KISII UNIVERSITY
UNIVERSITY EXAMINATIONS

SPECIAL/SUPPLEMENTARY EXAMINATIONS
THIRD YEAR EXAMINATION FOR THE AWARD OF THE DEGREE OF
BACHELOR OF SCIENCE IN COMMUNITY NUTRITION AND DIETETICS
FIRST SEMESTER, 2021/2022
(JULY, 2022)

CHEM 308: ANALYTICAL CHEMISTRY I

STREAM: Y3 S1   **TIME: 2 HOURS**

**DAY: MONDAY, 3:00 – 5:00 P.M.**   **DATE: 25/07/2022**

INSTRUCTIONS
1. Do not write anything on this question paper.
2. Answer ALL Questions in Section A and any TWO from Section B.

SECTION A: ATTEMPT ALL QUESTIONS (40 MKS)

1. Define the following: (3 marks)
    i)   Retention factor
    ii)   Elutropic series
    iii) Void Volume

2. Distinguish between: (5 marks)
    i)   Zonal and affinity eluent development
    ii)   Retention time and dead time
    iii) Analytical selectivity and analytical sensitivity
    iv)   Amplitude and wavelength
    v)   Column capacity and column efficiency

3. Repeated application of highly impure samples such as sera, urine, plasma or whole blood may eventually cause the column to lose its resolving power. How would you prevent such an occurrence?   (1 mark)

4. Why would you need a cotton plug at the bottom of a column   (1 mark

5. Describe the distinctive feature between packed and capillary columns
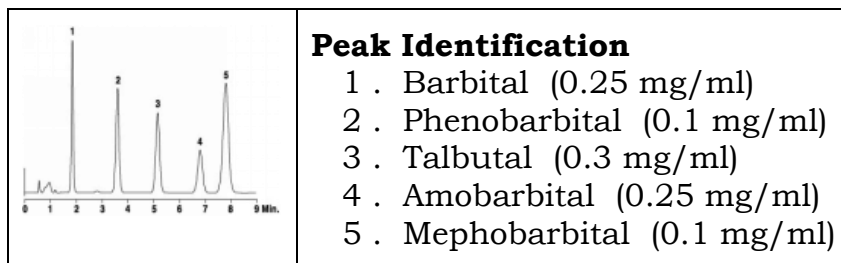   (1 mark)

6. State at least one function of a salt bridge in an electrochemical cell.(1 mark)

7. Describe the theoretical plate model (1 mark)

8. Give a reason why KNO_3 is used in salt bridge (1 mark)

9. Distinguish between Potentiometry and coulometry (1 mark)

10. Consider an electrochemical cell where the following reaction takes place: $2Au^{3+}_{(aq)} + 3Zn_{(s)} \rightarrow 2Au_{(s)} + 3Zn^{2+}_{(aq)}$.

    a. What is the correct cell notation for this cell? (1 mark)

    b. Write the half-cell reactions. (1 mark)

11. Given; $Ni^{2+}_{(aq)} + 2e^- \rightarrow Ni_{(s)}$ $E^0=-0.26V$ and $Sn^{4+}_{(aq)} + 2e^- \rightarrow Sn^{2+}_{(aq)}$ $E^0=-0.15V$

    i). Calculate the standard emf of the cell at 298K (1 mark)

    ii). What is the correct cell notation for this cell? (1 mark)

12. State one application of Ion selective electrodes (1mark)

13. The figure below represents four different types of electromagnetic radiations (a to d). The wavelengths are relative but not to scale. (1 mark)
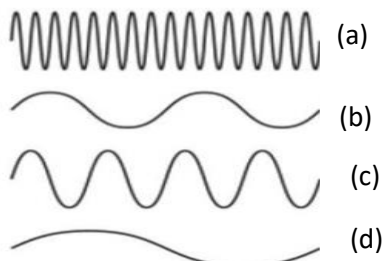
Arrange them in order of:
   i). Decreasing frequency
   ii). Increasing wavelength
   iii).Increasing energy

14. The effect of EMR on interaction with matter depends on energy associated with the radiation.State the effect when matter interacts with: (2 marks)

    i). Very energetic radiations
    ii). Radiations in the infra-red region of the spectrum with moderate energy

15. Why do cuvettes have two frosted faces? (1 mark)

16. Briefly describe the distinctive features between a single beam and a double beam spectrophotometer (1 mark)

17. Distinguish between a monochromatic and a polychromatic light(1 mark)

18. The light dispersion device inside the monochromator is either a _____or a _____. (1 mark)

19. State Beer-Lamberts law (1 mark)

20. What purpose does the blank solution serve in spectrophotometry?(1 mark)

21. Distinguish between Systematic error and random error (1 mark)

22. Unlike other spectroscopy methods, AAS has two additional requirements. Which ones? (2 marks)

23. State one importance of emf measurements (1 mark)

24. Refer to the HPLC chromatogram above for a mixture of barbiturates. Assuming that barbital is more polar than phenobarbital which is more polar than talbutal, etc, was this experiment run under normal or reverse phase conditions? Explain (1 mark)

25. State one cause of systematic errors (1 mark)

Peak Identification
1. Barbital (0.25 mg/ml)
2. Phenobarbital (0.1 mg/ml)
3. Talbutal (0.3 mg/ml)
4. Amobarbital (0.25 mg/ml)
5. Mephobarbital (0.1 mg/ml)

