Analytical chemistry year 12

Name _____

1) Which technique is most useful in identifying the levels of mercury in the blood of a patient affected by food contamination?

a) HPLC

b) Flame test

c) ^{13}C NMR

d) Atomic absorption spectroscopy

d) is correct.

1 mark

2) Give a reason for your answer to question 1) above.

HPLC is a separating technique while NMR is used to decipher the molecular structure of organic compounds. Flame test is a crude way of observing metallic ions and does not give an indication of low concentrations of metal pollutants as does AAS.

1 mark

3) A mixture of the following substances are to be separated using gas-liquid chromatography(GLC) with a non-polar stationary phase and nitrogen gas as the mobile phase.

CH_3OH, $CH_3\ CH_2\ CH_2CH_2OH$, $C_{56}H_{90}O_{30}N_{21}S_{12}$, CHOOH

a) Which component of the mixture is not suitable for separation using GLC?

$C_{56}H_{90}O_{30}N_{21}S_{12}$

1 mark

b) Explain your answer to question a) above.

GLC is suitable for low molecular weight compounds (less than 800 amu) that can be readily vapourised without decomposing.

1 mark

c) Place the three molecules that will be separated by GLC in order of longest to shortest retention time.

CH_3CH_2 CH_2CH_2OH , CH_3OH, CH_3COOH

1 mark

d) Give a clear explanation to c) above.

The more polar the molecule the greater its affinity for the mobile phase hence it will have move quickly through the column.

CH_3CH_2 CH_2CH_2OH has a long non-polar section which will interact more with the non-polar stationary phase, while CH_3COOH is the most polar of the three molecules and will interact more with the mobile phase than the non-polar stationary phase.

2 marks

4) A wine from Raz Cellars is analysed for its alcohol content. A 20.0 mL sample of the wine is placed in a 250 mL volumetric flask and distilled water added to the mark. A 25.0 mL aliquot is taken from the volumetric flask and titrated against 0.100 M $K_2Cr_2O_7$. The titration is repeated 5 times and the titres obtained are shown in the table below.

Titre	Volume (mL)
1	19.98
2	20.03
3	18.99
4	19.95
5	20.33

a) Calculate the average titre?

(19.98 + 20.03 +19.95)/3 = 19.99 mL

2 mark

b) Calculate the amount, in mol, of $Cr_2O_7^{2-}$ used in the average titre in a) above.
$n = C \times V = 0.100 \times 0.01999 = 2.00 \times 10^{-3}$

2 marks

c) The equation for the reaction between ethanol and $Cr_2O_7^{2-}$ is a redox reaction involving the oxidation of CH_3CH_2OH to CH_3COOH and the reduction of $Cr_2O_7^{2-}$ to Cr^{3+} .

i) Give the half equation for the oxidation of CH_3CH_2OH to CH_3COOH

$CH_3CH_2OH(aq) + H_2O(l) \Rightarrow CH_3COOH(aq) + 4H^+(aq) + 4e^-$

2 marks

ii) Give the half equation for the reduction of $Cr_2O_7^{2-}$ to Cr^{3+}

$Cr_2O_7^{2-}(aq) + 14H^+(aq) + 6e^- \Rightarrow 2Cr^{3+}(aq) + 7H_2O(l)$

2 marks

iii) Give the overall equation.

$2Cr_2O_7^{2-}(aq) + 16H^+(aq) + 3CH_3CH_2OH(aq) \Rightarrow 4Cr^{3+}(aq) + 3CH_3COOH(aq) + 11H_2O(l)$

2 marks

d) Calculate the amount, in grams, of ethanol, in the 25.0 mL aliquot of diluted wine.

2 marks

$n_{ethanol}$ is 3/2 times the mol of $Cr_2O_7^{2-}$ used according to the equation above..

⇨ *$n_{ethanol} = 2.00 \times 10^{-3} \times 3/2 = 3.00 \times 10^{-3}$*

⇨ *mass $_{ethanol} = 3.00 \times 10^{-3} \times 46.0 = 0.138$ grams*

e) Calculate the amount, in grams, of ethanol in the 20.0 mL original sample of wine.

