Titration Questions from 1998 – 2001:

Titrations are designed to work out the concentration of one substance by reacting it with a known substance, usually one made by the chemist from what is known as a primary standard.

A primary standard requires the following qualities:

- 1. Substance must have high purity
- 2. High molar Mass to decrease percentage error.
- 3. Must not change when exposed to air so not deliquescent or absorbs water
- 4. Must be soluble and have a know molecular formula

Titrations are usually done backwards.

Tips:

- 1. Do the basic steps for calculations 1] Find moles. 2] Find or balance the reaction 3] do the stoichiometry and finally 4] Calculate the missing concentration.
- Keep track of the volumes of the substances you are calculating the moles and concentrations
 off. Due to dilutions and titre samples being taken from the substances there are often 2-4
 different volumes of some of the substances reacting and a common error is to miss one of
 these steps.
- 3. If confused even slightly quickly draw a basic diagram of what is happening and in what order.
- 4. Keep your work clear and well labelled. This will improve the amount of follow through marks you get if you make a mistake and reduce your chance of error.

Now try the following questions:

1. [2008 Exam - 10 marks. Easy straight forward question. No tricks.] A bottle of anhydrous oxalic acid ($H_2C_2O_4$) was found to be contaminated with potassium chloride. 2.05 g of the mixture was dissolved in distilled water and the volume made up to 250.0 mL in a volumetric flask. 20.0 mL aliquots of the solution were titrated against the 0.115 mol L-1 sodium hydroxide solution and the following results were obtained:

Titration Results	Trials (mL)			
	1	2	3	4
Final Volume	32.05	32.10	31.11	33.25
Initial Volume	0.50	2.45	1.40	3.65
Titre				

- a) Write an equation for the reaction between oxalic acid and sodium hydroxide.
- b) Complete the table.
- c) Calculate the average titre.
- d) Calculate the concentration of the oxalic acid solution.
- e) Calculate the percentage purity of the oxalic acid mixture.

2. [1998 – 9 marks. Standard.] A copper sulfide ore was analysed by iodometry in the following way. The ore was finely crushed and a representative sample of 6.056g was brought into solution with a mixture of hydrochloric acid and concentrated nitric acid. The excess hydrochloric and nitric acids were removed by heating with a little concentrated sulphuric acid. The remaining solution was quantitatively measured and transferred to a 250.0mL volumetric flask and the solution made up to the mark with water.

25.00mL portions of the above solution were each treated as follows. Sodium acetate was added to bring the pH to 4. Then an excess of potassium iodide solution was added, and iodine was liberated according to the following equation.

$$2Cu^{2+} + 4I^{-} \rightarrow 2CUI_{(s)} + I_{2}$$

Each portion was titrated with $0.05103 \text{ molL}^{-1}$ sodium thiosulphate ($Na_2S_2O_3$) using starch indicator (which was added towards the end of the titration). The following figures were obtained.

Final reading (mL)	17	16.5	15.68	15.12	16.28	
Initial reading (mL)	0.22	1.10	0.66	0.06	1.24	

The equation for the reaction of iodine with sodium thiosulphate is as follows:

$$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^-$$

Starch is blue in the presence of I₂. The end-point is taken to be the loss of all blue colour.

a.

- i. How many moles of iodine are liberated from one mole of copper (II) ion?
- ii. How many moles of thiosulphate ion are required for one mole of iodine?
- iii. How many moles of thiosulphate ion are required for one mole of copper (II) ion?
- b. What titration volume should you use in the calculation?

c. Calculate the percentage by mass of copper in the ore.

3. [1998 - 9 marks]The Kjeldahl method is used to analyse for nitrogen in an organic substance. The substance is treated with concentrated H_2SO_4 , using anhydrous $CuSO_4$ as a catalyst; all nitrogen is converted into $NH4^+$ ion.

The mixture is then treated with excess OH^- to convert the NH_4^+ ion into NH_3 . The NH_3 is boiled off and absorbed in an excess of dilute HCl.