26. How long (in hours) must a current of 5.0 amperes be maintained to electroplate 60 g of calcium from molten $CaCl_2$? (Ca = 40 amu) (1 mark)

27. Arrange the following in order of increasing oxidizing strength: Ni^{2+} (-0.26V); I_2 (0.54V); Cu^{2+} (0.34V); Pb^{2+}(-0.13V); Ag^+ (0.80V). (1 mark)

28. Which of the following options explains the process that occurs in Hollow Cathode Lamp? (1 mark)

   a. *Positive ions collide with negative ions and photons are ejected*
   b. *Negative ions collide with cathode surface and metal atoms from anode are ejected*
   c. *Positive ions collide with cathode surface and metal atoms from cathode are ejected*
   d. *Positive ions collide with negative ions and metal atoms from anode are ejected*

29. A reaction takes place that is expected to yield 171.9 g of product, but only yields 154.8. What is the percent error for this experiment? (1 mark)

30. Why is it necessary to choose a proper combination of fuels and oxidant in Atomic absorption spectroscopy in order to produce recommended temperatures? (1 mark)

31. Under which two circumstances would one choose quartz or glass cuvettes over plasticcuvettes? (1 mark)

SECTION B (Answer Any Two)

QUESTION TWO

A. State and explain two factors that can oppose the formation of a narrow analyte peak thereby causing peak broadening (3marks)

B. Two analytes A and B were separated on a 25 cm long column. The observed retention times were 7 min 20 s and 8 min 20 s, respectively. The base peak width for analyte B was 10 s. When a reference compound, which was completely excluded from the stationary phase under the same elution conditions, was studied, its retention time was 1 min 20 s. What was the resolution of the two analytes? (5marks)

C. Describe the following column types; WCOT, SCOT and PLOT (3marks)

D. State comparative differences between AAS and AES under the following subtopics   (4 marks

  i). Process measured
  ii). Use of flame
  iii). Instrumentation
  iv).Beers law

QUESTION THREE

A. A direct current of 100.0mA flows for 2 hours through three cells in series. They contain solutions of silver nitrate, copper (II) Sulphate and Gold(III) nitrate. Calculate the mass deposited in each.   (4marks)

B. Briefly describe the working of a spectrophotometer   (3marks)

C. Seven pH measurements were made repeatedly on a water sample. The values obtained were 6.52, 6.61, 6.71, 6.64, 6.43, 6.24, and 6.54.

  i. Calculate the precision of the data set.   (3marks)

  ii. Using table 1 below, Calculate the confidence intervals at 95 and 99.9% confidence levels of the pH measurements in A above   (2marks)

  iii. Using the answers in (ii) above, which one of the following readings could not be possible at 99.9% confidence level? a) 6.175.   (b) 6.874   (c) 6.152 (d) 6.732   (1 mark)

Table 1: Values of student's t

Degrees of freedom	Confidence level (%)					
	50	90	95	98	99	99.9
2	0.816	2.920	4.303	6.965	9.925	31.598
3	0.765	2.353	3.182	4.541	5.841	12.924
4	0.741	2.132	2.776	3.747	4.604	8.610
5	0.727	2.015	2.571	3.365	4.032	6.869
6	0.718	1.943	2.447	3.143	3.707	5.959
7	0.711	1.895	2.365	2.998	3.500	5.408
8	0.706	1.860	2.306	2.896	3.355	5.041

  iv. If the data set in the example above contained an additional value of 7.37, could this value be regarded as an outlier point at the 90% and at 99% confidence levels?   (2marks)

Table 2: Q Values at different confidence level

Number of values:	3	4	5	6	7	8	9	10
Q90%:	0.941	0.765	0.642	0.560	0.507	0.468	0.437	0.412
Q95%:	0.970	0.829	0.710	0.625	0.568	0.526	0.493	0.466
Q99%:	0.994	0.926	0.821	0.740	0.680	0.634	0.598	0.568

QUESTION FOUR

A. Separations of hydrocarbons commonly found in auto petroleum were made on a 30-meter long, 0.52-mm diameter DB-1 capillary column. The data was obtained from a GC capillary column with a flame ionization detector. Table below contains the output from a typical integrator.

Analyte	Retention time (mins)	Area	Peak Width at the base (mins)
Solvent, t_m	1.782	Na	NA
Benzene	4.938	598833	0.099
Isooctane	6.505	523678	0.122
n-heptane	6.956	482864	0.100
Toluene	9.256	598289	0.092
Ethyl-benzene	13.359	510009	0.090

Calculate k', α, Rs, H, and N for Toluene and Ethyl-benzene compounds(6mks)

B. Given that (Mg^{2+}/Mg E^0 = -2.36V and Ag+/Ag E^0 = 0.80): Sketch a voltaic cell and on the sketch indicate:
   - i) Signs of electrodes   (2 marks)
   - ii) The cathode and the anode   (2 marks)
   - iii) The direction in which electrons move   (1 mark)
   - iv) Reaction at each electrode   (2marks)
   - v) Calculate cell voltage of the cell described above   (1mark)
   - vi) Show the cell notation   (1 mark)