if needed) with about 5 mL of dH_2O and then, once with about 5 mL of the $KMnO_4$ solution. Any excess $KMnO_4$ can be poured down the drain with the faucet running.

b. Record, on your data table, the molarity of the $KMnO_4$ solution.
c. Fill your buret slightly passed the zero line and drain some $KMnO_4$ through the tip.
d. Record an initial buret reading on your data table.
e. Titrate your two solutions. Remember, you are looking for the exact point where one drop of $KMnO_4$ soln turns the whole solution to light pink. This color change will be slight so be sure that you have a piece of white paper under the flask to make the change a little more visible. Make sure to *Swirl, Swirl, Swirl!!!*

8. Clean up your area
- Pour any excess $KMnO_4$ in the appropriate waste container.

***-Clean your buret by:
a. rinsing with tap H_2O b. then with dilute HCl c. then with tap water d. then with dH_2O
-Discard of titrated solution as instructed by Mr. Collins.
- Rinse and put away any glassware used.

Data Table:
Construct your own data table. Make sure to take into account that you are probably going to do two trials.

Calculations:
1. Determine the volume of $KMnO_4$ solution used during the titrations.
2. Determine the moles of $KMnO_4$ solution used.
3. Relative to the moles of $KMnO_4$ used in the titration, calculate the number of moles of Fe^{2+} in solution.
4. What is the half reaction for the original reduction of Fe^{3+} to Fe^{2+} Since you know how many moles of Fe^{2+} were produced, according to the half reaction, how many moles of electrons were gained? Assume that the number of electrons gained in the iron reduction must equal the moles of electrons lost during the oxidation of NH_2OH.
5. How many moles of NH_2OH were used in the original reaction?
6. How many electrons were lost per mole of NH_2OH used?
7. Make a prediction about the identity of the unknown "N" chemical produced. Some possible outcomes:

$$N_2,\ N_2O,\ NO,\ HNO_2,\ NO_3^-,\ H_2N_2O_3$$

BE SURE TO EXPLAIN YOUR PREDICTION IN YOUR *BRIEF* CONCLUSION. . .

Post Lab-Questions:
1. Why did you use a very accurate pipet for measuring out the NH_2OH, and only a graduated cylinder for measuring the iron(III)solution?
2. If hydroxylamine is partly lost due to evaporation due to boiling (before it is oxidized), what affect does this have on the interpretation? How can this possible loss be minimized?
3. What if some iron(III) is reduced upon heating the solutions, even when no hydroxylamine is present? What affect will this have on the interpretation?
4. What if the 5 minute heating period is not long enough to complete the oxidation? What affect will this have on the interpretation?