In a Kjeldahl determination 1.2540g of dried pet food was heated for an hour with concentrated H_2SO_4 and anhydrous $CuSO_4$, (together with K_2SO_4 to raise the boiling-point of the reaction mixture).

On cooling the reaction mixture, Zn pieces and an excess of concentrated NaOH solution were added and the flask quickly attached to a distillation apparatus as shown below. The mixture was gently boiled to drive the NH_3 into the receiving flask. (The Zn dissolves to give $Zn(OH)_4^{2-}$ and H_2 gas; the H_2 gas helps sweep out all the NH_3).

The NH₃ was distilled into 50.00mL of 0.01970 molL⁻¹ HCl.

a. Calculate the original number of moles of H⁺ in the solution in the receiving flask before any NH₃ was absorbed.

b. After the NH_3 had been absorbed by the HCl solution, the excess HCl was titrated with 0.1000 molL⁻¹ NaOH (in the burette). Methyl orange was used as an indicator; 5.62 mL of NaOH solution was needed for the colour change. Calculate the number of moles of H⁺ in this solution after the absorption of NH_3 .

C.	Calculate the number of moles of NH_3 absorbed by the HCl solution, and hence the percentage by mass of nitrogen in the 1.2540g of dried pet food.
d.	During the decomposition of the pet food (with H_2SO_4 , $CuSO_4$ and K_2SO_4) there is no stopper on the flask. Why is the nitrogen not lost?

- 4. [1999 11 marks] Sodium sulfite (Na₂SO₃) can be used as a preservative in hamburgers. In an experiment to determine the amount of Na₂SO₃ in a sample of hamburger, the following steps were followed.
 - Step 1: 1.00kg of minced meat was boiled with an excess of dilute sulphuric acid.

$$Na_2SO_3 + 2H^+ \rightarrow SO_2 + H_2O + 2Na^+$$

Step 2: The sulphur dioxide gas released was completely absorbed in an excess of sodium hydroxide solution.

$$SO_2 + 2OH^- \rightarrow SO_3^{2-} + H_2O$$

Step 3: The resulting solution was acidified with excess dilute sulphuric acid and titrated with 0.02023 molL⁻¹ potassium permanganate. 30.08 mL of the potassium permanganate solution was required to reach the end-point.

$$5SO_3^{2-} + 2MnO_4^- + 6H^+ \rightarrow 5SO_4^{2-} + 2Mn^{2+} + 3H_2O$$

- a. How many moles of SO_3^{2-} are required to react completely with 1 mole of $MnO_4^{-?}$?
- b. Calculate the number of moles of Na₂SO₃ present in 1.00 kg of hamburger meat.

c.	Government chemists often express the amount of Na_2SO_3 in meats as parts per million (ppm) [1 ppm = 1 mg Na_2SO_3 in 1 kg of meat]. Express the amount of Na_2SO_3 in the 1.00 kg meat sample in parts per million.
d.	In step 1, why must the dilute sulphuric acid be in excess?
e.	In step 3, what colour change in the reaction vessel would you observe at the end point?

5. In a method for volumetric determination of ethanol in wine, the ethanol (CH₃CH₂OH) is first distilled from a basic solution of the wine.

The distilled CH_3CH_2OH solution is then treated with an excess of a solution containing dilute H_2SO_4 and $K_2Cr_2O_7$. The mixture is then placed in a water bath at $60^{\circ}C$ for 30 minutes to ensure complete oxidation to CH_3COOH .

A redox indicator (N-phenylanthranilic acid) is then added and the amount of excess $K_2Cr_2O_7$ is determined by titration with a standard solution containing Fe2+ ion. The equation for this reaction is:

$$Cr_2O_7^{2-} + 6Fe^{2+} + 14H^+ \rightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O_7^{2-}$$

From this the amount of $Cr_2O_7^{2-}$ ion that combined with the CH_3CH_2OH and hence the amount of CH_3CH_2OH in the sample, can be calculated.

The following results were obtained for a 1.00mL sample of wine analysed as above.

