MACHAKOS UNIVERSITY

University Examinations 2016/2017

SCHOOL OF PURE AND APPLIED SCIENCES

DEPARTMENT OF PHYSICAL SCIENCES

FIRST YEAR SECOND SEMESTER EXAMINATION FOR DEGREE IN

BACHELOR OF EDUCATION (SPECIAL EDUCATION)

BACHELOR OF EDUCATION (SCIENCE)

BACHELOR OF SCIENCE (MATHS)

SCH 103: INTRODUCTION TO CLASSICAL ANALYSIS AND SEPARATION TECHNIQUES

DATE: 5/6/2017 **TIME: 8:30 – 10:30 AM**

INSTRUCTIONS:

- The paper consists of **two** sections.
- Section **A** is **compulsory**.
- Answer any **two** questions from section **B**.

Relevant Tables (Periodic table, T-test, F-test, G-test).

SECTION A (COMPULSORY)
QUESTION ONE (30 MARKS)

a) Define the following terms as used in analytical chemistry

i) Mechanical entrapment (1 mark)

ii) Occlusion (1 mark)

b) Explain the difference between the following terms:

i) Random error and determinate error (2 marks)

ii) Heterogeneous sample and homogenous sample (2 marks)

c) State four (4) properties of a good precipitate as obtained in the process of gravimetric analysis. (2 marks)

d) The results of many measurements of an experimental quantity follow a Gaussian distribution.

i) List an example of one case that we studied for comparison of means and write equation used in this case (2 marks)

ii) A trainee in a medical lab will be released to work on her own when her results agree with those of an experienced worker at the 95% confidence level. Results for a blood urea nitrogen analysis are shown below.

Trainee: $\bar{x}$ = 14.57 mg/dL s = 0.53 mg/dL n = 6 samples

Experienced worker: $\bar{x}$ = 13.95 mg/dL s = 0.42 mg/dL n = 5 samples

Should the trainee be released to work alone? (5 marks)

e) A Chemistry student prepared a solute labeled Y with a partition coefficient of 4.0 between water and dichloromethane.

i) If solute Y, was initially dissolved in 80.0 mL of water. It was then extracted six times with 10.0 mL portions of dichloromethane. Find the fraction of solute remaining in the aqueous phase (3 marks)

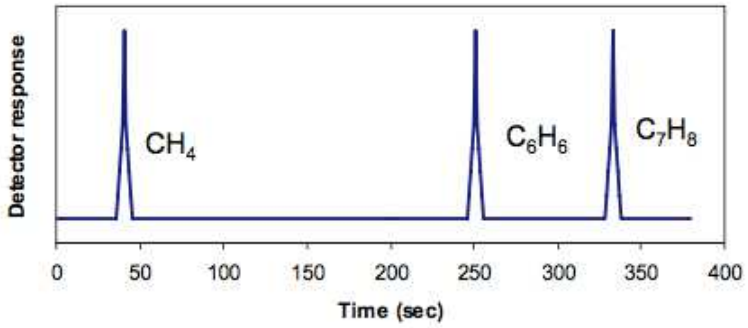
ii) Calculate the concentration of Y in dichloromethane if the concentration of Y in aqueous solution [Y_{aq}] is 0.04 M (1 mark)

iii) State the advantage of carrying out multiple extractions (1 mark)

f) A student was carrying out an experiment using trace amounts of a sample.

Briefly explain three (3) problems that she may encounter during analysis of her sample (3 marks)

g) Using the following chromatogram and assuming that methane (CH_4) is un-retained, calculate the:



i) Adjusted retention time for benzene and toluene (2.5 marks)

ii) Capacity factor for toluene (2.5 marks)

h) Sample X was postulated to contain both propane and propan-2-ol. A student carried out TLC separation on the sample X. Draw the likely outcome of the TLC plate after separation. Explain your diagram (2 marks)

