UNIVERSITI SAINS MALAYSIA

First Semester Examination
Academic Year 2004/2005

October 2004

KAA 501 – Quality Control in Chemistry

Time : 3 hours

Please make sure this paper consists of TEN printed pages before answering the questions.

SECTION A
Questions 1, 2 and 3 are COMPULSORY.

SECTION B
Choose TWO questions from questions No. 4 - 7.

Answer FIVE questions. Only the first five questions answered by the candidate will be marked.

SECTION A

1. (a) Five analysts each made six determinations of the mineral content of the same batch of tablets. The preliminary results are shown in the following table.

Analyst	Mineral Content (%)					
Ahmad	84.50	83.88	84.49	83.91	84.11	84.06
Tan	84.70	84.17	84.11	84.36	84.61	83.81
Suresh	84.31	84.52	84.52	84.63	84.61	84.63
John	84.28	84.38	84.38	84.29	84.39	84.69
Hayati	84.48	84.27	84.33	84.02	84.22	84.50

Based on the above data, calculate

(i) within-sample estimate of σ_o^2 ,

(ii) the sum of the squared terms within-sample,

(iii) between-sample mean variance,

(iv) between-sample mean square,

(16 marks)

(b) Categorize the type of the following samples with an appropriate reason.

(i) Chocolate bars kept in a 24-Hour minimarket along Jalan Sungai Dua.

(ii) Sea water near Penang Ferry Terminal.

(4 marks)

2. (a) Briefly differentiate the following terms normally addressed in a validation process of an analytical method:

(i) Specificity and selectivity

(ii) Method detection limit(MDL) and limit of detection (LOD)

(6 marks)

(b) Calculate the expected coefficient of variation if the concentration of analyte that you are trying to determine is at 1 part per billion (1 ppb) level.

(4 marks)

(c) A robustness or ruggedness test was done on a HPLC method for the identification and assay of an active main compound (MC) and for the detection of two related compounds (RC1 and RC2) in drug tablets. This study was carried out using Plackett Burmann Experimental Design for 11 factors (N=12) that includes 8 factors and 3 dummy factors. The results of the study is given in the following table.

Factors	Effects on						
	%MC	%RC1	%RC2	Rs(MC-RC1)	k'(MC)	Asf(MC)	tR(RC2)
pH	0.683	0.850	0.000	0.427	-0.547	0.204	0.039
Column	-0.450	-0.083	-0.300	1.011	1.269	-0.432	2.978
Dum1	-0.683	-0.917	-0.500	-0.154	-0.047	-0.065	-0.039
Temp	-0.717	-1.150	-0.367	0.408	-0.008	-0.103	-0.333
%B begin	-1.117	-0.617	-1.067	-0.226	-0.869	-0.147	-0.539
%B end	0.883	1.450	0.467	-0.584	-0.347	-0.013	-1.150
Dum2	-0.750	-1.150	-0.167	-0.198	-0.030	-0.003	-0.122
Flow	-0.017	-0.883	-0.300	0.031	-0.592	-0.146	-0.939
Wavelength	0.517	0.650	-0.533	0.041	0.047	0.067	0.084
Buffer conc.	-0.617	0.717	1.100	0.380	-0.019	0.029	0.022
Dum3	-0.250	-0.350	-2.500	0.106	0.036	-0.011	0.144

(i) Identify the factors that have significant effect on the determination of related compounds 2 (%RC2) by using Dong's algorithm at $\alpha = 0.05$ and $\alpha = 0.10$.

(ii) Produce a half normal plot for the case of %RC1 together with margin of error (ME) and simultaneous margin error (SME). Identify any known significant effects.

(10 marks)

3. Briefly discuss the following:

(a) The importance of good record keeping in laboratories in the context of intellectual property management of a research organisation.

(5 marks)

(b) The use of quality control charts as an early indicator in the monitoring of a quality system.

(5 marks)

(c) An accreditated laboratory constitutes as one of the components in the success of an organisation with Hazard Analysis Critical Control Point (HACCP) accreditation.

(5 marks)

(d) Apart from patent registration, intellectual property protection can also be carried out via other mechanisms.

(5 marks)

SECTION B

4. (a) How do you define the acceptance quality level (AQL) within the context of sampling?

