
First Semester Examination
Academic Year 2005/2006

November 2005

KAA 501 – Quality Control in Chemistry

Time : 3 hours

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. Please comment on the importance and the ensuing implications upon the implementations of (i) Comprehensive quality system namely, *ISO9000:2000* and (ii) Laboratory accreditation system such as *SAMM or ISO17025.* Discuss the advantages and special characteristics of each system.

(20 marks)

2. (a) (i) Two out of ten consumers have lodged the complaints that the sugar weight in the plastic bag in a sundry shop not conforming with the standards. If there are 480 bags of sugar still available in the shop, explain the most probable type of sampling.

(ii) The City Council of Butterworth is supposed to determine the acceptance air quality level surrounding the industrial area. An officer Ahmad is assigned to carry out this project. Describe the type of sampling to be carried out by him.

(8 marks)

(b) The analysis upon the water quality in Penang is carred out by collecting 128 samples (1 L) from eight different areas (I – VIII).

Area	Number of sample	Cl^- (ppm)	pH	NO_3^- (ppm)
I	2	11.2	7.2	2.3
II	6	12.1	7.2	3.0
III	5	10.6	6.8	1.8
IV	2	9.5	6.8	1.7
V	1	13.8	7.1	3.1
VI	9	11.9	7.0	1.6
VII	3	8.3	6.8	1.7
VIII	4	10.4	6.9	2.0

It is known that the permissible concentration of Cl^- and NO_3^- ions should be equivalent or less than 10 and 2 ppm, respectively. As for the pH, the optimum value falls within the range 7.0-9.0.

(i) What is the percentage of sample not conforming with the standard value?

(ii) What is the percentage per 100 samples not conforming with the standard?

(iii) What is the acceptance quality level (AQL) per 1000 for the above sampling ?

(12 marks)

3. You are a manager to an analytical laboratory that wants to do analysis of illicit drugs in body fluids provided by a legal firm.

(i) Describe possible factors that may influence you in the selection of a method that fits for the purpose.

(ii) You had also noticed that your HPLC instrument had quite a number of downtimes within the past few months. Describe the use of any two appropriate tools of the magnificent seven in statistical process control for you to find the root cause of this problem.

(iii) Assume that you have decided to replace this high performance liquid chromatograph (HPLC). Discuss various factors that you should consider in trying to aid in this decision making.

(20 marks)

4. (a) Briefly discuss all the options available for the protection of intellectual property(IP). The discussion should include all types of protection, period of protection and the suitability of the option used for IP protection.

(10 marks)

(b) During the process of considering the registration of a patent for an individual or an organisation, please state:

(i) Three important characteristics which will permit the registration of a patent for a product or process.

(ii) The importance and implications of a novelty search.

(10 marks)

...10/-

SECTION B

5. (a) (i) By based on any sample, discuss about the importance of acceptance sampling.

(iii) Three chemists from 3 different laboratories have received samples (in bulk) from the same client for the analytical purpose.

Chemist	Number of samples
Daniel	280
Kok	1030
Faizah	80

Discuss the suitable subsampling procedure and inspection for each analytical measurement.

(10 marks)

(b) Describe the calibration method used for each of the following equipment:

(i) Polarizing optical microscope.

(ii) High precision electronic balance

(10 marks)

6. 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.

(a) Factors	**Effects on**						
	%MC	**%RCI**	**%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
Butter 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 resolution of the critical peak pair, Rs, by using errors estimated from dummy factors at $\alpha = 0.05$ and $\alpha = 0.10$.

(ii) Produce a half normal plot for the case of the tailing or asymmetry factor, Asf of MC, together with margin of error (ME) and simultaneous margin error (SME). Identify any known significant effects.

(20 marks)

7. The results of three analysts performing the determination of chemical oxygen demand (COD) in water samples collected from a stream receiving a heavy industrial discharge are shown in the following Table.

Analyst A (mg/L)	**Analyst B (mg/L)**	**Analyst C (mg/L)**
262	257	266
260	257	269
255	259	266
257	254	264
259	54	267
269	258	279
264	257	295
271	258	279
265	261	266

(i) For each set of data, determine if there are any outliers or not.

(ii) Determine whether there exist significant differences at 95% confidence level between these data set.

(iii) Discuss possible quality controls that can be introduced to detect errors from contamination involving these collected water samples.

(20 marks)

Appendix 1

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

where

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

$M_z = 0.6745\ [X_i - X_{avg}] / \text{MAD}$

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

$$\bar{x} - \mu = \pm \frac{ts}{\sqrt{N}}$$

$$s_{\bar{x}} = \frac{s}{\sqrt{n}} = \sqrt{\frac{s^2}{n}}$$

$$s^2_{\text{pooled}} = \frac{SS_1 + SS_2}{n_1 + n_2 - 2}$$
$$= \frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{n_1 + n_2 - 2}.$$

$$t_s = \frac{(\bar{y}_1 - \bar{y}_2) - (\mu_1 - \mu_2)}{SE_{(\bar{y}_1 - \bar{y}_2)}} = \frac{(\bar{y}_1 - \bar{y}_2) - 0}{SE_{(\bar{y}_1 - \bar{y}_2)}} = \frac{\bar{y}_1 - \bar{y}_2}{SE_{(\bar{y}_1 - \bar{y}_2)}}$$

$$SE_{\text{pooled}} = \sqrt{s^2_{\text{pooled}}\left(\frac{1}{n_1} + \frac{1}{n_2}\right)}$$

$$df = \frac{\left(SE_1^2 + SE_2^2\right)^2}{\frac{SE_1^4}{(n_1 - 1)} + \frac{SE_2^4}{(n_2 - 1)}}$$

$$t = \frac{\bar{d}}{\sqrt{s^2 \ n}}$$

$X^2_s = \Sigma\ (O-E)^2/E$

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{n}}{\sqrt{\left[\sum X^2 - \frac{(\sum X)^2}{n}\right]\left[\sum Y^2 - \frac{(\sum Y)^2}{n}\right]}}$$