Volume of 0.1005 molL ⁻¹ K ₂ Cr ₂ O ₇ added to the distillate:	25.00 mL
Volume of 0.3005 molL ⁻¹ Fe ²⁺ required to titrate the excess K ₂ Cr ₂ O ₇	23.55 mL

The half equations for the oxidation of ethanol and the reduction of Cr₂O₇²-are:

$$CH_3CH_2OH + H_2O \rightarrow CH_3COOH + 4H^+ + 4e^-$$

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$$

a. Combine these two half equations to obtain an equation for the oxidation of CH_3CH_2OH to CH_3COOH by a solution containing dilute H_2SO_4 and $K_2Cr_2O_7$.

b. Calculate the amount (in mole) of K₂Cr₂O₇ added to the CH₃CH₂OH.

c.	Calculate the amount (in mole) of K ₂ Cr ₂ O ₇ in excess.
d.	Calculate the amount (in mole) of $K_2Cr_2O_7$ that combined with the CH_3CH_2OH .
e.	Calculate the concentration of CH_3CH_2OH in gram per 100 mL in the wine.

6. Ferrovanadium is an alloy of Fe and V.

When 0.8765g of the alloy was placed in hot, moderately concentrated sulphuric acid the alloy dissolved:

the iron was converted into iron (III) ion and the vanadium was converted into vanadate ion (VO₃⁻) $2V + 8H^+ + 5SO_4^{2-} \rightarrow 2VO_3^- + 5SO_2 + 4H_2O$

The solution was cooled and diluted to 250.0 mL with water. A 50.00 mL sample was made basic with NaOH solution to precipitate with Fe³⁺ as Fe(OH)₃. This was filtered out, and the vanadium containing solution re-acidified with sulphuric acid.

The acid solution was passed through a column of Zn wire reducing the VO_3^- to V^{2+}

$$2VO_3^- + 12H^+ + 3Zn \rightarrow 2V^{2+} + 3Zn^{2+} + 6H_2O$$

The solution was then treated with excess iron (III) sulphate solution

$$V^{2+} + 2Fe^{3+} + 2H_2O \rightarrow VO^{2+} + 2H^+ + 2Fe^{2+}$$

And the resulting iron (II) ion titrated with 0.03004 mol L-1 KMnO₄

$$5Fe^{2+} + MnO_4^- + 8H^+ \rightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$$

And 24.60mL of the KMnO₄ was required.

a. Use the above equations to work out how many moles of permanganate ion will be required to react with the iron (II) ion produced from 1 mole of vanadium originally.

b. Calculate the percentage by mass of vanadium metal in the alloy.

