



## **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

### **JULY EXAMINATION**

**DATE:** 07/2018

**SESSION:**

**ASSESSOR**

**PROF PN NOMNGONGO**

**INTERNAL MODERATOR**

**MS H DU PLESSIS**

**DURATION**     3 HOURS

**MARKS 120**

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**NUMBER OF PAGES: 5 PAGES INCLUDING 1 ANNEXURE**

**INSTRUCTIONS:** CALCULATORS ARE PERMITTED (ONLY ONE PER STUDENT).  
THE QUESTION PAPER MUST BE HANDED IN WITH THE ANSWER SCRIPT.

**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.

**QUESTION 1 STATISTICAL EVALUATION OF DATA**

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

$$y = \left[ \frac{7.81(\pm 0.04)}{187(\pm 6)} \right] + 3.25(\pm 0.03) \times 10^{-3} = 4.5015 \times 10^{-2} \quad (8)$$

- 1.2 The absolute uncertainty for a pH measurement is as follows:

$$\text{pH} = 3.552 \pm 0.007$$

Calculate the hydronium ion concentration and the absolute uncertainty associated with it and express both to the correct number of significant figures.

(4)  
[12]

**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 20.0 mL of a 0.0240 M  $\text{H}_2\text{C}_2\text{O}_4$  solution with 25.0 mL of a 0.0360 M  $\text{Na}_2\text{C}_2\text{O}_4$  solution.  
[For  $\text{H}_2\text{C}_2\text{O}_4$ :  $K_{a1} = 5.60 \times 10^{-2}$ ;  $K_{a2} = 5.42 \times 10^{-5}$ ] (7)

- 2.1.2 A solution obtained by mixing 50.0 mL of a 0.0750 M  $\text{H}_3\text{PO}_4$  solution with 30.0 mL of a 0.250 M NaOH solution.  
[For  $\text{H}_3\text{PO}_4$ :  $K_{a1} = 7.11 \times 10^{-3}$ ;  $K_{a2} = 6.32 \times 10^{-8}$  and  $K_{a3} = 4.5 \times 10^{-13}$ ] (9)

- 2.2 How many grams of potassium phthalate ( $\text{K}_2\text{P}$ ) solid need to be added to 500.0 mL of a 0.0750 M *o*-phthalic acid ( $\text{H}_2\text{P}$ ) solution to produce a buffer of pH 5.10? Give the appropriate reaction equation/s.  
[For  $\text{H}_2\text{P}$ :  $K_{a1} = 1.12 \times 10^{-3}$ ;  $K_{a2} = 3.91 \times 10^{-6}$ ]  
[Molar mass ( $\text{g mol}^{-1}$ ):  $\text{K}_2\text{P} = 242.314$ ] (10)

### QUESTION 2 (continued)

- 2.3 A 40.00 mL aliquot of a 0.0500 M HCl and a 0.0700 M ethylene diammonium ion ( $\text{H}_2\text{X}^{2+}$ ) **mixture** is titrated with a 0.160 M NaOH solution.  
[For  $\text{H}_2\text{X}^{2+}$ :  $K_{a1} = 1.42 \times 10^{-7}$ ;  $K_{a2} = 1.18 \times 10^{-10}$ ]
- 2.3.1 Calculate the pH at equivalence point 2. (8)
- 2.3.2 Calculate the pH at equivalence point 3. (10)
- 2.3.3 How many distinct end points will be observed for this titration? Fully explain your answer. (5)
- [49]**