The ANOVA Table

It is common to organize results from an ANOVA into a table as follows:

Source	df	SS	MS
Between Groups	$I - 1$	$\sum_i n_i(\bar{y}_{i\bullet} - \bar{y}_{\bullet\bullet})^2$	SS/df
Within Groups	$n^* - I$	$\sum_i \sum_j (y_{ij} - \bar{y}_{i\bullet})^2$	SS/df
Total	$n^* - 1$	$\sum_i \sum_j (y_{ij} - \bar{y}_{\bullet\bullet})^2$	

$$\text{Recovery}(\%) = \frac{\overline{X}_s - \overline{X}}{X_{add}} \times 100\%$$

$$CV = 2(1-0.5\log C)$$

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

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

$$ME = t_{(1-\alpha/2,df)} \cdot s_1$$

$$\alpha^* = 1 - (1 - \alpha)^{(1/m)}$$

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

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

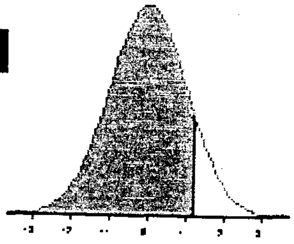
$$s_o = 1.5 \cdot \underset{i}{median} \mid E_i \mid$$

$$EX = \frac{\sum Y(+)}{N/2} - \frac{\sum Y(-)}{N/2}$$

Critical Values of the F Distribution
(α = .05)

df within	df between										
	1	2	3	4	5	6	7	8	12	24	∞
5	6.61	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.68	4.53	4.37
6	5.99	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.00	3.84	3.67
7	5.59	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.57	3.41	3.23
8	5.32	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.28	3.12	2.93
9	5.12	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.07	2.90	2.71
10	4.96	4.10	3.71	3.48	3.33	3.22	3.14	3.07	2.91	2.74	2.54
11	4.84	3.98	3.59	3.36	3.20	3.09	3.01	2.95	2.79	2.61	2.41
12	4.75	3.89	3.49	3.26	3.11	3.00	2.91	2.85	2.69	2.51	2.30
13	4.67	3.81	3.41	3.18	3.03	2.92	2.83	2.77	2.60	2.42	2.21
14	4.60	3.74	3.34	3.11	2.96	2.85	2.76	2.70	2.53	2.35	2.13
15	4.54	3.68	3.29	3.06	2.90	2.79	2.71	2.64	2.48	2.29	2.07
16	4.49	3.63	3.24	3.01	2.85	2.74	2.66	2.59	2.42	2.24	2.01
17	4.45	3.59	3.20	2.96	2.81	2.70	2.61	2.55	2.38	2.19	1.96
18	4.41	3.55	3.16	2.93	2.77	2.66	2.58	2.51	2.34	2.15	1.92
19	4.38	3.52	3.13	2.90	2.74	2.63	2.54	2.48	2.31	2.11	1.88
20	4.35	3.49	3.10	2.87	2.71	2.60	2.51	2.45	2.28	2.08	1.84
21	4.32	3.47	3.07	2.84	2.68	2.57	2.49	2.42	2.25	2.05	1.81
22	4.30	3.44	3.05	2.82	2.66	2.55	2.46	2.40	2.23	2.03	1.78
23	4.28	3.42	3.03	2.80	2.64	2.53	2.44	2.37	2.20	2.01	1.76
24	4.26	3.40	3.01	2.78	2.62	2.51	2.42	2.36	2.18	1.98	1.73
25	4.24	3.39	2.99	2.76	2.60	2.49	2.40	2.34	2.16	1.96	1.71
26	4.23	3.37	2.98	2.74	2.59	2.47	2.39	2.32	2.15	1.95	1.69
27	4.21	3.35	2.96	2.73	2.57	2.46	2.37	2.31	2.13	1.93	1.67
28	4.20	3.34	2.95	2.71	2.56	2.45	2.36	2.29	2.12	1.91	1.66
29	4.18	3.33	2.93	2.70	2.55	2.43	2.35	2.28	2.10	1.90	1.64
30	4.17	3.32	2.92	2.69	2.53	2.42	2.33	2.27	2.09	1.89	1.62
40	4.08	3.23	2.84	2.61	2.45	2.34	2.25	2.18	2.00	1.79	1.51
60	4.00	3.15	2.76	2.53	2.37	2.25	2.17	2.10	1.92	1.70	1.39
80	3.96	3.11	2.72	2.49	2.33	2.21	2.13	2.06	1.88	1.65	1.33
100	3.94	3.09	2.70	2.46	2.31	2.19	2.10	2.03	1.85	1.63	1.28
120	3.92	3.07	2.68	2.45	2.29	2.18	2.09	2.02	1.83	1.61	1.26
∞	3.84	3.00	2.61	2.37	2.22	2.10	2.01	1.94	1.75	1.52	1.00

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

—ooo O ooo—