



DEDAN KIMATHI UNIVERSITY OF TECHNOLOGY
UNIVERSITY EXAMINATIONS 2021/2022
YEAR ONE SPECIAL/SUPPLEMENTARY EXAMINATION FOR THE DEGREE OF
MASTER OF SCIENCE IN CHEMISTRY
SCH 6104: ADVANCED ANALYTICAL CHEMISTRY

DATE: 11TH APRIL 2022

TIME: 11.00-02.00 P.M.

INSTRUCTIONS: Attempt ALL Questions

QUESTION ONE [15 MARKS]

- a) Describe extraction with regards to separation **[2 Marks]**
- b) A solute with a retention time of 300 s has a width at the base of 13 s. A neighboring peak is eluted at 324 s with a width of 16 s. Find the resolution for these two components. **[3 Marks]**
- c) Substances A and B were found to have retention times of 14.40 and 15.45 min, respectively, on a 30.0 cm column. An unretained species passed through the column in 1.30 min. The peak widths (at base) for A and B were 1.11 and 1.21 min, respectively. Calculate:
- i. the column resolution; **[2 Marks]**
 - ii. the average number of plates in the column. **[2 Marks]**
 - iii. the plate height. **[1 Mark]**
 - iv. the length of column required to achieve a resolution of 1.5; and **[3 Marks]**
 - v. the time required to elute substance B at a resolution of 1.5. **[2 Marks]**

QUESTION TWO [15 MARKS]

- a) Below is a list of analytical techniques that we have covered during the semester and four analytical “problems” that might be solved using one of these techniques. Match each problem with one technique that you think would best address the problem. Use each technique only once. **[5 Marks]**
- i. HPLC with UV/VIS Absorbance Detection
 - ii. HPLC with Mass Spectrometry
 - iii. Infrared Absorption Spectrometry
 - iv. Differential scanning calorimetry
 - v. Chemiluminescence
 - vi. Mass Spectrometry with electrospray ionization

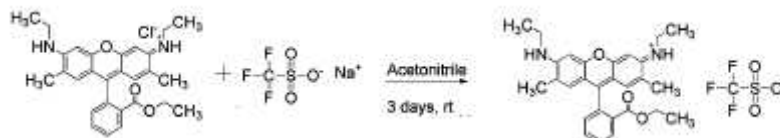
- vii. Gas Chromatography with Flame Ionization Detection
- viii. NMR
- ix. Thermal gravimetric analysis

Technique

Problem

Selected

- A. _____ You have synthesized an organic compound to be used as a candidate for retroviral therapy. You would like to confirm the structure of this compound by accurately determining its molecular weight.
- B. _____ You have a mixture of small proteins (MW = 5,000 to 10,000 Da), and you want to separate and quantify the individual proteins in this mixture.
- C. _____ The reactions below were carried out, and a product was formed. Which technique would you use to try to identify the structure of this product?



- D. _____ You have synthesized a new polymer and you want to determine its glass transition temperature as well as its crystallization and melting point temperatures.
- E. _____ Forensic experts visited the apartment of a murder suspect and wanted to check for traces of blood

- b) With the help of a well labeled Jablonski diagram, discuss Phosphorescence and fluorescence spectroscopy **[10 Marks]**

QUESTION THREE [15 MARKS]

- a) With the help of a diagram, discuss how GC-MS works. **[10 Marks]**
- b) In GC open tubular, narrower and longer columns are preferred. Explain the reason for this preference. **[3 Marks]**
- c) What is column bleeding in GC and what causes it? **[2 Mark]**

QUESTION FOUR [15 MARKS]

- a) Differentiate between Rayleigh and Raman Scattering **[4 Marks]**
- b) With the help of a diagram discuss the instrumentation of Raman scattering. **[8 Marks]**
- a) State three desirable properties of an Atomic Emission Spectrometer. **[3 Marks]**

THE FOLLOWING FORMULAE MAY OR MAY NOT BE USEFUL

$$u = L/t_m$$

$$V_m = t_m F$$

$$k' = (t_r - t_m)/t_m$$

$$K = \frac{k' V_m}{V_s}$$

$$\alpha = \frac{(t_r)_y - t_m}{(t_r)_x - t_m}$$

$$R_s = \frac{2[(t_r)_y - (t_r)_x]}{W_x + W_y}$$

$$R_s = \frac{\sqrt{N}}{4} \left(\frac{\alpha - 1}{\alpha} \right) \frac{k'_y}{(1 + k'_y \alpha' g)}$$

$$N = 16 \left(\frac{t_r}{W} \right)^2 \text{ or } 5.5 \left(\frac{t_r}{W_{1/2}} \right)^2$$

$$(t_r)_y = \frac{16 R_s^2 H}{u} \left(\frac{\alpha}{\alpha - 1} \right)^2 \frac{(1 + k'_y)^3}{(k'_y)^2}$$