(4 marks)

(b) The following is the result obtained from the validation of nutrition carried out on the cereal products. The inspection on the number of units deficient in vitamins B_{12}, C and protein have been performed on four brands (**A, B, C** and **D**).

Brand	Total Units	Lack of Vitamin B_{12}	Lack of Vitamins B_{12} and C	Lack of Vitamins B_{12}, C and Protein
A	25000	6	1	2
B	10100	2	12	3
C	5005	10	1	3
D	3800	4	2	2

(i) What are the minimum and maximum numbers of samples required for the validation on each of these brands (**A, B, C** and **D**)?

(4 marks)

(ii) What are the nonconformities per 100 units for each brand?

(4 marks)

(iii) What are the AQL per 10000 units for each of products **B** and **D**?

(4 marks)

(c) Briefly describe the calibration method for the following equipment:

(i) pH meter

(ii) Furnace

(4 marks)

5. A laboratory was measuring the content of Cu^{2+} ion in samples weekly using atomic spectroscopy technique. The following table gives the mean values of a calibration data using a known standard of 10.00 ppm Cu^{2+} . Two standard samples were run each time.

week	Concentration, mg/L
1	10.09
2	9.97
3	9.99
4	10.08
5	9.08
6	10.08
7	9.99
8	9.98
9	9.98
10	9.99
11	10.08
12	9.99
13	9.98
14	10.07
15	9.98
16	9.98
17	10.09

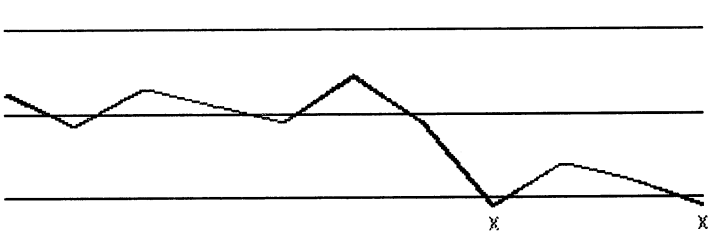
(i) Plot a histogram using the method of grouped data. Comment on the normality of the data.

(ii) Plot x-bar control chart and comment whether the Cu^{2+} ion determination process is under control or not.

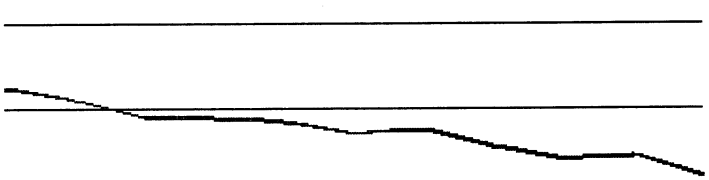
(20 marks)

6. (a) You are the quality control manager of an analytical laboratory doing regular analysis of metal ion contents in biological samples. As a way of implementing quality control, your laboratory developed monthly control charts of the analytical process used. Analyze the pattern of x-bar control charts, describe the type, and the possible causes of the observed pattern. Suggest possible corrective actions that you should take to stabilize the process.

i)



ii)



(10 marks)

(b) An analytical laboratory has regular breakdowns in atomic absorption (AA) spectrophotometer . Describe the Ishikawa or fish-bone diagram approach. Provide an example of this fish-bone diagram in order to find the root cause of this problem.

(10 marks)

7. Write a short essay to describe the internal processes and procedures that are required by a quality manager who is responsible to obtain the Hazard Analysis Critical Control Point (HACCP) certification for a prawn packaging factory. Your discussion must include the purpose, the scope of the certification and the critical steps for the success of the application. Please also include the proposed Gantt Chart of the project management aspects for HACCP Accreditation.

