LAIKIPIA UNIVERSITY

UNIVERSITY EXAMINATIONS

SECOND SEMESTER 2023/2024 ACADEMIC YEAR

THIRD YEAR EXAMINATION FOR THE DEGREES OF BACHELOR OF SCIENCE (GENERAL) & BACHELOR OF EDUCATION (SCIENCE)

CHEM 323: ANALYTICAL CHEMISTRY I

STREAM: R ***TIME: 2 HRS***

DAY: FRIDAY[8.30AM-10.30AM] ***DATE: 12/04/2024***

THIS QUESTION PAPER CONSISTS OF SEVEN (7) PAGES

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INSTRUCTIONS: Attempt **All** Questions

QUESTION ONE (30 MARKS)

a) Briefly explaining the following sampling methods and give an example to illustrate its application **(3 Marks)**

i) grab sampling

ii) In situ sampling

b) Define the following terms **(3 Marks)**

i) Precision

ii) Accuracy

c) Differentiate between determinate and indeterminate errors stating their sources and how to minimize them **(4 Marks)**

d)) i) Differentiate sample and population **(2 Marks)**

ii) What is the 95% confidence interval for the amount of aspirin in a single analgesic tablet drawn from a population for which μ is 250 mg and σ^2 is 25? **(2 Marks)**

iii) When determining the amount of Na_2CO_3 in a sample, the procedure is checked by analyzing a sample known to be 98.76% w/w Na_2CO_3. Five replicate determinations of the %w/w Na_2CO_3 in the standard gave the following results: a mean of 98.59 and a standard deviation of 0.0973 Using $\alpha = 0.05$, is there any evidence that the analysis is giving inaccurate results? **(3 Marks)**

e) i) The amount of lead poisoning from a lab follows normal distribution with $\mu = 250\ mg$ and $\sigma^2 = 25$. In a random sampling, what percentage of patients is expected to have between 243 mg and 262 mg of lead poisoning? **(3 Marks)**

ii) Two production lots of aspirin tablets were sampled and analysed for the amount of aspirin and the following results in mg aspirin/tablet were obtained

LOT 1: 256, 248, 245, 245, 244, 248, 261 giving a standard deviation of 6.451 mg

LOT 2: 241, 258, 241, 244, 256, 254 giving a standard deviation of 7.849 mg

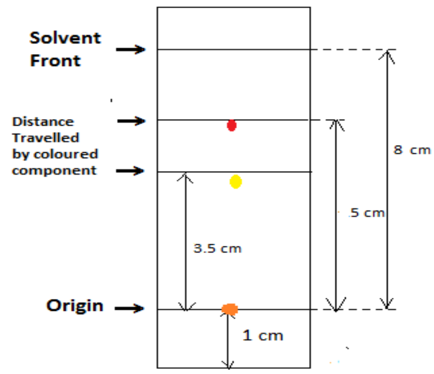
Is there any evidence at $\alpha = 0.05$ that there is a significant difference in the variance between the results of the two samples **(3 Marks)**

f) i) State the principle behind the working of an ion selective electrode **(1 Mark)**

ii) A glass electrode is a specific example of an ion selective electrode. How does a glass electrode work? **(3 Marks)**

g) i) Define the term retention in chromatography **(1 Mark)**

ii) Calculate the retention factors from the chromatogram for the yellow and red solutes, respectively. **(2 Marks)**



QUESTION TWO (20 MARKS)

a) i) what is Chromatography? **(1 Mark)**

ii) Name and describe the two types of columns used in gas Chromatography **(4 Marks)**

iii) Define the following detection properties: bulk properties and specific properties **(2 Marks)**

b) Define the terms **(3 Marks)**

i) Stationary phase
ii) Mobile phase
iii) Elution

c)A GC separation was conducted on a sample containing a pesticide analyte, compound X. This sample was treated with an internal standard of Q, giving a concentration of 15.0 ppm. A 1.0 µL i njection onto the GC gave an FID response of 1012 for Q and 3411 for X. A 1.0 µL standard solu tion of 30.0 ppm Q with 15.0 ppm of X was injected giving a response of 899 and 2791 respecti vely. What is the concentration of Q in the sample? **(3 Marks)**

d) i) What would be the relative advantages and disadvantages of using FT- IR as a HPLC detector? Comment on the universality (or lack of) of the detector **(3 Marks)**

ii) Describe some advantages and disadvantages of the electron capture detector (ECD) as a GC detection system. **(2 Marks)**

e) What are the physical principles behind Normal Phase and Reverse Phase chromatography? **(2 Marks)**

