KIBABII UNIVERSITY

UNIVERSITY EXAMINATIONS
2021/2022 ACADEMIC YEAR

THIRD YEAR SECOND SEMESTER
MAIN EXAMINATIONS

FOR THE DEGREE OF BSC (CHEMISTRY)

COURSE CODE: SCH 323

COURSE TITLE: INSTRUMENTAL ANALYTICAL CHEMISTRY AND QUALITY CONTROL

DATE: 29/08/2022 **TIME:** 2:00PM-4:00PM

INSTRUCTIONS TO CANDIDATES:

- Answer **Question ONE (Compulsory)** and any other **TWO (2)** questions

TIME: 2 Hours

Constants: h = 6.626×10^{-34} Js; c=3.0×10^{8} ms^{-1}

This paper consists of **4** printed pages. Please Turn Over

Question 1 [30 Marks]

a. Differentiate between gravimetry and titrimetry and spectroscopy [3 Marks]
b. Explain the importance of analytical instrumentation [2 Marks]
c. Contrast classical and instrumental techniques as used in analytical chemistry [6 Marks]
d. Explain the choice of gas used in most spectrometers [2 Marks]
e. compare column chromatography and TLC [4 Marks]
f. Define back titration [2 Marks]
g. Thermogravimetric studies of a carbon black filled rubber sample is performed in inert atmosphere up to 950 °C then the atmosphere is quickly changed to air. The observed weight loss is ~ 66.41 % up to 500 °C and the mass becomes constant, a further mass loss is observed between 1300 °C and 2800 °C . Explain the decomposition of rubber.[4 Marks]
h. Discuss qualitative and quantitative measurements [4 Marks]
i. Highlight three analytical techniques which apply X-ray radiation [3 Marks]

Question 2 [20 Marks]

When separating two substances, X and Y, on a 30.0 cm column. A student obtained a chromatogram that gave retention times of 15.80 and 17.23 min for X and Y, respectively, and an elution time of an unretained compound of 1.60 min. The base peak widths for X and Y were 1.25 and 1.38 min, respectively.
Calculate the following

a. Standard deviation of each peak [2 Marks]
b. average number of theoretical plates for the column [5 Marks]
c. plate height [2 Marks]
d. resolution of X and Y and determine whether baseline separation was achieved [3 Marks]
e. length of column that would be required to achieve a resolution of 1.5 [8 Marks]

Question 3 [20 Marks]

a. Explain the difference between normal and reverse phase HPLC and the advantages of each [6 Marks]
b. Describe the procedure for obtaining a HPLC chromatogram [4 Marks]
c. During a routine analysis it was observed that the signal for an interferent I in the analysis of analyte A was 6 units when that of an equimolar solution of A was 40 units.
    i. Calculate the selectivity coefficient of the method. [4 Marks]
    ii. If the concentration of A must be known with an accuracy of ±0.50%, what is the maximum relative concentration of A that can be present? [6 Marks]

Question 4 [20 Marks]

a. Discuss the importance of quality control in chemical analysis [6 Marks]

b. Calculate the potential for the following reactions [4 Marks]

i. $Zn(s) + Pb^{2+}(aq) \rightarrow Zn^{2+}(aq) + Pb(s)$

ii. $Al(s) + Fe^{3+}(aq) \rightarrow Al^{3+}(aq) + Fe(s)$

c. The concentration of copper in a sample of sea water is determined by anodic stripping voltammetry using the method of standard additions. The analysis of a 50.0 mL sample gives a peak current of 0.886 μA. After adding a 5.00 μL spike of 10.0 mg/L Cu^{2+}, the peak current increases to 2.52 μA. Calculate the μg/L copper in the sample of sea water. [10 Marks]

Question 5 [20 Marks]

a. Draw and label the main parts of a spectrophotometric instrument [10 Marks]

b. The %w/w I^- in a 0.67 g sample is determined by a Volhard titration. After adding 50.00 mL of 0.056 M $AgNO_3$ and allowing the precipitate to form, the remaining silver is back titrated with 0.053 M KSCN, requiring 35.14 mL to reach the end point.

Calculate

i. Moles of I^- [6 Marks]

ii. Concentration (%w/w) of I^- in the sample. [4 Marks]

PERIODIC TABLE OF THE ELEMENTS

1 IA	2 IIA	3 IIIB	4 IVB	5 VB	6 VIB	7 VIIB	8	9 VIIIB	10	11 IB	12 IIB	13 IIIA	14 IVA	15 VA	16 VIA	17 VIIA	18 VIIIA
1 H 1.008																	2 He 4.00
3 Li 6.94	4 Be 9.01											5 B 10.81	6 C 12.01	7 N 14.1	8 O 16.00	9 F 19.00	10 Ne 20.18
11 Na 22.99	12 Mg 24.30											13 Al 26.98	14 Si 28.09	15 P 30.97	16 S 32.06	17 Cl 35.45	18 Ar 39.95
19 K 39.10	20 Ca 40.08	21 Sc 44.96	22 Ti 47.90	23 V 50.94	24 Cr 52.00	25 Mn 59.94	26 Fe 55.85	27 Co 58.93	28 Ni 58.69	29 Cu 63.55	30 Zn 65.39	31 Ga 69.72	32 Ge 72.59	33 As 74.92	34 Se 78.96	35 Br 79.90	36 Kr 83.80
37 Rb 85.47	38 Sr 87.62	39 Y 88.91	40 Zr 91.22	41 Nb 92.91	42 Mo 95.94	43 Tc (98)	44 Ru 101.1	45 Rh 102.91	46 Pd 106.42	47 Ag 107.87	48 Cd 112.41	49 In 114.82	50 Sn 118.71	51 Sb 121.75	52 Te 127.60	53 I 126.91	54 Xe 131.29
55 Cs 132.91	56 Ba 137.33	57 *La 138.91	72 Hf 178.49	73 Ta 180.95	74 W 183.85	75 Re 186.21	76 Os 190.2	77 Ir 192.2	78 Pt 195.08	79 Au 196.97	80 Hg 200.59	81 Tl 204.38	82 Pb 207.2	83 Bi 208.98	84 Po (209)	85 At (210)	86 Rn (222)
87 Fr (223)	88 Ra 226.02	89 †Ac 227.03	104 Rf (261)	105 Db (262)	106 Sg (266)	107 Bh (264)	108 Hs (277)	109 Mt (268)	110 Ds (271)	111 Rg (272)							