(20 marks)

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APPENDIX

$$M_j = \frac{0.6745(X_i - \tilde{X})}{MAD}$$

$$z_i = \frac{(x_i - \bar{x})}{s}$$

Where

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

$$s_{ai} = \sqrt{\frac{n\sum (x_i - \bar{x})^2(1-u_i^2)^4}{\left|(1-u_i^2)(1-5u_i^2)\right|}}$$

Where

$$u = \frac{x_i - \bar{x}}{9\ MAD}$$

$$R_{i+1} = \frac{\left|x^{(i)} - \bar{x}^{(i)}\right|}{s(i)}$$

$$Qexp = Xq - Xn / w$$

$$t = \frac{|E_X|}{(SE)_e}$$

E_X = $t_{critical}$ multiply (SE)e

$$(SE)_e \sqrt{\frac{s^2}{N/2} + \frac{s^2}{N/2}} = \sqrt{\frac{4S^2}{N}}$$

$$\sqrt{(\sum E^2{}_{error} / n_{error})}$$

$$s_o = 1.5\,\text{median}\,|E_i|$$

$$S_1 \sqrt{m^{-1}\sum E_i^2}$$

$$M\,E = t_{(1-\alpha/2,\,df)}\,S_1$$

FACTORS FOR COMPUTING LIMITS FOR X BAR AND R CHARTS

Sample Size	For Averages	For Ranges		Standard Deviation
n	**A_2**	**D_3**	**D_4**	**d_2**
2	1.88	0	3.29	1.13
3	1.02	0	2.58	1.69
4	.73	0	2.28	2.06
5	.58	0	2.11	2.33
6	.48	0	2.00	2.53
7	.42	0	1.92	2.70
8	.37	.14	1.87	2.85
9	.34	.18	1.82	2.97
10	.31	.31	1.78	3.08

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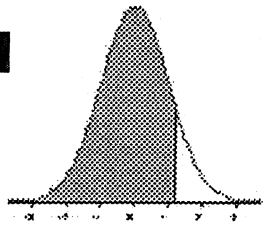
Student's t-distribution table



