UNIVERSITY OF JOHANNESBURG

FACULTY OF SCIENCE

DEPARTMENT OF APPLIED CHEMISTRY

NATIONAL DIPLOMA: ANALYTICAL CHEMISTRY (4 YEARS)

MODULE CET1BA3
ANALYTICAL CHEMISTRY 2 (THEORY)

CAMPUS **DFC**

DECEMBER EXAMINATION

DATE: 01/12/2014 **SESSION: 8:00 – 11:00**

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 Mohr method was used for the titration of 22.00 mL of a 0.0315 M calcium chloride solution with a 0.0755 M $AgNO_3$ solution.
[For AgCl: $K_{sp} = 1.82 \times 10^{-10}$]

1.1.1 Give reaction equation(s) to illustrate how the endpoint is detected in the Mohr method. (2)

1.1.2 Describe two ways of correcting positive systematic errors that may result from using a lower concentration of the indicator. (4)

1.1.3 Give the pH range at which the Mohr method must be carried out. (1)

1.1.4 Calculate the pAg after the addition of 10.00 mL of the $AgNO_3$ solution. (7)

1.2 A 0.4347 g sample, containing only potassium chloride and magnesium bromide, was dissolved and diluted to a final volume of 200.00 mL in a volumetric flask. A 25.00 mL aliquot of this solution required 13.69 mL of a 0.05064 M $AgNO_3$ solution to completely react with all bromide and chloride in the sample. Calculate the weight percentages of bromide and chloride in the sample.
[Molar masses (g mol^{-1}): KCl = 74.551; $MgBr_2$ = 184 113; Br = 79.904; Cl = 35.405] (14)

1.3 Fajans method was used for the precipitation titration of a sample containing chloride ions.

1.3.1 Name the indicator that is used in Fajans method. (1)

1.3.2 Explain the principles on which the adsorption indicator in Fajans method operates, by using the titration of chloride ions with silver nitrate as an example. (7)

[36]

QUESTION 2 COMPLEXOMETRIC TITRATIONS

2.1 Discuss the influence of the following parameters on the shape of the EDTA titration curve. (2)

2.1.1 Concentration of an auxillary complexing agent (2)

2.1.2 pH (3)

Question 2 (CONTINUED)

2.2 In EDTA titrations, back-titration is one of the methods that is used.

2.2.1 List three circumstances in which an EDTA back-titration might be necessary.
2.2.2 Explain the principles of back-titration method that can be used in EDTA titrations. (5)

2.3 A 0.3652 g piece of alloyed steel sample which is composed of nickel, zinc and other impurities was dissolved in acid and diluted to a volume of 250.00 mL in a volumetric flask. A 25.00 mL aliquot of this solution was treated with 40.00 mL of 0.01563 M EDTA solution to complex all Ni^{2+} and Zn^{2+}. The excess EDTA was determined by back-titration with 11.26 mL of a 0.01056 M standard Mg^{2+} solution. An excess 2, 3 – dimercapto-1-propanol was added to displace zinc from its EDTA complex. The liberated EDTA required 22.63 mL of the 0.01056 M of standard Mg2+ solution to reach the endpoint. Calculate the weight percentages of nickel and zinc in the steel alloy.
[Molar masses (g mol^{-1}): Ni = 58.693; Zn = 65.39] (13)

2.4 Define the following terms:

2.4.1 *Masking agent* (2)
2.4.2 *Multidentate ligand* (2)

[30]

QUESTION 3 SEPARATION METHODS

3.1 Copper (solute) has a distribution coefficient of 4.0 between water and acetone. In an experiment you want to extract the copper (solute) from 80 mL sample of water. Do a calculation to prove that it is more efficient to do two consecutive extractions with 40 mL of acetone than one extraction with 80 mL of acetone. (6)

3.2 Calculate the minimum number of extractions needed to achieve 95 % extraction for a solute of KD = 3.0, if the volumes of the original solvent 1 and of the solvent 2 (extractant) are both equal to 100 mL. (5)

3.3 The following retention times and base line peak widths were obtained from a chromatographic column with a length of 90 cm:

Mobile phase	Retention time (s)	Peak width (s)
Air	15	–
Acetone	140	16
Ammonia and butanol	170	19

3.3.1 Calculate the resolution obtained between the vitamin C and Citric acid peaks. **Comment on the separation**. (3)
3.3.2 Calculate the average number of theoretical plates for the column. (4)
3.3.3 Calculate the plate height of the column using the average number of theoretical plates. **Comment on the efficiency of the column**. (3)

QUESTION 3 (CONTINUED)

3.4 Explain the principles on which column chromatography is based. (4)

3.5 Differentiate an *eluent* from an *eluate*. (2)

[27]

QUESTION 4: GRAVIMETRY

4.1 Why is gravimetric analysis favoured over volumetric analysis? (2)

4.2 Read the following paragraph from An Introduction to Chemical Analysis by W E Harris, B Kratochvil, 5th Edition, Saunders College Publishing, New York, 1981, p189.

Quantitative precipitation requires low solubility of the precipitated compound. But it is difficult to ensure lo super saturation ratio when precipitating a sparingly soluble compound; consequently co-precipitation of contaminants may become serious. One way to reduce contamination of the precipitate is to control the super saturation ratio during nucleation by adding the precipitating agent slowly and uniformly to the solution containing the sample thereby avoiding a large local excess of the reagent. An elegant technique of this type is the precipitation from homogeneous solution.

4.2.1 Explain in your own words what is meant by the following terms:
4.2.1.1 *Homogeneous solution* (2)
4.2.1.2 *Precipitate* (1)
4.2.2 Discuss the quality of the precipitate obtained from homogeneous solution. (3)

4.3 Humans use iron in the haemoglobin of the red blood cells to transport oxygen from the lungs to the tissues. It is required that an iron supplement must contain: 60 % iron (w/w) on dry basis. A sample of HIV supplement that was bought by South African Health Department was analysed and the following results were reported for the sample on 'as received' basis: 6.55 % moisture, 56.55 % Fe (w/w).
Does this HIV supplement conform to the specification? **Hint: assume you have 100 g of wet sample**) (6)

4.4 Compare the crystalline and colloidal precipitates. (3)

4.5 A 0.5474 metal alloy sample, containing only magnesium and aluminium, was dissolved in acid. Upon treatment with urea in a homogeneous precipitation, magnesium was precipitated as magnesium hydroxide and aluminium as $Al_2O_3 \cdot xH_2O$. This mixed precipitate was filtered off, washed and ignited to produce a residue of magnesium oxide and aluminium (lll) oxide with a total mass of 0.9944g. Determine the weight percentages of magnesium and aluminium in the sample.
[Molar masses (g mol^{-1}): Mg = 24.305; Al = 26.982; MgO = 40.304; Al_2O_3 = 101.961] (10)

[27]