SECTION B: ATTEMPT ANY TWO QUESTIONS

QUESTION TWO (20 MARKS)

a) Explain the following two (2) modes of extraction

i) Single stage operation (2 marks)

ii) Counter current distribution (2 marks)

b) A research scientist isolates a compound (compound X) from a plant and conducts the tests using liquid – liquid extraction to optimize its extraction. Using water and 1-octanol, the following distribution coefficients, KD (= [X]octanol/[X]water) are calculated as a function of pH (with water being the extractant phase): pH 2, 4, 6, 8 and 10: KD 0.008, 0.8, 39, 76 and 75 respectively

i) Does compound X appear to have acidic functional groups, basic functional groups or both types of groups? Explain your answer (3 marks)

ii) If 50 mL of an aqueous plant extract buffered to a pH of 8.0 is extracted with octanol, calculate the volume of octanol needed to extract 98.0% of compound X to the octanol phase (4 marks)

iii) Consider an amine B with a partition coefficient K = 3 with the acid dissociation constant of BH^+, $K_a = 1x10^{-9}$. If 50 mL of 0.01 M aqueous amine is extracted with 100 mL of solvent, calculate the formal concentration remaining in the aqueous phase at

I. pH 10 (3 marks)

II. pH 8 (2 marks)

III. Comment on the results that you have obtained in question 2 part b(iii)I and b(iii)II above. (1 mark)

c) A solute with a partition coefficient of 4.0 is extracted from 10 mL of phase X into phase Y. What volume of phase Y is needed to extract 99% of the solute in one extraction (3 marks)

QUESTION THREE (20 MAKRS)

a) Describe the principles and operational features that distinguish each of the following chromatographic methods of separation.

i) Thin layer chromatography (3 marks)

ii) Gas chromatography (3 marks)

b) On a gas chromatographic column L = 30 m compounds elute in 5 minutes with $W_{1/2} = 5s$, while on a liquid chromatographic column L = 25 cm, compounds elute in 5 minutes with $W_{1/2} = 5s$. For each column, calculate;

i) The number of theoretical plates (2 marks)

ii) The plate height (3 marks)

iii) Which of the two columns will give a better separation. Explain your answer (2 marks)

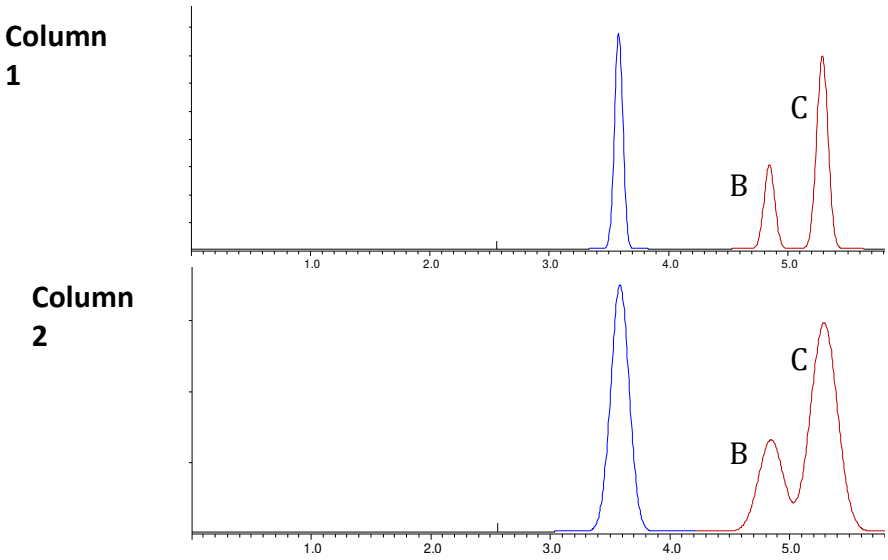
c) Briefly explain how the column and flow rate affect the plate height (4 marks)

d) Chromatograms of compounds B and C were obtained at the same flow rate with two columns of equal length. Indicate the column, which:

i) Has more theoretical plates. (1 mark)

ii) Has larger plate heights (1 mark)

iii) Gives a greater relative retention (1 mark)



QUESTION FOUR (20 MARKS)

a) Differentiate between the following terms

i) Titrimetric analysis and gravimetric analysis (2 marks)

ii) Molarity and normality (2 marks)

b) In determination of iron from a dietary supplement, using gravimetric analysis, it was determined that each tablet provided 15 mg of iron. Calculate the number of tablets that a student should analyze to obtain 0.25 g of Fe_2O_3 product (4 marks)

c) 0.7122 g sample of iron is dissolved in nitric acid (HNO_3) to oxidize all iron present. The solution is filtered to remove solid matrix and made basic with addition of sodium hydroxide. The iron precipitates as the $Fe(OH)_3.xH_2O$ gel. The precipitate is collected in a cistern crucible and ignited to produce Fe_2O_3. Calculate the percentage weight (wt%) of iron if the analysis produced $4.41X10^{-4}$ Kg Fe_2O_3 (4 marks)