	p										
df	0.75	0.80	0.85	0.90	0.95	0.975	0.980	0.990	0.995	0.9975	0.9990
1	1.0000	1.3764	1.9626	3.0777	6.3137	12.706	15.895	31.821	63.656	127.32	318.29
2	0.8165	1.0607	1.3862	1.8856	2.9200	4.3027	4.8487	6.9645	9.9250	14.089	22.329
3	0.7649	0.9785	1.2498	1.6377	2.3534	3.1824	3.4819	4.5407	5.8408	7.4532	10.214
4	0.7407	0.9410	1.1896	1.5332	2.1318	2.7765	2.9985	3.7469	4.6041	5.5975	7.1729
5	0.7267	0.9195	1.1558	1.4759	2.0150	2.5706	2.7565	3.3649	4.0321	4.7733	5.8935
6	0.7176	0.9057	1.1342	1.4398	1.9432	2.4469	2.6122	3.1427	3.7074	4.3168	5.2075
7	0.7111	0.8960	1.1192	1.4149	1.8946	2.3646	2.5168	2.9979	3.4995	4.0294	4.7853
8	0.7064	0.8889	1.1081	1.3968	1.8595	2.3060	2.4490	2.8965	3.3554	3.8325	4.5008
9	0.7027	0.8834	1.0997	1.3830	1.8331	2.2622	2.3984	2.8214	3.2498	3.6896	4.2969
10	0.6998	0.8791	1.0931	1.3722	1.8125	2.2281	2.3593	2.7638	3.1693	3.5814	4.1437
11	0.6974	0.8755	1.0877	1.3634	1.7959	2.2010	2.3281	2.7181	3.1058	3.4966	4.0248
12	0.6955	0.8726	1.0832	1.3562	1.7823	2.1788	2.3027	2.6810	3.0545	3.4284	3.9296
13	0.6938	0.8702	1.0795	1.3502	1.7709	2.1604	2.2816	2.6503	3.0123	3.3725	3.8520
14	0.6924	0.8681	1.0763	1.3450	1.7613	2.1448	2.2638	2.6245	2.9768	3.3257	3.7874
15	0.6912	0.8662	1.0735	1.3406	1.7531	2.1315	2.2485	2.6025	2.9467	3.2860	3.7329
16	0.6901	0.8647	1.0711	1.3368	1.7459	2.1199	2.2354	2.5835	2.9208	3.2520	3.6861
17	0.6892	0.8633	1.0690	1.3334	1.7396	2.1098	2.2238	2.5669	2.8982	3.2224	3.6458
18	0.6884	0.8620	1.0672	1.3304	1.7341	2.1009	2.2137	2.5524	2.8784	3.1966	3.6105
19	0.6876	0.8610	1.0655	1.3277	1.7291	2.0930	2.2047	2.5395	2.8609	3.1737	3.5793
20	0.6870	0.8600	1.0640	1.3253	1.7247	2.0860	2.1967	2.5280	2.8453	3.1534	3.5518
21	0.6864	0.8591	1.0627	1.3232	1.7207	2.0796	2.1894	2.5176	2.8314	3.1352	3.5271
22	0.6858	0.8583	1.0614	1.3212	1.7171	2.0739	2.1829	2.5083	2.8188	3.1188	3.5050
23	0.6853	0.8575	1.0603	1.3195	1.7139	2.0687	2.1770	2.4999	2.8073	3.1040	3.4850
24	0.6848	0.8569	1.0593	1.3178	1.7109	2.0639	2.1715	2.4922	2.7970	3.0905	3.4668
25	0.6844	0.8562	1.0584	1.3163	1.7081	2.0595	2.1666	2.4851	2.7874	3.0782	3.4502
26	0.6840	0.8557	1.0575	1.3150	1.7056	2.0555	2.1620	2.4786	2.7787	3.0669	3.4350
27	0.6837	0.8551	1.0567	1.3137	1.7033	2.0518	2.1578	2.4727	2.7707	3.0565	3.4210
28	0.6834	0.8546	1.0560	1.3125	1.7011	2.0484	2.1539	2.4671	2.7633	3.0470	3.4082
29	0.6830	0.8542	1.0553	1.3114	1.6991	2.0452	2.1503	2.4620	2.7564	3.0380	3.3963
30	0.6828	0.8538	1.0547	1.3104	1.6973	2.0423	2.1470	2.4573	2.7500	3.0298	3.3852
31	0.6825	0.8534	1.0541	1.3095	1.6955	2.0395	2.1438	2.4528	2.7440	3.0221	3.3749
32	0.6822	0.8530	1.0535	1.3086	1.6939	2.0369	2.1409	2.4487	2.7385	3.0149	3.3653
33	0.6820	0.8526	1.0530	1.3077	1.6924	2.0345	2.1382	2.4448	2.7333	3.0082	3.3563
34	0.6818	0.8523	1.0525	1.3070	1.6909	2.0322	2.1356	2.4411	2.7284	3.0020	3.3480
35	0.6816	0.8520	1.0520	1.3062	1.6896	2.0301	2.1332	2.4377	2.7238	2.9961	3.3400
36	0.6814	0.8517	1.0516	1.3055	1.6883	2.0281	2.1309	2.4345	2.7195	2.9905	3.3326
37	0.6812	0.8514	1.0512	1.3049	1.6871	2.0262	2.1287	2.4314	2.7154	2.9853	3.3256
38	0.6810	0.8512	1.0508	1.3042	1.6860	2.0244	2.1267	2.4286	2.7116	2.9803	3.3190
39	0.6808	0.8509	1.0504	1.3036	1.6849	2.0227	2.1247	2.4258	2.7079	2.9756	3.3127
40	0.6807	0.8507	1.0500	1.3031	1.6839	2.0211	2.1229	2.4233	2.7045	2.9712	3.3069
50	0.6794	0.8489	1.0473	1.2987	1.6759	2.0086	2.1087	2.4033	2.6778	2.9370	3.2614
60	0.6786	0.8477	1.0455	1.2958	1.6706	2.0003	2.0994	2.3901	2.6603	2.9146	3.2317
75	0.6778	0.8464	1.0436	1.2929	1.6654	1.9921	2.0901	2.3771	2.6430	2.8924	3.2024
100	0.6770	0.8452	1.0418	1.2901	1.6602	1.9840	2.0809	2.3642	2.6259	2.8707	3.1738
∞	0.6745	0.8416	1.0364	1.2816	1.6449	1.9600	2.0537	2.3263	2.5758	2.8070	3.0902

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The Rankit Table

Effect	Design size		
	N=8	*N*=12	*N*=16
1	0.09	0.06	0.04
2	0.27	0.17	0.12
3	0.46	0.29	0.21
4	0.66	0.41	0.29
5	0.90	0.53	0.38
6	1.21	0.67	0.47
7	1.71	0.81	0.57
8		0.98	0.67
9		1.19	0.78
10		1.45	0.89
11		1.91	1.02
12			1.18
13			1.36
14			1.61
15			2.04

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