FACULTY OF SCIENCE

DEPARTMENT OF APPLIED CHEMISTRY
NATIONAL DIPLOMA: ANALYTICAL CHEMISTRY (4 YEARS)

MODULE   CET1BA3
ANALYTICAL CHEMISTRY 2 (THEORY)

CAMPUS   DFC

NOVEMBER EXAMINATION

DATE: 07/11/2014   **SESSION:**   8:30 – 11:30

ASSESSORS   **DR N MABUBA**

INTERNAL MODERATOR   **DR M MAOELA**

DURATION   3 HOURS   **MARKS 120**

NUMBER OF PAGES: 5 PAGES INCLUDING 1 ANNEXURE

INSTRUCTIONS: CALCULATORS ARE PERMITTED (ONLY ONE PER STUDENT).

REQUIREMENTS: ONE ANSWER SCRIPT.

INSTRUCTIONS TO STUDENTS:

1.   Answer **all** the questions. Questions may be answered in any order as long as each answer is clearly numbered.

2.   Report all numerical answers to the **correct number of significant figures** and with the **appropriate units**. Marks will be deducted for incorrect significant figures and answers without units.

3.   Report p – values to 3 decimals digits.

QUESTION 1 PRECIPITATION TITRATIONS

1.1   The Volhard method was used for the following analysis:
A 2.0412 g sample that contains only sodium chloride and barium iodide was dissolved and diluted to a volume of 250.00 mL volumetric flask. A 25.00 mL aliquot of the sample was treated with 39.02 mL of a 0.09876 M $AgNO_3$ solution and the excess Ag^+ was back-titrated with 13.98 mL of a 0.1045 M SCN^-.
[Molar masses (g mol $^{-1}$): NaCl = 58.443; BaI_2 = 391.15]

1.1.1   Calculate the weight percentages of NaCl and BaI_2 in the sample.   (13)
1.1.2   Which indicator will be used to detect the endpoint in a Volhard titration?   (1)
1.1.3   Use reaction equations to illustrate how the endpoint is detected in Volhard titration.   (3)
1.1.4   The AgCl solid is removed by filtration before the back-titration of the excess silver nitrate. Explain why this is necessary. You may use reaction equations to aid your explanation.   (4)

1.2   Consider the titration of 21.00 mL of a 0.0325 M barium bromide solution with 0.0470 M $AgNO_3$.

1.2.1   Calculate the volume of $AgNO_3$ at the equivalence point.   (3)
1.2.2   Calculate pAg after addition of 10.00 mL of the $AgNO_3$ reagent.   (7)
[Ksp (AgBr) = 5.0×10^{-13}]
1.2.3   How does the concentration of the solution containing the analyte affect the shape of the precipitation titration curve?   (3)

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QUESTION 2 COMPLEXOMETRIC TITRATIONS

2.1   Discuss the following with respect to EDTA titrations and give applicable examples:

2.1.1   An auxillary complexing agent   (2)
2.1.2   Methods based on the use of Eriochrome Black T indicator for an added metal cation.   (6)

Question 2 (CONTINUED)

2.2   The Pb^{2+} and Ni^{2+} in a 20.00 mL sample were treated with 36.67 mL of 0.01722 M EDTA solution to complex all Pb^{2+} and Ni^{2+}. The excess EDTA was determined by back-titration with 10.79 mL of 0.01152 M Mg^{2+} standard solution. An excess of 2, 3 − dimercaptol−1−propanol was introduced to displace nickel from its EDTA complex. The titration of the liberated EDTA required 22.97 mL of 0.009865 M Zn^{2+} standard solution to reach the endpoint. Calculate the molar concentration of Pb^{2+} and Ni^{2+} in the original sample.   (9)

2.3   List three advantages of using multidentate ligands in complexometric titrations.   (3)

2.4   Define the following terms:

2.4.1   *Hard water*   (2)
2.4.2   *Coordination number*   (1)

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QUESTION 3 SEPARATION METHODS

3.1   A solute with a distribution coefficient of 5.0 is extracted from 40.0 mL of solvent 1 into solvent 2.

3.1.1   What volume of solvent 2 is required to extract 90% of the solute in only one extraction?   (4)
3.1.2   What is the total volume of solvent 2 needed to remove 90% of the solute in three equal volume extractions?   (4)

3.2   The following retention times and base line peak widths were obtained from a chromatographic column with a length of 100 cm:

Component	Retention time (s)	Peak width (s)
H_2	160	16
CO_2	190	19

3.2.1   Calculate the resolution obtained between H_2 and CO_2 peaks. **Comment on the separation**.   (4)
3.2.2   Calculate the number of theoretical plates using the data of CO_2 component.   (3)
3.2.3   Calculate the plate height of the column using the data of CO_2 component. **Comment on the efficiency of the column**.   (4)

3.3   Explain the principles on which gas−liquid chromatography is based.   (3)

3.4   Define the term, *chelate*.   (2)

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QUESTION 4: GRAVIMETRY

4.1   Explain the principles of volatilization methods in gravimetric analysis.   (3)

4.2   Particle size of precipitates is related to relative supersaturation of the system according to the equation below:

$$Relative\ supersaturation = \frac{Q-S}{S}$$

Where Q is the concentration of species at any instant and S is the equilibrium solubility of the precipitate.

4.2.1   State the relationship between the particle size and relative supersaturation of the system.   (1)

4.2.2   Use this equation to discuss the control of experimental conditions to ensure crystalline precipitates.   (6)

4.3   A sedimentary rock sample with mass 0.7600 g gave an ignited residue of mixed oxides, SeO_3 and As_2O_3 with a mass of 0.0704 g. In a separate analysis the original sample was found to contain 2.65% Se (w/w). Calculate the weight percentage As in the sedimentary rock sample.
[Molar masses (g mol^{-1}): As_2O_3 = 197.8414; SeO_3= 126.9582; As = 74.9216; Se = 78.96]   (10)

4.4   Colloidal precipitates are prone to co-precipitation due to mixed crystal formation.

4.4.1   Define *mixed crystal formation* and give one example.   (3)

4.4.2   Give one experimental way to minimize co-precipitation due to mixed crystal formation.   (2)

4.5   A 0.7532 sample of feldspar was decomposed; sodium and potassium were isolated as mixed precipitate of sodium chloride and potassium chloride with a mass of 0.2205 g. This mixed precipitate was dissolved in a mixture of water and alcohol, treated with an excess of chloroplatanic acid (H_2PtCl_6), precipitating only the potassium as K_2PtCl_6. The K_2PtCl_6 precipitate was ignited in hydrogen gas and after washing the residue with water, 0.0885 g if metallic platinum was obtained. Calculate the weight percentage of Na_2O in the feldspar sample.
[Molar masses (g mol^{-1}): NaCl = 58.443; KCl = 74.551; Pt = 195.078; Na_2O = 61.979]   (10)

4.6   Calculate the minimum volume of a 0.80 M H_2SO_4 solution required to completely precipitate barium as barium sulphate from 1.50 g sample of barium chloride dehydrate ranging in purity from 80 to 90% (w/w), given that at least a 25% excess of sulphate ions is needed to ensure a complete precipitation and minimise solubility losses.
[Molar masses (g mol^{-1}):$BaCl_2 \cdot 2H_2O$ = 244.263]   (6)

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