d) A solution made from pure barium hydroxide contained 2.74 g in exactly 100 cm^3 of water. Using phenolphthalein indicator, titration of 20.0 cm^3 of this solution required 18.7 cm^3 of a hydrochloric acid solution for complete neutralization. [Atomic masses: Ba = 137, O = 16, H = 1)

i) Write the equation for the titration reaction. (1 mark)

ii) Calculate the moles of barium hydroxide neutralized. (2 marks)

iii) Calculate the molarity of the hydrochloric acid (2.5 marks)

e) Calculate the molality of 40.0 wt% HCl, given that the density of the reagent is 1.19 g/mL (2.5 marks)

QUESTION FIVE (20 MARKS)

a) An effective analytical process should go through various steps. Supposing you are requested by the Kenya Power and Lightening Company (KPLC), to analyze the amount of Arsenic in their chromated copper arsenate (CCA) wood treated timber, Explain in details, the analytical processes that will enable you successively achieve the objectives of KPLC (7 marks)

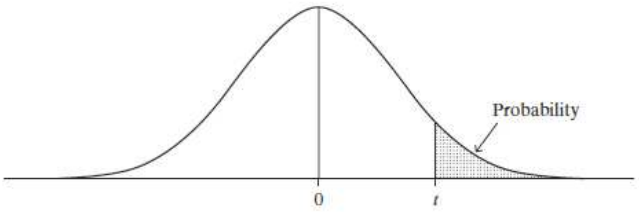
b) A research analyst dissolved silver from a galvanized key ring and measured the mass lost by the key ring to tell how much of it was silver. Here are six (6) results in mass loss %; 10.2, 10.8, 11.6, 9.2, 7.9, 11.3. Which one of these values is an outlier? Should it be retained or discarded before averaging? (4 marks)

c) Six samples were analyzed using two methods, New and Standard. Determine whether these two methods differ significantly at 98% confidence level for the following individual samples from A to F as shown on the table below. (5 marks)

Sample	**New Method**	**Standard method**
A	10.2	10.5
B	12.7	11.9
C	8.6	8.7
D	17.5	16.9
E	11.2	10.9
F	11.5	11.1

d) A standard water sample is known to contain 89 wt% calcium. Using a new analytical method the following wt% of calcium were measured: 102, 97, 99, 98, 101, and 106. At 95% confidence level, determine if the new method is valid. (4 marks)

