

UNIVERSITY OF SWAZILAND

FINAL EXAMINATION 2017/2018

TITLE OF PAPER: THERMAL AND ELECTROANALYTICAL METHODS

COURSE NUMBER: CHE609

TIME: THREE (3) HOURS

INSTRUCTIONS:

This Examination paper has **two sections**, A and B. You are required to answer four (4) questions (in total), **with at least one** question from each section

NB: Each question should start on a new page.

A data sheet and a periodic table are attached

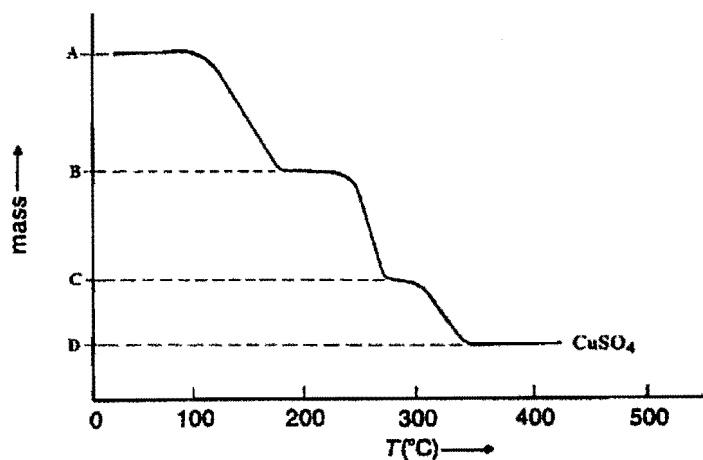
A non-programmable electronic calculator may be used

DO NOT OPEN THIS PAPER UNTIL PERMISSION TO DO SO HAS BEEN GRANTED BY THE CHIEF INVIGILATOR.

SECTION A

Question 1 [25]

- a) A 0.6025 g sample was dissolved, and the Ca^{2+} and Ba^{2+} ions were precipitated as $\text{BaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ and $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$. The oxalates were then heated in a thermogravimetric apparatus leaving a residue that weighed 0.5713 in the range of 320°C to 400°C and 0.4673 g in the range of 280°C to 620°C . Calculate the percentage Ca and the Ba in the Sample. [10]
- b) The figure below was obtained by subjecting $\text{CuSO}_4 \cdot n\text{H}_2\text{O}$ to thermal gravimetric analysis (TGA). The transition from A to D represents the loss of all water of crystallization. The sample masses at points A and D were 12.484 mg and 7.980 mg respectively. Estimate the value of n at each of the points A, B, C and D. [8]



- c) Describe the techniques of evolved gas analysis (EGA) and their use in combination with other thermal analysis techniques. Give examples and applications [7]

Question 2 [25]

- a) What are the main differences between differential thermal analysis (DTA) and differential scanning calorimetry (DSC). Include in your answer the different instrumentation and different results obtained. [16]

- b) A simultaneous technique of TG and DTA is often used nowadays. In this case, a DTG, DTA and TG curves are obtained. Would you expect to have analogous peaks for the DTG and DTA plots for any given sample? Explain your answer with reference to the fundamental definitions [4]
- c) A compound that contains Cu(II), Ammonia, and chlorides is subjected to TG analysis. A 50.0 mg sample of the compound had a weight loss of 28.2mg. If all the loss is ammonia, what is the formula of the sample? [5]

Question 3 [25]

An undergraduate student melt-mixed ground powder of sugarcane bagasse with Ethylene vinyl acetate polymer in order to improve the thermal stability of the resultant polymer composite. As a graduate student mentor, advise the student on the instrument (s) that can be used to see the effect of adding the cellulosic material. In your advice, explain fully why that particular instrument (s) should be used. Also assist the student in outlining a brief procedure that can be used to determine if the cellulosic material improved the thermal stability of the polymer. [25]

QUESTION 4 [25]

- a) For both thermometric titration(TT) and direct injection enthalpimetry (DIE) experiment discuss:
- The parameters that must be known prior to their successful application [3]
 - How relevant data are usually obtained from their respective curves. [3]
- b) For the adiabatic cell of the TT experimental set up
- Discuss its main function
 - Give a typical example
 - How is its performance evaluated?
 - What physical feature of the cell enhances its performance and how? [4]
- c) A thermometric titration was carried out at 25⁰C for the reaction
- $$M + L \leftrightarrow ML$$
- The following data were obtained

Time (s)	Heat Evolved (cal.)
5.0	1.95
10.0	3.87
15.0	5.73
20.0	7.42
25.0	8.68
30.0	9.30
35.0	9.56
40.0	9.69
50.0	9.89
60.0	9.97
70.0	10.0
80.0	10.0

Given that the initial sample concentration for both (M) and (L) was 0.01 M, and that the titration rate was 0.04 mL/s

- I) Sketch the appropriate titration curve [4]
- II) Calculate the equilibrium constant K and ΔG [4]
- III) Identify the equivalent point and calculate the corresponding titrant volume [2]

(Take Gas constant, $R = 1.9872 \text{ cal/kmol}$)

SECTION B

QUESTION 5 [25]

- a) Briefly discuss the following electrochemical techniques and give examples of their uses:
 - i. Electrogravimetry [5]
 - ii. Coulometry [5]
 - iii. Potentiometry [5]
 - iv. Voltammetry [5]
 - v. Cyclic voltammetry [5]

- b) Explain using examples why electrochemistry is well suited for chemical and biochemical sensing applications. What types of sensors have been developed? What are their advantages and special features? [5]

QUESTION 6 [25]

- a) Describe how a student can use anodic stripping voltammetry for the detection of Pb^{2+} in a water sample. Use diagrams to illustrate your answer.



- b) Describe and discuss electrode modification, its importance and applications [10]

THE END

/100/
