NATIONAL UNIVERSITY OF LESOTHO

FACULTY OF HEALTH SCIENCES

DEPARTMENT OF PHARMACY

BACHELOR OF PHARMACY (HONOURS)

PHA 3401 – PHARMACEUTICAL ANALYSIS

SUPPLEMENTARY EXAMINATION

AUGUST 2023 TIME: 3 HOURS TOTAL: 100 MARKS

INSTRUCTIONS:

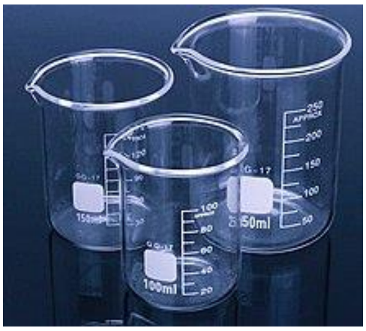
- **THIS PAPER CONSISTS OF 5 QUESTIONS, EACH CARRYING 20 MARKS**
- **ANSWER ALL QUESTIONS**
- **START EACH QUESTION ON A NEW PAGE**
- **MARKS ARE SHOWN IN PARENTHESIS AT THE END OF EACH QUESTION**

Question 1 **[20 marks]**

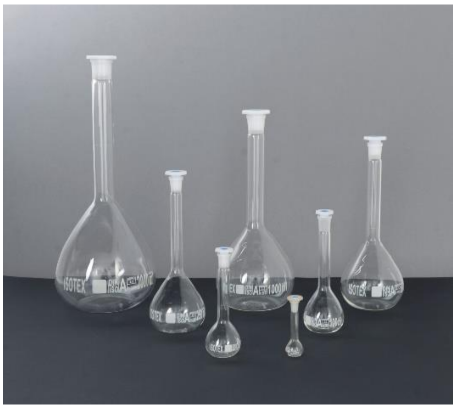
a. Define the following terms [8]

i. Buffer
ii. Analyte
iii. Sampling
iv. Equivalence point
v. Standard deviation
vi. Significance level
vii. Primary standard
viii. Complexiometric titration

b. Identify the following laboratory consumables and give **one** function of each [12]



i.



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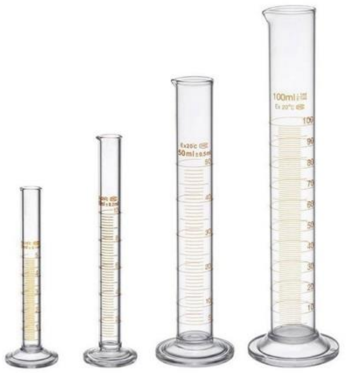

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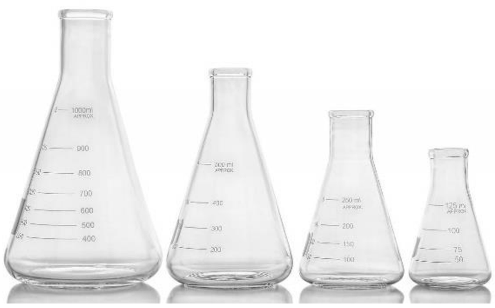

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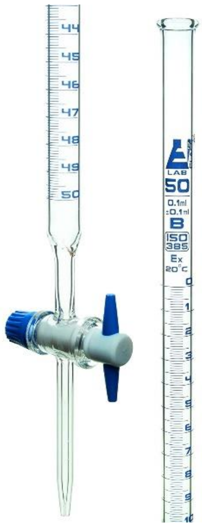

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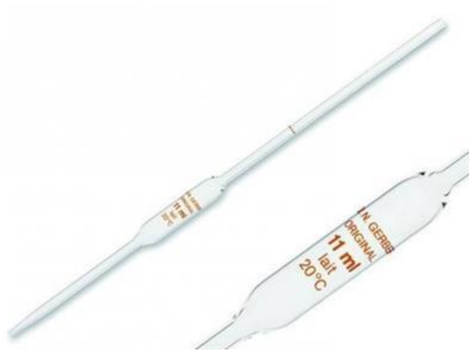

vi.