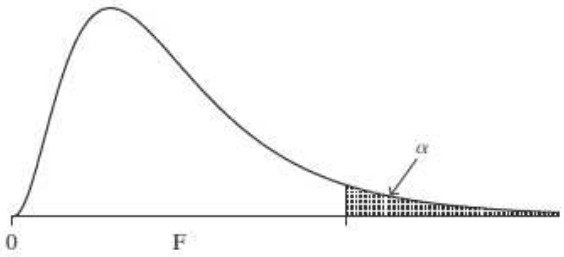
TABLE B: t Distribution Critical Values



	Confidence Level					
	80%	90%	95%	98%	99%	99.8%
	Right-Tail Probability					
df	$t_{.100}$	$t_{.050}$	$t_{.025}$	$t_{.010}$	$t_{.005}$	$t_{.001}$
1	3.078	6.314	12.706	31.821	63.656	318.289
2	1.886	2.920	4.303	6.965	9.925	22.328
3	1.638	2.353	3.182	4.541	5.841	10.214
4	1.533	2.132	2.776	3.747	4.604	7.173
5	1.476	2.015	2.571	3.365	4.032	5.894
6	1.440	1.943	2.447	3.143	3.707	5.208
7	1.415	1.895	2.365	2.998	3.499	4.785
8	1.397	1.860	2.306	2.896	3.355	4.501
9	1.383	1.833	2.262	2.821	3.250	4.297
10	1.372	1.812	2.228	2.764	3.169	4.144
11	1.363	1.796	2.201	2.718	3.106	4.025
12	1.356	1.782	2.179	2.681	3.055	3.930
13	1.350	1.771	2.160	2.650	3.012	3.852
14	1.345	1.761	2.145	2.624	2.977	3.787
15	1.341	1.753	2.131	2.602	2.947	3.733
16	1.337	1.746	2.120	2.583	2.921	3.686
17	1.333	1.740	2.110	2.567	2.898	3.646
18	1.330	1.734	2.101	2.552	2.878	3.611
19	1.328	1.729	2.093	2.539	2.861	3.579
20	1.325	1.725	2.086	2.528	2.845	3.552
21	1.323	1.721	2.080	2.518	2.831	3.527
22	1.321	1.717	2.074	2.508	2.819	3.505
23	1.319	1.714	2.069	2.500	2.807	3.485
24	1.318	1.711	2.064	2.492	2.797	3.467
25	1.316	1.708	2.060	2.485	2.787	3.450
26	1.315	1.706	2.056	2.479	2.779	3.435
27	1.314	1.703	2.052	2.473	2.771	3.421
28	1.313	1.701	2.048	2.467	2.763	3.408
29	1.311	1.699	2.045	2.462	2.756	3.396
30	1.310	1.697	2.042	2.457	2.750	3.385
40	1.303	1.684	2.021	2.423	2.704	3.307
50	1.299	1.676	2.009	2.403	2.678	3.261
60	1.296	1.671	2.000	2.390	2.660	3.232
80	1.292	1.664	1.990	2.374	2.639	3.195
100	1.290	1.660	1.984	2.364	2.626	3.174
∞	1.282	1.645	1.960	2.326	2.576	3.091

Source: "Table of Percentage Points of the t-Distribution." Computed by Maxine Merrington, Biometrika, 32 (1941): 300. Reproduced by permission of the Biometrika trustees.

TABLE D: *F* Distribution



$\alpha = .05$										
	df_1									
df_2	1	2	3	4	5	6	8	12	24	∞
1	161.4	199.5	215.7	224.6	230.2	234.0	238.9	243.9	249.0	254.3
2	18.51	19.00	19.16	19.25	19.30	19.33	19.37	19.41	19.45	19.50
3	10.13	9.55	9.28	9.12	9.01	8.94	8.84	8.74	8.64	8.53
4	7.71	6.94	6.59	6.39	6.26	6.16	6.04	5.91	5.77	5.63
5	6.61	5.79	5.41	5.19	5.05	4.95	4.82	4.68	4.53	4.36
6	5.99	5.14	4.76	4.53	4.39	4.28	4.15	4.00	3.84	3.67
7	5.59	4.74	4.35	4.12	3.97	3.87	3.73	3.57	3.41	3.23
8	5.32	4.46	4.07	3.84	3.69	3.58	3.44	3.28	3.12	2.93
9	5.12	4.26	3.86	3.63	3.48	3.37	3.23	3.07	2.90	2.71
10	4.96	4.10	3.71	3.48	3.33	3.22	3.07	2.91	2.74	2.54
11	4.84	3.98	3.59	3.36	3.20	3.09	2.95	2.79	2.61	2.40
12	4.75	3.88	3.49	3.26	3.11	3.00	2.85	2.69	2.50	2.30
13	4.67	3.80	3.41	3.18	3.02	2.92	2.77	2.60	2.42	2.21
14	4.60	3.74	3.34	3.11	2.96	2.85	2.70	2.53	2.35	2.13
15	4.54	3.68	3.29	3.06	2.90	2.79	2.64	2.48	2.29	2.07
16	4.49	3.63	3.24	3.01	2.85	2.74	2.59	2.42	2.24	2.01
17	4.45	3.59	3.20	2.96	2.81	2.70	2.55	2.38	2.19	1.96
18	4.41	3.55	3.16	2.93	2.77	2.66	2.51	2.34	2.15	1.92
19	4.38	3.52	3.13	2.90	2.74	2.63	2.48	2.31	2.11	1.88
20	4.35	3.49	3.10	2.87	2.71	2.60	2.45	2.28	2.08	1.84
21	4.32	3.47	3.07	2.84	2.68	2.57	2.42	2.25	2.05	1.81
22	4.30	3.44	3.05	2.82	2.66	2.55	2.40	2.23	2.03	1.78
23	4.28	3.42	3.03	2.80	2.64	2.53	2.38	2.20	2.00	1.76
24	4.26	3.40	3.01	2.78	2.62	2.51	2.36	2.18	1.98	1.73
25	4.24	3.38	2.99	2.76	2.60	2.49	2.34	2.16	1.96	1.71
26	4.22	3.37	2.98	2.74	2.59	2.47	2.32	2.15	1.95	1.69
27	4.21	3.35	2.96	2.73	2.57	2.46	2.30	2.13	1.93	1.67
28	4.20	3.34	2.95	2.71	2.56	2.44	2.29	2.12	1.91	1.65
29	4.18	3.33	2.93	2.70	2.54	2.43	2.28	2.10	1.90	1.64
30	4.17	3.32	2.92	2.69	2.53	2.42	2.27	2.09	1.89	1.62
40	4.08	3.23	2.84	2.61	2.45	2.34	2.18	2.00	1.79	1.51
60	4.00	3.15	2.76	2.52	2.37	2.25	2.10	1.92	1.70	1.39
120	3.92	3.07	2.68	2.45	2.29	2.17	2.02	1.83	1.61	1.25
∞	3.84	2.99	2.60	2.37	2.21	2.09	1.94	1.75	1.52	1.00