$n_{ethanol\ in\ original\ sample} = (250.0/25.0) \times 0.138 = 1.38$ g

1 mark

f) If the density of ethanol is 0.789 g/mL what volume of ethanol was present in the 20.0 mL sample of wine?

V =m/D = 1.38/ 0.789 = 1.75 mL

1 mark

g) What is the percentage by volume (%v/v) of ethanol in the wine expressed in the appropriate number of significant figures?
(1.75/20.0) X 100 = 8.75% v/v

1 mark

h) Errors due to rinsing of equipment readily occur in volumetric analysis. What will be the outcome on the final value of the % v/v of ethanol in each of the following scenarios given in the table below. Circle the appropriate response.

Error	Outcome on the final %v/v of ethanol in the wine.
Water was left in the 25.0 mL pipette.	less
Water was left in the 250 mL volumetric flask.	no change
The burette was washed with water and not left to dry.	Greater

3 marks

5) Below is a list of analytical techniques.

^{1}H NMR, ^{13}C NMR, Mass spectroscopy, Atomic absorption spectroscopy, UV-visible spectroscopy, IR spectroscopy, Emission spectroscopy, Flame test, High pressure liquid chromatography, GLC.

a) Which techniques involve the excitation of electrons to higher energy levels?
   AAS, UV-visible, emission, flame test
b) Which techniques involve the excitation of nucleons to higher energy levels?
   ^{1}H NMR, ^{13}C NMR
c) Which technique involves the absorption of electromagnetic radiation to change the behaviour of bonds within a molecule?
   IR
d) Which technique involves the destruction of the sample under investigation?
   MS
e) Which techniques can be used to obtain the concentration of a metal atom in a solution?
   AAS,MS,UV-visible
f) Name all the techniques that distinguish between fragments of different mass.
   MS
g) Which technique would you use to separate and identify an organic compound, with a high molecular mass, in blood plasma?
   HPLC
h) Name all the techniques that can be used to identify a compound and its concentration by first producing a calibration curve.
   AAS,UV-visible, MS, IR
i) Which techniques involve the measurement of energy due to electrons returning from a higher energy state?
   Flame test, emission
j) Name two techniques which can be used together to separate, identify and accurately measure concentrations of compounds in a mixture of low molecular mass hydrocarbons?
   GLC => MS

10 marks

6) An impure sample of iron(II)sulphate, weighing 2.45 grams was treated to produce a precipitate of Fe_2O_3. If the mass of the dried precipitate was 0.610 calculate the percentage of iron in the sample given to appropriate number of significant figures

Mol of Fe_2O_3 = m/M = 0.610/159.8 = 3.82 X 10^{-3}
Mol of fe = 2 X 3.82 X 10^{-3} = 7.64 X 10^{-3}
Mass of Fe = n X M = 7.64 X 10^{-3} X 55.9 = 0.427 g
% of Fe in sample = (0.427 / 2.45) X 100 = 17.4%

4 marks

7) A water way was contaminated with barium chloride. A 100.00 mL sample of the contaminated water was reacted with excess sodium sulphate to produce the white precipitate barium sulphate ($BaSO_4$). After the reaction the precipitate was filtered and repeatedly washed. Periodically the filtrate is analysed by reacting it with silver nitrate until no more white precipitate (AgCl) formed. At this point the barium sulphate was dried and weighed. A mass of 1.13 g of $BaSO_4$ was recorded.

a) Why was it necessary to test the filtrate with silver nitrate?

To make sure that all the salts (spectator ions) have been washed from the precipitate leaving pure $BaSO_4$ in the filter paper.

1 marks

b) Calculate the concentration of barium in the water in ppm. Give the answer to the right number of significant figures.
mole of $BaSO_4$ = 1.13/ 233.4 =.00484
mass of Ba = 0.00484 X 0.00484 =0.665
ppm = mg/L = 665/0.1 = 6650 ppm

3 marks

c) What would happen to the concentration of barium calculated if the filtrate were not tested with silver nitrate? Write the ionic equation for the reaction taking place to produce AgCl.
It would be artificially high.
Ag^+ (aq) + Cl^-(aq) => AgCl(s)

2 mark