CHUKA  **UNIVERSITY**

UNIVERSITY EXAMINATIONS

EXAMINATION FOR THE AWARD OF DEGREE OF BACHELOR OF SCIENCE

CHIN 433: INDUSTRIAL PHYSICAL METHODS OF ANALYSIS

STREAMS: BSC **TIME: 2 HOURS**

DAY/DATE: WEDNESDAY 15/04/2020 **2.30 P.M. – 4.30 P.M.**

INSTRUCTIONS: ANSWER QUESTION ONE AND ANY OTHER TWO QUESTIONS

QUESTION ONE (30 MARKS)

(a) Explain why H and He cannot be detected by Auger electron spectroscopy [1½ marks]

(b) Why are differential spectra preferred in AES analysis? Why are direct spectra often used for quantitative analysis? [2 marks]

(c) Why are Mg $K \propto$ and $Al\ K \propto$ used as x-ray sources for xPs? Do they excite 1s photoelectron emission of metals such as Ti and Fe? If not, how do we detect such elements? [3 marks]

(d) Explain the differences in electron energy analysis for AES and XPS [3 marks]

(e) What are the two main types of detectors used in AES and XPS systems? Explain how CMA work. [5 marks]

(f) How can you tell Auger peaks from photoelectron peaks in an XPS spectrum?[1½ marks]

(g) Why is AES a more qualitative than quantitative techniques? [1½ marks]

(h) Outline the three steps of Auger spectroscopy. [1½ marks]

(i) What are the characteristics differences of AES and XPS? [4 marks]

(j) Draw the energy level diagram and write down the kinetic energy equations for Auger electron spectroscopy [3 marks]

(k) List the similarities of AES and XPS [3 marks]

QUESTION TWO (20 MARKS)

(a) Which method, AES or XPS, is better for resolving chemical shifts and why? Which direction would a peak in a given spectrum move if an atom loses a valence charge? What if the atom gains a valence charge? [2 marks]

(b) Label the following analyzer [2 marks]

(c) Describe the working principle of ion scattering spectroscopy. [7 marks]

(d) Why is an Auger spectrum presented as a derivative plot? [1½ marks]

(e) State the advantage of using Auger for elemental analysis relative to other techniques [1½ marks]

(f) State the analytical applications of Sims. [4 marks]

(g) Outline three modes in which ion beam can be utilized in ion microprobe mass spectrometry. [2 marks]

QUESTION THREE (20 MARKS)

(a) (i) In the accompanying figure curve A is the weight-loss thermogram from pure $CaCO_3$, curve C shows a similar trace from $MgCO_3$, and Curve B is the thermogram of a limestone sample. [6 marks]

(I) Derive an expression for the direct quantitative analysis of CaO and MgO

(II) Write equations for the solid-state decomposition of $MgCO_3$

(III) Calculate the percent CaO and MgO in the limestone sample

(ii) A simultaneous DTA-TG curve manganese (H) carbonate in a porous crucible is shown (solid line) [2½ marks]

(I) What are the transitions involved at each peak on the DTA trace, and what are the products at each TG plateau?

(II) Another laboratory, using a controlled atmosphere with 13 atm CO_2, obtained the curves show (dashed line). Why is the initial oxide different?

(iii) From the thermomechanical penetration curve shown, deduce the nature of the two transitions [1 mark]

(iv) Three successive runs on the same sample of a fiberglass mat impregnated with an uncured epoxy resin are shown. The scan in run A was stopped at 90°C, and the sample cooled and rerun (run B). Run C is the sample from run B after cooling. Discuss the features observed in the DSC scans. [3 marks]

(b) Give the advantages and disadvantages of differential scanning calorimetry [7½ marks]

QUESTION FOUR (20 MARKS)

(a) Write short notes on sample characteristics in thermogravimetric analysis [6½ marks]

(b) The following data were obtained from the sample containing magnesium oxalate and magnesium oxide using TGA

Original weight of the sample	25.0 mg
Weight of the sample after heating to 500°C	10.40 mg

Determine the percentage of magnesium oxalate and magnesium oxide in the sample. [3½ marks]

(c) Calculate the percentages of calcium oxalate and magnesium oxalates in the sample containing the mixture of only the two given that, the weight loss above 600°C was 18%. [5 marks]

(d) Discuss the classification of thermogravimetric according to their shapes [5 marks]