Source: From Table V of R. A. Fisher and F. Yates, *Statistical Tables for Biological, Agricultural and Medical Research*, published by Longman Group Ltd., London, 1974. (Previously published by Oliver & Boyd, Edinburgh.) Reprinted by permission of the authors and publishers.

CRITICAL VALUES OF G FOR REJECTION OF OUTLIER

Number of observations	G (95% Confidence)
4	1.463
5	1.672
6	1.822
7	1.938
8	2.032
9	2.11
10	2.176
11	2.234
12	2.285
15	2.409
20	2.557

1																	18
1 **H** 1.0079	2											13	14	15	16	17	2 **He** 4.0026
3 **Li** 6.941	4 **Be** 9.0122											5 **B** 10.811	6 **C** 12.011	7 **N** 14.007	8 **O** 15.999	9 **F** 18.998	10 **Ne** 20.180
11 **Na** 22.990	12 **Mg** 24.305	3	4	5	6	7	8	9	10	11	12	13 **Al** 26.982	14 **Si** 28.086	15 **P** 30.974	16 **S** 32.065	17 **Cl** 35.453	18 **Ar** 39.948
19 **K** 39.098	20 **Ca** 40.078	21 **Sc** 44.956	22 **Ti** 47.867	23 **V** 50.942	24 **Cr** 51.996	25 **Mn** 54.938	26 **Fe** 55.845	27 **Co** 58.933	28 **Ni** 58.693	29 **Cu** 63.546	30 **Zn** 65.409	31 **Ga** 69.723	32 **Ge** 72.64	33 **As** 74.922	34 **Se** 78.96	35 **Br** 79.904	36 **Kr** 83.798
37 **Rb** 85.468	38 **Sr** 87.62	39 **Y** 88.906	40 **Zr** 91.224	41 **Nb** 92.906	42 **Mo** 95.94	43 **Tc** (98)	44 **Ru** 101.07	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** 121.76	52 **Te** 127.60	53 **I** 126.90	54 **Xe** 131.29
55 **Cs** 132.91	56 **Ba** 137.33	57-71 *	72 **Hf** 178.49	73 **Ta** 180.95	74 **W** 183.84	75 **Re** 186.21	76 **Os** 190.23	77 **Ir** 192.22	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)	89-103 #	104 **Rf** (261)	105 **Db** (262)	106 **Sg** (266)	107 **Bh** (264)	108 **Hs** (270)	109 **Mt** (268)	110 **Ds** (281)	111 **Rg** (272)	112 **Uub** (285)	113 **Uut** (284)	114 **Uuq** (289)	115 **Uup** (288)	116 **Uuh** (291)		118 **Uuo** (294)

* Lanthanide series	57 **La** 138.91	58 **Ce** 140.12	59 **Pr** 140.91	60 **Nd** 144.24	61 **Pm** (145)	62 **Sm** 150.36	63 **Eu** 151.96	64 **Gd** 157.25	65 **Tb** 158.93	66 **Dy** 162.50	67 **Ho** 164.93	68 **Er** 167.26	69 **Tm** 168.93	70 **Yb** 173.04	71 **Lu** 174.97
# Actinide series	89 **Ac** (227)	90 **Th** 232.04	91 **Pa** 231.04	92 **U** 238.03	93 **Np** (237)	94 **Pu** (244)	95 **Am** (243)	96 **Cm** (247)	97 **Bk** (247)	98 **Cf** (251)	99 **Es** (252)	100 **Fm** (257)	101 **Md** (258)	102 **No** (259)	103 **Lr** (262)