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University Examinations 2024/2025

FIRST YEAR FIRST SEMESTER EXAMINATION FOR THE DEGREE MASTER OF
SCIENCE IN CHEMISTRY

SCH 7110: ADVANCED ANALYTICAL CHEMISTRY

DATE: JANUARY 2025

TIME: 3 HOURS

INSTRUCTIONS: Answer question *one* and any other *two* questions

QUESTION ONE (30 MARKS)

a) Define the following terms. (5 marks)

- (i) Precision
- (ii) Accuracy
- (iii) Bias
- (iv) Random sample
- (v) Optimization

(b) The response of a colorimetric test for glucose was checked with the aid of standard glucose solutions.

Glucose concentration, mM	0	2	4	6	8	10
Absorbance	0.002	0.150	0.294	0.434	0.570	0.704

Determine:

- (i) Correlation coefficient (4 marks)
- (ii) Coefficient of determination. Explain. (2 marks)
- (iii) Slope (1 mark)
- (iv) Intercept (1 mark)

(v) Linear regression equation (1 mark)

(c)(i) Illustrate the features of a Shewhart chart. (3 marks)

(ii) State two main commonly applied criteria in the interpretation of Shewhart chart. (2 marks)

(d) Distinguish between proficiency testing and collaborative trials. (4 marks)

(e) The following figures refer to the concentration of albumin, in gL^{-1} in the blood sera of 8 males and 8 female healthy adults:

Male	37	39	37	42	39	45	42	39
Female	44	40	39	45	47	47	43	41

Test whether the mean concentrations for men and women differ significantly. (5 marks).

(f) The optimum pH for an enzyme-catalysed reaction is known to lie between 5 and 9. Determine the pH values at which the first two experiments of an optimization process should be performed to give a maximum range of 0.1 pH units. (2 marks)

QUESTION TWO (15 MARKS)

a) A standard sample of pooled human blood serum contains 42.0 g of albumin per litre. Three laboratories (A, B and C) did six determinations each (on the same day) of the albumin concentration, with the following results (gL^{-1}).

Laboratory	Results					
A	42.5	41.6	42.1	41.9	41.1	42.2
B	43.5	42.8	43.8	43.1	42.7	43.3
C	42.2	41.6	42.0	41.8	42.6	39.0

Comment on the bias, precision and accuracy of each of these sets of results. (6 marks)

b) The number of glassware breakages by three different laboratory workers over a given period are stated below:

Worker	Breakages
A	14
B	19
C	12

Test whether the number of breakages by the three worker differs significantly. (6 marks)

- c) Identify three ways of tackling systematic errors. (3 marks)

QUESTION THREE (15 MARKS)

- a) Four students (A- D) each performed an analysis in which exactly 10.0 mL of exactly 0.1 M sodium hydroxide was titrated with exactly 0.1 M hydrochloric acid. Each student performed five replicate titrations, with the results shown in the table below.

Student	Results (mL)				
A	10.0	10.1	10.0	10.1	10.2
B	9.9	10.1	10.0	9.8	10.2
C	10.2	9.8	9.7	10.0	9.8
D	10.0	9.9	10.0	9.9	10.0

- (i) Test whether there is any significant difference between the means obtained by the four students. (12 marks)
- (ii) Calculate the 95% confidence limits of the mean for student C and use them to decide whether there is any evidence of systematic error. (3 marks)

QUESTION FOUR (15 MARKS)

- (a) What are the stages of experimental design? (3 marks)
- (b) Mercury is lost from solutions stored in polypropylene flasks by combination with traces of tin in the polymer. The absorbance of a standard aqueous solution of mercury stored in such flasks was measured for two levels of the following factors:

Factor	Low	High
A - Agitation of flask	-	+
C - Cleaning of flask	-	+
T - Time of standing	-	+

The following results were obtained.

