UNIVERSITY OF JOHANNESBURG

FACULTY OF SCIENCE

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

MODULE CET2AY1/CETATA2
ANALYTICAL CHEMISTRY 2AY1 (THEORY)
ANALYTICAL CHEMISTRY 1B (THEORY)

CAMPUS **DFC**

JUNE EXAMINATION

DATE: 04/06/2017 **SESSION: 8:30-11:30**

ASSESSOR **PROF PN NOMNGONGO**

INTERNAL MODERATOR **MS H DU PLESSIS**

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 pH to 3 decimal places.

4. **The question paper must be handed in with the answer script.**

QUESTION 1 STATISTICAL EVALUATION OF DATA

1.1 A standard solution of Na_2CO_3 was prepared by weighing 1.313 (±0.007) g of anhydrous sodium carbonate (Na_2CO_3) and dissolving it in deionized water in a volumetric flask up to a volume of 250.00 (±0.06) mL. Calculate the molar concentration of the Na_2CO_3 solution and the uncertainty associated with it, expressing both numbers to the correct number of significant figures.
[Molar mass (g mol^{-1}): Na_2CO_3 = 105.9888 (±0.0008)] (6)

1.2 Calculate the absolute uncertainty in the answers of the following calculations. Give the answer of the calculation and the calculated uncertainty to the correct number of significant figures.
(The numbers in parentheses are absolute standard deviations.)

1.2.1 $$\frac{[14.18(\pm 0.04) - 4.795(\pm 0.006)]}{3.75(\pm 0.05) \times 1.351(\pm 0.003)} = 1.85245$$ (7)

1.2.2 $y = \log [7.65 (\pm 0.07) \times 10^{-2}] = -1.11634$ (3)

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QUESTION 2 TITRATIONS FOR COMPLEX ACID/BASE SYSTEMS

2.1 Calculate the pH of the following solutions, after writing suitable balanced reaction equations for any reactions that occur.

2.1.1 A solution obtained by mixing 25.0 mL of a 0.0420 M Na_3PO_4 solution with 30.0 mL of a 0.0350 M HCl solution.
[For H_3PO_4: $K_{a1} = 7.11 \times 10^{-3}$; $K_{a2} = 6.32 \times 10^{-8}$ and $K_{a3} = 4.5 \times 10^{-13}$] (6)

2.1.2 A solution obtained by mixing 30.0 mL of a 0.0350 M malic acid (H_2A) solution with 20.0 mL of a 0.0750 M NaOH solution.
[For H_2A: $K_{a1} = 3.48 \times 10^{-4}$; $K_{a2} = 8.00 \times 10^{-6}$] (9)

2.2 How many grams of disodium hydrogen phosphate must be added to 200 mL of a 0.0255 M HCl solution to produce a buffer of pH 7.265?
[For H_3PO_4: $K_{a1} = 7.11 \times 10^{-3}$; $K_{a2} = 6.32 \times 10^{-8}$ and $K_{a3} = 4.5 \times 10^{-13}$]
[Molar mass (g mol^{-1}): Na_2HPO_4 = 141.96] (10)

QUESTION 2 (continued)

2.3 A 50.00 mL aliquot of a 0.0420 M NaOH and a 0.0350 M sodium carbonate (Na_2CO_3) **mixture** is titrated with a 0.0700 M hydrochloric acid solution.
[For H_2CO_3: $K_{a1} = 4.45 \times 10^{-7}$; $K_{a2} = 4.69 \times 10^{-11}$]

2.3.1 Calculate the pH at equivalence point 1. (9)
2.3.2 Calculate the pH at equivalence point 2. (7)
2.3.3 How many distinct end points will be observed for this titration? Fully explain your answer. (6)
[47]

QUESTION 3 APPLICATIONS OF NEUTRALIZATION TITRATIONS

3.1 Explain what is meant by the 'carbonate error' and give *two* approaches which will eliminate this error. (4)

3.2 The protein content in a sample of wheat flour is analysed using the Kjeldahl method. After digesting a 0.6535 g sample of wheat flour, the nitrogen is converted to ammonium ions, and consequently converted to ammonia with NaOH and then distilled into a collection flask containing 25.00 mL of 0.05937 M HCl. The excess HCl is back-titrated with 0.02512 M NaOH, requiring 12.56 mL to reach the bromothymol blue end point. Calculate the weight percent protein in the flour, given that there are 5.70 grams of protein for every gram of nitrogen. Remember to show all the relevant balanced reaction equations for the analysis.
[Molar mass (g mol^{-1}): N = 14.0067] (11)