QUESTION THREE (20 MARKS)

a) Explain the principle behind

i) Atomic Absorption Spectroscopy (AAS) **(2 Marks)**

ii) Atomic EmissionSpectroscopy (AES) **(2 Marks)**

b) i) Give four potential sources of error in sample preparation prior to atomic absorption analysis. **(2 Marks)**

ii) State the light source for AAS; explain how it works, including why a different lamp must be used for each element. **(2 Marks)**

iii) The drug tolbutamine (MW = 270.0 g mol-1), a treatment for type 2 diabetes, has a molar absorptivity of 703 $M^{-1}cm^{-1}$ at 262 nm. One tablet was dissolved in 250.00 mL of water. A 10.00 mL aliquot of this solution was diluted to 100.00 mL in a volumetric flask. This diluted solution exhibited an absorbance of 0.275 at 262 nm in a 1.00-cm cell. Calculate the mass (**in mg**) of tolbutamine in the tablet. **(4 Marks)**

c) Using a diagram, explain the instrumental components (and their arrangement) of an atomic emission spectrometer. **(4 Marks)**

d) i) What is meant by spectral interference? **(1 Mark)**

ii) Explain how the following background correction methods are used to correct spectral interferences

I) Use of a continuum source such as a Deuterium (D_2) lamp **(1.5 Marks)**

II) Zeeman correcion **(1.5 Marks)**

Appendices

Single-Sided Normal Distribution

z	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
0.0	0.5000	0.4960	0.4920	0.4880	0.4840	0.4801	0.4761	0.4721	0.4681	0.4641
0.1	0.4602	0.4562	0.4522	0.4483	0.4443	0.4404	0.4365	0.4325	0.4286	0.4247
0.2	0.4207	0.4168	0.4129	0.4090	0.4502	0.4013	0.3974	0.3396	0.3897	0.3859
0.3	0.3821	0.3783	0.3745	0.3707	0.3669	0.3632	0.3594	0.3557	0.3520	0.3483
0.4	0.3446	0.3409	0.3372	0.3336	0.3300	0.3264	0.3228	0.3192	0.3156	0.3121
0.5	0.3085	0.3050	0.3015	0.2981	0.2946	0.2912	0.2877	0.2843	0.2810	0.2776
0.6	0.2743	0.2709	0.2676	0.2643	0.2611	0.2578	0.2546	0.2514	0.2483	0.2451
0.7	0.2420	0.2389	0.2358	0.2327	0.2296	0.2266	0.2236	0.2206	0.2177	0.2148
0.8	0.2119	0.2090	0.2061	0.2033	0.2005	0.1977	0.1949	0.1922	0.1894	0.1867
0.9	0.1841	0.1814	0.1788	0.1762	0.1736	0.1711	0.1685	0.1660	0.1635	0.1611
1.0	0.1587	0.1562	0.1539	0.1515	0.1492	0.1469	0.1446	0.1423	0.1401	0.1379
1.1	0.1357	0.1335	0.1314	0.1292	0.1271	0.1251	0.1230	0.1210	0.1190	0.1170
1.2	0.1151	0.1131	0.1112	0.1093	0.1075	0.1056	0.1038	0.1020	0.1003	0.0985
1.3	0.0968	0.0951	0.0934	0.0918	0.0901	0.0885	0.0869	0.0853	0.0838	0.0823
1.4	0.0808	0.0793	0.0778	0.0764	0.0749	0.0735	0.0721	0.0708	0.0694	0.0681
1.5	0.0668	0.0655	0.0643	0.0630	0.0618	0.0606	0.0594	0.0582	0.0571	0.0559
1.6	0.0548	0.0537	0.0526	0.0516	0.0505	0.0495	0.0485	0.0475	0.0465	0.0455
1.7	0.0466	0.0436	0.0427	0.0418	0.0409	0.0401	0.0392	0.0384	0.0375	0.0367
1.8	0.0359	0.0351	0.0344	0.0336	0.0329	0.0322	0.0314	0.0307	0.0301	0.0294
1.9	0.0287	0.0281	0.0274	0.0268	0.0262	0.0256	0.0250	0.0244	0.0239	0.0233
2.0	0.0228	0.0222	0.0217	0.0212	0.0207	0.0202	0.0197	0.0192	0.0188	0.0183
2.1	0.0179	0.0174	0.0170	0.0166	0.0162	0.0158	0.0154	0.0150	0.0146	0.0143
2.2	0.0139	0.0136	0.0132	0.0129	0.0125	0.0122	0.0119	0.0116	0.0113	0.0110
2.3	0.0107	0.0104	0.0102		0.00964		0.00914		0.00866	
2.4	0.00820		0.00776		0.00734		0.00695		0.00657	
2.5	0.00621		0.00587		0.00554		0.00523		0.00494	
2.6	0.00466		0.00440		0.00415		0.00391		0.00368	
2.7	0.00347		0.00326		0.00307		0.00289		0.00272	
2.8	0.00256		0.00240		0.00226		0.00212		0.00199	
2.9	0.00187		0.00175		0.00164		0.00154		0.00144	
3.0	0.00135									
3.1	0.000968									
3.2	0.000687									
3.3	0.000483									
3.4	0.000337									
3.5	0.000233									
3.6	0.000159									
3.7	0.000108									
3.8	0.0000723									
3.9	0.0000481									
4.0	0.0000317									