Question 2 **[20 marks]**

a. A quantitative determination of paracetamol in an oral solution with a specified content of 24 mg/mL, gave the following results when six individual measurements were conducted:

Measurement No	Quantity (mg/ml)
1	21.6
2	23.1
3	23.2
4	23.3
5	23.6
6	23.7

i. If it is necessary, reject the outlying data at 95% confidence level using the Q-test. [4]

ii. Calculate the mean of the paracetamol [2]

iii. Calculate the standard deviation. [2]

iv. Calculate the confidence interval at 95 % confidence level. [2]

b. 200 ml of a 0.25 M sodium borate (NaB) buffer with pH 8.0 was prepared. Then 20 ml of 0.1 M HCl was added to it. Given that boric acid pKa is 9.14, calculate:

i. The pH of the new buffer solution. [8]

ii. Molarity of the new buffer solution. [1]

Question 3 **[20 marks]**

a. A thymol mouth wash is formulated by adding 127.78 mg (0.12778g) of thymol in mixture of ethanol (96%v/v) and methanol, and the solution is dissolved in 20 ml of water. The density of water is 0.997044 g/ml at room temperature 25 °C and the molecular weight of thymol ($C_{10}H_{14}O$) is 150.217 g/mol.

i. Calculate the molarity of the solution. [3]

ii. Calculate the molality of the solution. [3]

iii. Express the concentration of the solution in % w/v [2]

iv. Express the concentration of the solution in ppm. [2]

b. What mass of $AgNO_3$ (169.9 g/mol) is needed to convert 2.33 g of Na_2CO_3 (106.0 g/mol) to Ag_2CO_3? What mass of Ag_2CO_3 (275.7 g/mol) will be formed? [6]

c. Calculate the molar analytical concentration of Na_2CO_3 in the solution produced when 25.0 ml of 0.200 M $AgNO_3$ is mixed with 50.0 ml of 0.0800 M Na_2CO_3? [4]

Question 4 **[20 marks]**

a. Ni^{2+} can be analyzed by indirect titration, using standard Zn^{2+} at pH 5.5 with xylenol orange indicator. A solution containing 50.00 ml of Ni^{2+} in dilute HCl is treated with 50.00 ml of 0.1057 M Na_2EDTA. The solution is neutralized with NaOH, and the pH is adjusted to 5.5 with acetate buffer. The solution turns yellow when a few drops of indicator are added. Titration with 0.04598 M Zn^{2+} requires 35.22 ml to reach the red end point. What is the molarity of Ni^{2+} in the unknown? [4]

b. 50.0 mL of a 0.0200 M metal Mn^{2+} solution is titrated with 0.020M EDTA at pH 9.00. The value of log K for the complex is 14.30 and αY^{4-} is 5.4 x 10^{-2}.

i. Describe any two types of Complexiometric titrations . [4]

ii. Calculate the missing pMn^{2+} in the table below. [4,4,4]

mL	0.00	25.0	49.9	50.0	50.1	55.0
pMn^{2+}	1.70		4.70		10.30	

Question 5 **[20 marks]**

a. A 2.00-g sample of dolomite, a calcium supplement, was dissolved in hydrochloric acid (HCl). To the resulting solution was added excess ammonium oxalate ($(NH_3)_2C_2O_4$) that precipitated calcium as calcium oxalate (CaC_2O_4). The precipitate was filtered then placed together with the filter paper in a 30.1025-g crucible. This was then ignited and weighed several times to a constant weight as 30.7100g CaO.

i. Why was the precipitate ignited? [3]

ii. What is the benefit of converting and using the precipitate as CaO? [1]

iii. Calculate the percentage content of Ca in the supplement. [7]

b. A 50 ml aliquot of 0.05 M NaCN (Ka for HCN = 6.2×10^{-10}) is titrated with 0.100 M HCl at the following acid volumes:

Va (ml)	10.0	25.0	26.0
pH			

i. From the provided data, calculate the pH of the solution at all titration volumes [9]