Combination of factor levels	Absorbance
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l	0.099
a	0.084
c	0.097
t	0.076
ac	0.082
ta	0.049
tc	0.080
atc	0.051

Calculate interaction effects between:

- (i) AT (9 marks)
(ii) ACT (3 marks)

Table 1: The *t*-distribution

Value of <i>t</i> for a confidence interval of Critical value of $ t $ for <i>P</i> values of number of degrees of freedom	90% 0.10	95% 0.05	98% 0.02	99% 0.01
1	6.31	12.71	31.82	63.66
2	2.92	4.30	6.96	9.92
3	2.35	3.18	4.54	5.84
4	2.13	2.78	3.75	4.60
5	2.02	2.57	3.36	4.03
6	1.94	2.45	3.14	3.71
7	1.89	2.36	3.00	3.50
8	1.86	2.31	2.90	3.36
9	1.83	2.26	2.82	3.25
10	1.81	2.23	2.76	3.17
12	1.78	2.18	2.68	3.05
14	1.76	2.14	2.62	2.98
16	1.75	2.12	2.58	2.92
18	1.73	2.10	2.55	2.88
20	1.72	2.09	2.53	2.85
30	1.70	2.04	2.46	2.75
50	1.68	2.01	2.40	2.68
∞	1.64	1.96	2.33	2.58

The critical values of $|t|$ are appropriate for a two-tailed test. For a one-tailed test the value is taken from the column for twice the desired *P*-value, e.g. for a one-tailed test, $P = 0.05$, 5 degrees of freedom, the critical value is read from the $P = 0.10$ column and is equal to 2.02.

Table 2: Critical values of χ^2 ($P = 0.05$)

Number of degrees of freedom	Critical value
1	3.84
2	5.99
3	7.81
4	9.49
5	11.07
6	12.59
7	14.07
8	15.51
9	16.92
10	18.31

Table 3: Critical values of F for a one-tailed test ($P = 0.05$)

v_2	v_1													
	1	2	3	4	5	6	7	8	9	10	12	15	20	
1	161.4	199.5	215.7	224.6	230.2	234.0	236.8	238.9	240.5	241.9	243.9	245.9	248.0	
2	18.51	19.00	19.16	19.25	19.30	19.33	19.35	19.37	19.38	19.40	19.41	19.43	19.45	
3	10.13	9.552	9.277	9.117	9.013	8.941	8.887	8.845	8.812	8.786	8.745	8.703	8.660	
4	7.709	6.944	6.591	6.388	6.256	6.163	6.094	6.041	5.999	5.964	5.912	5.858	5.803	
5	6.608	5.786	5.409	5.192	5.050	4.950	4.876	4.818	4.772	4.735	4.678	4.619	4.568	
6	5.987	5.143	4.757	4.534	4.387	4.284	4.207	4.147	4.099	4.060	4.000	3.938	3.874	
7	5.591	4.737	4.347	4.120	3.972	3.866	3.787	3.726	3.677	3.637	3.575	3.511	3.445	
8	5.318	4.459	4.066	3.838	3.687	3.581	3.500	3.438	3.388	3.347	3.284	3.218	3.150	
9	5.117	4.256	3.863	3.633	3.482	3.374	3.293	3.230	3.179	3.137	3.073	3.006	2.936	
10	4.965	4.103	3.708	3.478	3.326	3.217	3.135	3.072	3.020	2.978	2.913	2.845	2.774	
11	4.844	3.982	3.587	3.357	3.204	3.095	3.012	2.948	2.896	2.854	2.788	2.719	2.646	
12	4.747	3.885	3.490	3.259	3.106	2.996	2.913	2.849	2.796	2.753	2.687	2.617	2.544	
13	4.667	3.806	3.411	3.179	3.025	2.915	2.832	2.767	2.714	2.671	2.604	2.533	2.459	
14	4.600	3.739	3.344	3.112	2.958	2.848	2.764	2.699	2.646	2.602	2.534	2.463	2.388	
15	4.543	3.682	3.287	3.056	2.901	2.790	2.707	2.641	2.588	2.544	2.475	2.403	2.328	
16	4.494	3.634	3.239	3.007	2.852	2.741	2.657	2.591	2.538	2.494	2.425	2.352	2.276	
17	4.451	3.592	3.197	2.965	2.810	2.699	2.614	2.548	2.494	2.450	2.381	2.308	2.230	
18	4.414	3.555	3.160	2.928	2.773	2.661	2.577	2.510	2.456	2.412	2.342	2.269	2.191	
19	4.381	3.522	3.127	2.895	2.740	2.628	2.544	2.477	2.423	2.378	2.308	2.234	2.155	
20	4.351	3.493	3.098	2.866	2.711	2.599	2.514	2.447	2.393	2.348	2.278	2.203	2.124	

v_1 = number of degrees of freedom of the numerator; v_2 = number of degrees of freedom of the denominator.