Lesson 2 Volumetric analysis - titrations involving redox reactions, %m/m and errors.

 A wine bottle was labelled 3.0 %m/m of ethanol. Volumetric analysis was used to determine and verify the alcohol content of the wine. A 110.0 gram sample of the wine was placed into a 250 mL volumetric flask and made up to the mark with deionised water. A 20.00 mL aliquot was placed into a 100 mL conical flask and two drops of the appropriate indicator added before titrating with a 0.450 M K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution. Four titres were obtained 10.10 mL, 9.88 mL, 10.00 mL and 10.05 mL.

The <u>unbalanced</u> overall reaction is shown below.

 $Cr_2O_7^{-2}$  (aq) +  $C_2H_5OH(aq)$  +  $H^+(aq) \rightarrow Cr^{3+}(aq)$  +  $CH_3COOH(aq)$  +  $H_2O(I)$ 

a) Write a balanced equation for the:

- i. oxidation half reaction  $C_2H_5OH(aq) + H_2O(l) \rightarrow CH_3COOH(aq) + 4H^+ + 4e$
- ii. reduction half reaction  $Cr_2O_7^{-2}(aq) + 14H^*(aq) + 6e \rightarrow 2Cr^{3*}(aq) + 7H_2O(I)$

iii. overall reaction

- $2Cr_2O_7^{-2}(aq) + 3C_2H_5OH(aq) + 16H^{+}(aq) \rightarrow 4Cr^{3+}(aq) + 3CH_3COOH(aq) + 11H_2O(I)$
- b) Calculate the average titre (10.10 + 10.00 + 10.05) / 3 = 10.05
- c) Calculate the amount in mol of  $Cr_2O_7^{-2}$  in the average titre.

 $n_{dichromate} = C X V = 0.450 X 0.01005 = 0.00452$ 

d) Calculate the amount of ethanol in the conical flask.

According to the stoichiometric ratio, derived from the balanced overall equation for every 2 mol of  $Cr_2O_7^-$  used 3 mol of ethanol reacts.

- *=> 0.00452 X 3/2 = 0.00678*
- e) Calculate the amount of ethanol, in mol, in the volumetric flask
- => 0.00678 X 250/20.0 = 0.0848
- f) Calculate the concentration of ethanol in %m/m.
- Step 1 find the mass of ethanol in the volumetric flask

=> 0.00848 x Fm = 0.0848 X 46.1 = 3.91 g

- => Calculate the concentration in %m/m = (3.91/110.0) X 100 = 3.55%
- g) Explain how the result in f) above will differ if :
  - the burette was washed with water increase
    - the pipette was washed with water *decrease*
    - the conical flask was washed with the potassium dichromate solution.- decrease
    - the volumetric flask was washed with water no change

- 2) An outline of a titration method, to determining the concentration of acetic acid in a particular brand of vinegar, is given below.
  - 1. A burette is filled with a standard solution of sodium carbonate.
  - 2. The vinegar is diluted in a volumetric flask.

3. A pipette is used to transfer 20.00 mL of diluted vinegar to a conical flask and a few drops of phenolphthalein indicator is added.

4. The diluted vinegar is titrated with the base. Titrations are repeated until three concordant results are obtained.

The equation for the reaction is

 $Na_{2}CO_{3}(aq) + 2CH_{3}COOH(aq) \rightarrow 2CH_{3}COONa(aq) + H_{2}O(I) + CO_{2}(g)$ 

- a) The glassware was rinsed before the titration. Indicate which solution should be used to finally rinse each of these pieces of glassware
  - i. volumetric flask *distilled water*
  - ii. pipette *diluted vinegar solution*
  - iii. burette *Na<sub>2</sub>CO<sub>3</sub> solution*
  - iv. conical flask *distilled water*
- b) Explain why the vinegar must be diluted

So as not to use too large a volume of the  $Na_2CO_3$  solution or

To use and obtain a reasonably sized titre.

c) One set of results are given below. The data shown in the student's laboratory book was

-concentration of  $Na_2CO_3(aq) = 0.108 M$ 

- volume of undiluted vinegar = 12.00 mL
- total volume of diluted vinegar = 200.00 mL
- volume of diluted vinegar used in each titration = 20.00 mL

- average titre of Na<sub>2</sub>CO<sub>3</sub> = 16.36 mL

Based on these results, calculate the concentration, in mol  $L^{-1}$ , of acetic acid in the undiluted vinegar solution. Give the answer to the correct number of significant figures.

Step 1 Calculate the mol of Na<sub>2</sub>CO<sub>3</sub> in the average titre.

 $\Rightarrow n = C X V = 0.108 X 0.01636 = 0.001767$ 

Step 2 Calculate the mol of vinegar in the 20.00 mL aliquot of the diluted vinegar

=> According to the stoichiometry the ratio of  $Na_2CO_3$  to ethanoic acid is 1:2

 $Na_2CO_3(aq) + 2CH_3COOH(aq) \rightarrow 2CH_3COONa(aq) + H_2O(l) + CO_2(g)$ =>  $n_{ethanoic aicd} = 2 \times 0.001767 = 0.003534$ 

Step 3 calculate the mol of ethanoic acid that was in the volumetric flask

 $=> n_{ethanoic acid in the volumetric flask} = 0.003534 X 200/20 = 0.03534$ 

Step 4 calculate the concentration

=> C = n/V = 0.03534 / 0.01200 = 2.95 M (3 sig fig)