Experiment 21C

ANALYSIS OF VODKA

MATERIALS:

pH-voltmeter, electrodes, $K_2Cr_2O_7$, $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$, 25 mL, 10 mL and 5 mL pipets, vodka, 100 mL and 250 mL volumetric flasks, 400 mL beaker, magnetic stirrer, 70% H_2SO_4, 10 mL graduated cylinder, buret.

DISCUSSION:

The determination of ethanol is usually made on samples such as wines, beer, blood and urine that may contain free alcohol. In nearly all of these however, it is necessary to separate the ethanol by distillation before analysis. We will determine the amount of alcohol in a commercial vodka, since vodka is relatively free of substances that would interfere with the ethanol analysis.

Most chemical methods of analysis involve an oxidation of the alcohol with a strong oxidizing agent and measurement of the amount of oxidant consumed in this reaction. The products of the oxidation of ethanol with most oxidants depend greatly on the experimental conditions used and it is necessary, therefore, to adhere closely to those conditions where the stoichiometry of the reaction is known. Using acid dichromate, small amounts of ethanol can be oxidized to acetic acid at room temperature, according to the (unbalanced) equation:

$$___\ Cr_2O_7^{-2} + ___\ H^+ + ___\ C_2H_5OH \rightarrow ___\ Cr^{+3} + ___\ H_2O + ___\ CH_3COOH$$

In order to measure the amount of dichromate used by the alcohol, an excess of dichromate will be added and then the unreacted dichromate will be back titrated with standard iron(II) solution, according to the (unbalanced) equation:

$$___\ Fe^{+2} + ___\ Cr_2O_7^{-2} + ___\ H^+ \rightarrow ___Fe^{+3} + ___\ Cr^{+3} + ___\ H_2O$$

PROCEDURE: (work in pairs)

Prepare 100 mL of a standard solution of potassium dichromate by dissolving 4 g of dried material, weighed on an analytical balance, in distilled water in a 100 mL volumetric flask. Next prepare a solution of ferrous ammonium sulfate hexahydrate by dissolving 11-12 g of material in 250 mL of water. This solution should also contain 7 mL of 70% sulfuric acid solution. (Since the Fe^{2+} solution must be standardized, there is no need to weigh the solid with any great degree of accuracy, nor is it important to use a volumetric flask.)

Part A:

First, using a clean pipet, pipet 10 mL of the vodka into a *250 mL* volumetric flask and dilute to the mark. Set this solution aside. Next, pipet 25 mL of the standard $K_2Cr_2O_7$ solution into a *100 mL* volumetric flask. Add 10 mL of 70% sulfuric acid solution and cool the resulting solution to room temperature with tap water. Now pipet a 10 mL aliquot of the accurately diluted vodka into the flask and shake. Allow this solution to stand at room temperature for at least 30 minutes. In the meantime, proceed with Part B.

Part B:

Pipet 5 mL of your standard dichromate solution into a 400 mL beaker which contains 100 mL of distilled water. Add 10 mL of 70% sulfuric acid solution and start stirring. With the set up shown in the diagram, begin titrating the dichromate solution with the iron(II) solution. Be careful to record sufficient readings near the equivalence point so you can construct a well defined curve. Note: What portion of the overall titration curve are you interested in? Plot this portion and submit it with your report.

Repeat the above standardization at least once. Your values should agree to within 0.10 mL.

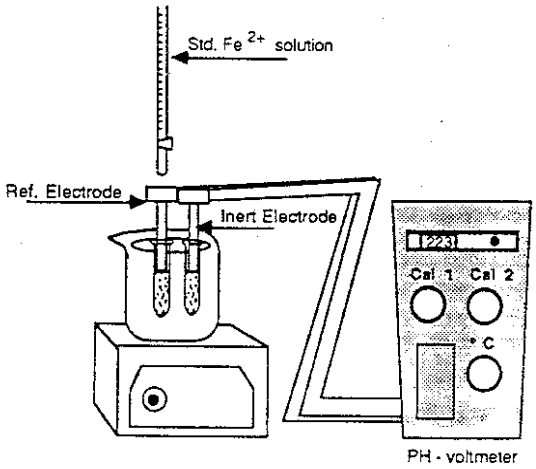

Figure 1

Part A (continued):

Having waited at least 30 minutes, dilute the alcohol dichromate solution up to 100 mL and shake. Now remove a 25 mL aliquot with a pipet and place it in the 400 mL beaker along with 75 mL of water and 7.5 mL of 70% sulfuric acid solution. Titrate the resulting solution with your standardized iron(II) solution, again taking care near the equivalence point. A duplicate titration should also agree within 0.10 mL.

CALCULATIONS:

Once you have balanced the appropriate equations and experimentally determined the equivalence points of the titrations, it is possible to determine the weight of ethanol present in the original sample. However, since ethanol solutions are frequently reported in percent by volume, you should report your results in this manner.

$$\%\text{ by volume ethanol} = \frac{\left(\dfrac{\text{g. ethanol found}}{\text{density of ethanol}}\right)}{\text{mL of original sample}} \times 100$$

The value for the density of ethanol can be found in the CRC Handbook of Chemistry and Physics.

Note: Hand in the titration curves with your report. Data manipulation, graphing, and regression analysis can easily be accomplished using a computer spreadsheet such as QUATTRO.

Name _____ Section _____

Partner _____ Date _____

DATA SECTION
Experiment 21C

mass $K_2Cr_2O_7$ used _____

vol. Fe^{+2} solution in part B _____

_____

(average) _____

vol. Fe^{+2} solution used for back titration in part A _____

_____

(average) _____

% by volume ethanol _____

Show your calculations for percent by volume ethanol.