Appendix

Formulas

$$Molarity = \frac{moles\ of\ solute}{volume\ of\ solution\ in\ litres\ (L)}$$

$$Molality = \frac{moles\ of\ solute}{mass\ of\ solvent\ in\ lkilograms\ (kg)}$$

$$parts\ per\ million = \frac{mass\ of solute}{mass\ of\ sample}\ X\ 10^6$$

$$parts\ per\ billion = \frac{mass\ of solute}{mass\ of\ sample}\ X\ 10^9$$

$$\bar{x} = \frac{x_1+x_2+x_3 \ldots x_n}{\text{n}} = \sum_i \frac{x_i}{n}$$

$$s = \sqrt{\frac{\sum_{i=1}^{i=n}(x_i-\bar{x})^2}{n-1}}$$

$$\boldsymbol{\mu} = \bar{\boldsymbol{x}} \pm \frac{\boldsymbol{t \cdot s}}{\sqrt{\boldsymbol{n}}}$$

$$Q_{calculated} = \frac{|x_i - x_{critical}|}{|x_1 - x_{critical}|}$$

$$Weight\ percent = \%(w/w) = \frac{mass\ of solute}{mass\ of\ solution}\ X\ 100\%$$

$$Volume\ percent = \%(v/v) = \frac{volume\ of solute}{volume\ of\ solution}\ X\ 100\%$$

$$Weight\ volume\ percent = \%(w/v) = \frac{mass\ of solute\ (g)}{volume\ of\ solution\ (ml)}\ X\ 100\%$$

Values of student's t

Degree of freedom (n-1)	Confidence level				
	80%	**90%**	**95%**	**99%**	**99.9%**
1	3.08	6.31	12.7	63.7	637
2	1.89	2.92	4.30	9.92	31.6
3	1.64	2.35	3.18	5.84	12.9
4	1.53	2.13	2.78	4.60	8.61
5	1.48	2.02	2.57	4.03	6.87
6	1.44	1.94	2.45	3.71	5.96
7	1.42	1.90	2.36	3.50	5.41
8	1.40	1.86	2.31	3.36	5.04
9	1.38	1.83	2.26	3.25	4.78
10	1.37	1.81	2.23	3.17	4.59
15	1.34	1.75	2.13	2.95	4.07
20	1.32	1.73	2.09	2.84	3.85
40	1.30	1.68	2.02	2.70	3.55
60	1.30	1.67	2.00	2.62	3.46
∞	1.28	1.64	1.96	2.58	3.29

Q-values for Q-test

Number of measurements	Confidence level		
	90%	**95%**	**99%**
3	0.94	0.98	0.99
4	0.76	0.85	0.93
5	0.64	0.73	0.82
6	0.56	0.64	0.74
7	0.51	0.59	0.68
8	0.47	0.54	0.63
9	0.44	0.51	0.60
10	0.41	0.48	0.57

Values for EDTA

Table 13-1 Values of $\alpha_{Y^{4-}}$ for EDTA at 20°C and μ = 0.10 M

pH	$\alpha_{Y^{4-}}$
0	1.3×10^{-23}
1	1.9×10^{-18}
2	3.3×10^{-14}
3	2.6×10^{-11}
4	3.8×10^{-9}
5	3.7×10^{-7}
6	2.3×10^{-5}
7	5.0×10^{-4}
8	5.6×10^{-3}
9	5.4×10^{-2}
10	0.36
11	0.85
12	0.98
13	1.00
14	1.00

Table 13-2 Formation constants for metal-EDTA complexes

Ion	log K_f	Ion	log K_f	Ion	log K_f
Li^+	2.79	Mn^{3+}	25.3 (25°C)	Ce^{3+}	15.98
Na^+	1.66	Fe^{3+}	25.1	Pr^{3+}	16.40
K^+	0.8	Co^{3+}	41.4 (25°C)	Nd^{3+}	16.61
Be^{2+}	9.2	Zr^{4+}	29.5	Pm^{3+}	17.0
Mg^{2+}	8.79	Hf^{4+}	29.5 (μ = 0.2)	Sm^{3+}	17.14
Ca^{2+}	10.69	VO^{2+}	18.8	Eu^{3+}	17.35
Sr^{2+}	8.73	VO_2^+	15.55	Gd^{3+}	17.37
Ba^{2+}	7.86	Ag^+	7.32	Tb^{3+}	17.93
Ra^{2+}	7.1	Tl^+	6.54	Dy^{3+}	18.30
Sc^{3+}	23.1	Pd^{2+}	18.5 (25°C, μ = 0.2)	Ho^{3+}	18.62
Y^{3+}	18.09			Er^{3+}	18.85
La^{3+}	15.50	Zn^{2+}	16.50	Tm^{3+}	19.32
V^{2+}	12.7	Cd^{2+}	16.46	Yb^{3+}	19.51
Cr^{2+}	13.6	Hg^{2+}	21.7	Lu^{3+}	19.83
Mn^{2+}	13.87	Sn^{2+}	18.3 (μ = 0)	Am^{3+}	17.8 (25°C)
Fe^{2+}	14.32	Pb^{2+}	18.04	Cm^{3+}	18.1 (25°C)
Co^{2+}	16.31	Al^{3+}	16.3	Bk^{3+}	18.5 (25°C)
Ni^{2+}	18.62	Ga^{3+}	20.3	Cf^{3+}	18.7 (25°C)
Cu^{2+}	18.80	In^{3+}	25.0	Th^{4+}	23.2
Ti^{3+}	21.3 (25°C)	Tl^{3+}	37.8 (μ = 1.0)	U^{4+}	25.8
V^{3+}	26.0	Bi^{3+}	27.8	Np^{4+}	24.6 (25°C, μ = 1.0)
Cr^{3+}	23.4				