### QUESTION 3 APPLICATIONS OF NEUTRALIZATION TITRATIONS

- 3.1 Briefly explain why the concentration of a sodium hydroxide solution will apparently be unaffected by the absorption of carbon dioxide when it is used to titrate an acid sample with bromocresol green as indicator. Use appropriate reaction equations in your explanation. (4)
- 3.2 The *Merck Index* indicates that 10 mg of the medication guanidine,  $\text{CH}_5\text{N}_3$ , may be administered for each kilogram of body weight in the treatment of the medical condition called myasthenia gravis. The nitrogen in a 4-tablet sample, that weighed a total of 7.50 g, was converted to ammonia by using the Kjeldahl method and the evolved ammonia was collected in 100.00 mL of a 0.1750 M HCl solution. The excess acid was back-titrated with 11.37 mL of a 0.1080 M NaOH solution. How many of these tablets represent a proper dose for a patient who weighs 50 kg?  
[Molar mass ( $\text{g mol}^{-1}$ ):  $\text{CH}_5\text{N}_3 = 59.0706$ ] (10)
- 3.3 In Question 3.2 a standard solution of HCl was used in the Kjeldahl method to collect the ammonia. List *two* primary standards that can be used to standardise a HCl solution. (2)
- 3.4 The digestion (decomposition) step is the most time-consuming step during Kjeldahl determination. List **three** ways in which the digestion time can be reduced. (3)
- 3.5 A borehole water sample may contain NaOH,  $\text{Na}_2\text{CO}_3$  and  $\text{NaHCO}_3$ , separately or in compatible combination. A 20.00 mL aliquot of the borehole water sample required 15.44 mL of 0.1965 M HCl for titration to the phenolphthalein end point. Titration of a second 20.00 mL aliquot of this sample required 39.52 mL of the same HCl solution to reach the bromocresol green end point.
- 3.5.1 What is the composition of this borehole water sample? Give a reason for your answer. (2)
- 3.5.2 Calculate the molar concentration of each solute present in the borehole water sample. (8)

**QUESTION 3** (continued)

- 3.5.3 The accuracy of the above method is not entirely satisfactory and relative errors of 1% or more can be expected. Give a brief description of the alternative method which can be used to improve the accuracy when analysing such a mixture.

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**QUESTION 4 REDOX TITRIMETRY**

- 4.1 Why is a Waldon reductor always used with analyte solutions that contain appreciable concentrations of HCl? (2)
- 4.2 Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) can be used as an auxiliary oxidizing reagent during sample preparation to convert all the analyte to the same oxidation state. Explain how any excess reagent will be removed before determination of the analyte with a standard reducing agent. (2)
- 4.3 Cerium(IV) and potassium permanganate are both strong oxidizing agents used in redox titrations. List **two** disadvantages of using cerium(IV) instead of potassium permanganate in redox titrations. (2)
- 4.4 Explain why KMnO<sub>4</sub> solutions are generally stored in dark reagent bottles. (2)
- 4.5 The sodium hypochlorite (NaOCl) content of household bleach is analysed by diluting 10.00 mL of the bleach solution to a volume of 200.00 mL in a volumetric flask. A 25.00 mL aliquot of the diluted bleach solution is treated with an excess of potassium iodide in an acidic solution, resulting in the liberation of iodine:
- $$\text{OCl}^- + 2\text{I}^- + 2\text{H}^+ \rightarrow \text{Cl}^- + \text{I}_2 + \text{H}_2\text{O}$$
- The liberated iodine is titrated with 29.98 mL of a 0.1233 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution to reach the end point.
- 4.5.1 Write out the balanced half-reaction equations and the overall reaction equation for the titration reaction. (3)
- 4.5.2 Calculate the weight/volume percent of NaOCl in the household bleach.  
[Molar mass (g mol<sup>-1</sup>): NaOCl = 74.4419] (8)
- 4.6 In Question 4.5 a standard solution of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> was used in the titration of the generated I<sub>2</sub>.
- 4.6.1 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> is not a primary standard. Give two primary standards other than KIO<sub>3</sub> that can be used to standardise Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. (2)
- 4.6.2 Which indicator can be used to detect the end point in this titration? (1)
- 4.6.3 Suggest when this indicator will be added and give a reason for your answer. (2)
- 4.6.4 Give the expected colour change for this indicator at the end point. (1)
- 4.7 List **two** precautions that need to be taken when using Karl Fischer reagent. (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: 
$$[\text{H}_3\text{O}^+] = \sqrt{\frac{K_{a2}C_{\text{NaHA}} + K_w}{1 + C_{\text{NaHA}} / K_{a1}}}$$