3.3 In **Question 3.2** a standard solution of NaOH was used in the back-titration of the excess hydrochloric acid. List ***two*** primary standards other than potassium hydrogen phthalate that can be used to standardise a NaOH solution. (2)

3.4 Briefly describe how sulphur in organic or biological materials can be determined by using neutralization titration. (3)

3.5 A 0.5167 g sample containing NaOH, Na_2CO_3 and $NaHCO_3$, alone or in a compatible combination, as well as inert impurities was dissolved in distilled water and diluted to a final volume of 250.00 mL in a volumetric flask. A 25.00 mL aliquot of this diluted sample required 22.25 mL of 0.01351 M HCl for titration to the phenolphthalein end point. Titration of a second 25.00 mL aliquot of this sample required 34.28 mL of the same HCl solution to reach the bromocresol green end point.

3.5.1 What is the composition of this solid sample? (2)
3.5.2 Calculate the weight percent of each known compound present in the solid sample.
[Molar mass (g mol^{-1}): NaOH = 39.997; $NaHCO_3$ = 84.007; Na_2CO_3 = 105.989] (11)
[33]

QUESTION 4 REDOX TITRIMETRY

4.1 In an iodometric titration, a solution of KIO_3 can be used as a source of known quantities of iodine. Explain the basic principles of this method and also give the stoichiometric ratio between KIO_3 and I_2. (4)

4.2 Explain why the concentration of a standard iodine solution will increase with standing. Support your answer with a suitable balanced equation. (3)

4.3 Three multivitamin tablets containing Vitamin C (ascorbic acid, $C_6H_6O_6$) were dissolved and diluted to 250.00 mL in a volumetric flask. A 50.00 mL aliquot of this solution was acidified and treated with 28.32 mL of a standard 0.01305 M iodine solution, resulting in the following reaction:

$$C_6H_6O_6 + I_2 + 2H^+ \rightarrow C_6H_8O_6 + 2I^-$$

The excess iodine was back-titrated with 17.11 mL of a 0.02102 M $Na_2S_2O_3$ solution to reach the end point.

4.3.1 Write out the balanced half-reaction equations and the overall reaction equation for the titration reaction. (3)

4.3.2 Calculate the milligrams of ascorbic acid (Vitamin C) per tablet.
[Molar mass (g mol^{-1}): $C_6H_6O_6$ = 174.110] (8)

4.4 Explain why $KMnO_4$ solutions are filtered before they are standardized. (2)

4.5 List ***two*** advantages of using potassium dichromate as the titrant in redox titration instead of potassium permanganate. (2)

4.6 Explain why the titration of oxalic acid against potassium permanganate solution will yield incorrect results when HCl is used to acidify the solution. (2)

4.7 List ***two*** ways that are used to detect the end point in Karl Fischer titrations. (2)

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APPENDIX

Table: Error propagation in Arithmetic calculations

Type of calculation	Example*	Standard Deviation of y
Addition or subtraction	$y = a + b - c$	$s_y = \sqrt{s_a^2 + s_b^2 + s_c^2}$
Multiplication or division	$y = a \cdot b/c$	$\frac{s_y}{y} = \sqrt{\left(\frac{s_a}{a}\right)^2 + \left(\frac{s_b}{b}\right)^2 + \left(\frac{s_c}{c}\right)^2}$
Exponentiation	$y = a^x$	$\frac{s_y}{y} = x \frac{s_a}{a}$
Logarithm	$y = \log_{10} a$	$s_y = 0.434 \frac{s_a}{a}$
Antilogarithm	$y = \text{antilog}_{10}\, a$	$\frac{s_y}{y} = 2.303\, s_a$

**a, b* and *c* are experimental variables whose standard deviations are s_a, s_b and s_c respectively.

For an amphiprotic salt, NaHA: $$[H_3O^+] = \sqrt{\frac{K_{a2}C_{NaHA} + K_w}{1 + C_{NaHA}/K_{a1}}}$$