The Periodic Table of the Elements

1 **H** Hydrogen 1.00794																	2 **He** Helium 4.003
3 **Li** Lithium 6.941	4 **Be** Beryllium 9.012182											5 **B** Boron 10.811	6 **C** Carbon 12.0107	7 **N** Nitrogen 14.00674	8 **O** Oxygen 15.9994	9 **F** Fluorine 18.9984032	10 **Ne** Neon 20.1797
11 **Na** Sodium 22.989770	12 **Mg** Magnesium 24.3050											13 **Al** Aluminum 26.981538	14 **Si** Silicon 28.0855	15 **P** Phosphorus 30.973761	16 **S** Sulfur 32.066	17 **Cl** Chlorine 35.4527	18 **Ar** Argon 39.948
19 **K** Potassium 39.0983	20 **Ca** Calcium 40.078	21 **Sc** Scandium 44.955910	22 **Ti** Titanium 47.867	23 **V** Vanadium 50.9415	24 **Cr** Chromium 51.9961	25 **Mn** Manganese 54.938049	26 **Fe** Iron 55.845	27 **Co** Cobalt 58.933200	28 **Ni** Nickel 58.6934	29 **Cu** Copper 63.546	30 **Zn** Zinc 65.39	31 **Ga** Gallium 69.723	32 **Ge** Germanium 72.61	33 **As** Arsenic 74.92160	34 **Se** Selenium 78.96	35 **Br** Bromine 79.904	36 **Kr** Krypton 83.80
37 **Rb** Rubidium 85.4678	38 **Sr** Strontium 87.62	39 **Y** Yttrium 88.90585	40 **Zr** Zirconium 91.224	41 **Nb** Niobium 92.90638	42 **Mo** Molybdenum 95.94	43 **Tc** Technetium (98)	44 **Ru** Ruthenium 101.07	45 **Rh** Rhodium 102.90550	46 **Pd** Palladium 106.42	47 **Ag** Silver 107.8682	48 **Cd** Cadmium 112.411	49 **In** Indium 114.818	50 **Sn** Tin 118.710	51 **Sb** Antimony 121.760	52 **Te** Tellurium 127.60	53 **I** Iodine 126.90447	54 **Xe** Xenon 131.29
55 **Cs** Cesium 132.90545	56 **Ba** Barium 137.327	57 **La** Lanthanum 138.9055	72 **Hf** Hafnium 178.49	73 **Ta** Tantalum 180.9479	74 **W** Tungsten 183.84	75 **Re** Rhenium 186.207	76 **Os** Osmium 190.23	77 **Ir** Iridium 192.217	78 **Pt** Platinum 195.078	79 **Au** Gold 196.96655	80 **Hg** Mercury 200.59	81 **Tl** Thallium 204.3833	82 **Pb** Lead 207.2	83 **Bi** Bismuth 208.98038	84 **Po** Polonium (209)	85 **At** Astatine (210)	86 **Rn** Radon (222)
87 **Fr** Francium (223)	88 **Ra** Radium (226)	89 **Ac** Actinium (227)	104 **Rf** Rutherfordium (261)	105 **Db** Dubnium (262)	106 **Sg** Seaborgium (263)	107 **Bh** Bohrium (262)	108 **Hs** Hassium (265)	109 **Mt** Meitnerium (266)	110 (269)	111 (272)	112 (277)	113	114				