- 1) Solutions
 - a) $H_2C_2O_{4(aq)} + 2OH_{(aq)}^- \rightarrow C_2O_{4(aq)}^{2-} + 2H_2O_{(l)}$
 - b) 31.55, 29.65, 29.71, 29.60
 - c) 29.65 mL
 - d) $n(OH^-)$ =cv=0.115(29.65/1000) = 0.00341 mol $n(H_2C_2O_4) = \frac{1}{2}0.00341 = 0.00171 \, mol$ $[H_2C_2O_4] = \frac{n}{v} = \frac{0.00171}{0.02} = 0.0853 \, mol \, L^{-1}$
 - e) $n(H_2C_2O_4)_{250} = 0.0171\left(\frac{250}{20}\right) = 0.0213 \ mol$ $m(H_2C_2O_4) = nM = 0.0213(90.036) = 1.92 \ g$ % $purity = \frac{1.92}{2.05}100 = 93.6 \ \%$
- 2) Translation: 6.056g was put into a 250 mL flask as copper ions. 25 mL were reacted with 0.05103 mol L^{-1} S₂O₃ with the amount given in the table.
 - a) I hope it is clear these are redox reactions, balance these in the standard manner
 - i) 0.5 moles
 - ii) 2 moles
 - iii) 1 mole
 - b) 15.04 mL
 - c) $n(S_2O_3)_{15.04}^{2-} = 0.001504 \times 0.051103 = 0.0007675 \, mol$ $n(Cu^{2+})_{25} = \frac{1}{1}0.0007675 = 0.0007675 \, mol$ $n(Cu^{2+})_{250} = \frac{250}{25}0.0007675 = 0.007675 mol$ $m(Cu^{2+}) = nM = 0.007675 \times 63.54 = 0.4877 \, g$ % $purity = \frac{0.4877}{6.056}100 = 8.053 \, \%$
- 3) .
 - a) $n(H^{+})=cv=0.0197x0.05=0.000985 \text{ mol}$
 - b) n(OH⁻)=cv=0.1x0.00562=0.000562 mol
 - c) $n(HCl)_{absorbed} = n(HCl)_{original} n(HCl)_{leftover} = 0.000984 0.000562 = 0.000423 mol since <math>n(NH_3) = n(HCl) = n(N) = 0.000423 mol m(N) = 0.000423 x 14.01 = 0.005926 g$ so % N = (0.005926/1.2540) x 100 = 0.473 %
- 4) .
 - a) 2.5
 - b) $n(MnO_4)=cv=0.02023x0.03008=0.00060852 \text{ mol}$ $n(SO_3)=2.5n(MnO_4)=2.5x0.00060852=0.00152 \text{ mol} = n(Na_2SO_3)$
 - c) $m(Na_2SO_3) = nM = 0.00152x126.04 = 0.192 = 192 \text{ mg} = >192 \text{mg}/1 \text{kg} = 192 \text{ ppm}$
 - d) to ensure all HSO₃ ions are removed.
 - e) pink colour of MnO₄ will change to colourless Mn²⁺

5) .

a)
$$2Cr_2O_7^{2-} + 16H_{(aq)}^+ + 3CH_3CH_2OH \rightarrow 4Cr^{3+} + 11H_2O + 3CH_3COOH$$

b)
$$n(Cr_2O_7^{2-})=cv=0.1005x25/1000=0.002513 \text{ mol}$$

c)
$$n(Cr_2O_7^{2-})_{\text{excess}} = n(\text{Fe}^{2+})$$

reaction is $Cr_2O_7^{2-} + 14H_{(aq)}^+ + 6\text{Fe}^{2+} \rightarrow 2Cr^{3+} + 7H_2O + 6\text{Fe}^{3+}$
 $n(\text{Fe}^{2+}) = \text{cv} = 0.3005 \times 0.02355 = 0.0070768 \text{ mol}$
 $n(Cr_2O_7^{2-})_{\text{excess}} = \frac{1}{6} \text{x} 0.0070768 = 0.0011795 \text{ mol}$

d)
$$\text{n}(Cr_2O_7^{2-})_{\text{reacted}} = \text{n}(Cr_2O_7^{2-})_{\text{original}} - \text{n}(Cr_2O_7^{2-})_{\text{excess}} = 0.0025125 - 0.0011795 = 0.0013330 \text{ mol}$$

e)
$$n(CH_3CH_2OH) = \frac{3}{2} n(Cr_2O_7^{2-}) = \frac{3}{2} \times 0.0013330 = 0.0019996 \text{ mol}$$

 $m(CH_3CH_2OH) = \text{nM} = 0.0019996 \times 46.068 = 0.092116 \text{ g in 1 mL}$
so 0.092116 x 100 = 9.21 g per 100 mL

6) .

b)
$$n(MnO_4^-)=cv=0.03004x\ 0.02460=0.0007389\ mol$$
 $n(V^{2+})_{50}=\frac{5}{2}n(MnO_4^-)=2.5x0.0007389=0.000018475$ $n(V^{2+})_{250}=\frac{250}{50}n(V^{2+})_{50}=5x0.000018475=0.00092373\ mol$ $n(V)_{250}=n(V^{2+})_{250}=0.00092373\ mol$ $m(V)=nM=0.00092373\ x\ 50.94=0.4705\ g$ % $V=\frac{0.4705}{0.8765}\ x\ 100=53.68\ \%$