Critical Values for t-Test

Values of *t* for…				
…a confidence interval of:	**90%**	**95%**	**98%**	**99%**
…an α value of:	**0.10**	**0.05**	**0.02**	**0.01**
Degrees of Freedom				
1	6.314	12.706	31.821	63.657
2	2.920	4.303	6.965	9.925
3	2.353	3.182	4.541	5.841
4	2.132	2.776	3.747	4.604
5	2.015	2.571	3.365	4.032
6	1.943	2.447	3.143	3.707
7	1.895	2.365	2.998	3.499
8	1.860	2.306	2.896	3.255
9	1.833	2.262	2.821	3.250
10	1.812	2.228	2.764	3.169
12	1.782	2.179	2.681	3.055
14	1.761	2.145	2.624	2.977
16	1.746	2.120	2.583	2.921
18	1.734	2.101	2.552	2.878
20	1.725	2.086	2.528	2.845
30	1.697	2.042	2.457	2.750
50	1.676	2.009	2.311	2.678
∞	1.645	1.960	2.326	2.576

Critical Values for F-Test

F(0.05,vnum, vdenom) for a One-Tailed F-Test													
ν_{num}⇒ ⇓ν_{denom}	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**15**	**20**	∞
1	161.4	199.5	215.7	224.6	230.2	234.0	236.8	238.9	240.5	241.9	245.9	248.0	254.3
2	18.51	19.00	19.16	19.25	19.30	19.33	19.35	19.37	19.38	19.40	19.43	19.45	19.50
3	10.13	9.552	9.277	9.117	9.013	8.941	8.887	8.845	8.812	8.786	8.703	8.660	8.526
4	7.709	6.994	6.591	6.388	6.256	6.163	6.094	6.041	5.999	5.964	5.858	5.803	5.628
5	6.608	5.786	5.409	5.192	5.050	4.950	4.876	4.818	4.722	4.753	4.619	4.558	4.365
6	5.591	5.143	4.757	4.534	4.387	4.284	4.207	4.147	4.099	4.060	3.938	3.874	3.669
7	5.591	4.737	4.347	4.120	3.972	3.866	3.787	3.726	3.677	3.637	3.511	3.445	3.230
8	5.318	4.459	4.066	3.838	3.687	3.581	3.500	3.438	3.388	3.347	3.218	3.150	2.928
9	5.117	4.256	3.863	3.633	3.482	3.374	3.293	3.230	3.179	3.137	3.006	2.936	2.707
10	4.965	4.103	3.708	3.478	3.326	3.217	3.135	3.072	3.020	2.978	2.845	2.774	2.538
11	4.844	3.982	3.587	3.257	3.204	3.095	3.012	2.948	2.896	2.854	2.719	2.646	2.404
12	4.747	3.885	3.490	3.259	3.106	2.996	2.913	2.849	2.796	2.753	2.617	2.544	2.296
13	4.667	3.806	3.411	3.179	3.025	2.915	2.832	2.767	2.714	2.671	2.533	2.459	2.206
14	4.600	3.739	3.344	3.112	2.958	2.848	2.764	2.699	2.646	2.602	2.463	2.388	2.131
15	4.534	3.682	3.287	3.056	2.901	2.790	2.707	2.641	2.588	2.544	2.403	2.328	2.066
16	4.494	3.634	3.239	3.007	2.852	2.741	2.657	2.591	2.538	2.494	2.352	2.276	2.010
17	4.451	3.592	3.197	2.965	2.810	2.699	2.614	2.548	2.494	2.450	2.308	2.230	1.960
18	4.414	3.555	3.160	2.928	2.773	2.661	2.577	2.510	2.456	2.412	2.269	2.191	1.917
19	4.381	3.552	3.127	2.895	2.740	2.628	2.544	2.477	2.423	2.378	2.234	2.155	1.878
20	4,351	3.493	3.098	2.866	2.711	2.599	2.514	2.447	2.393	2.348	2.203	2.124	1.843
∞	3.842	2.996	2.605	2.372	2.214	2.099	2.010	1.938	1.880	1.831	1.666	1.570	1.000