
UNIVERSITI SAINS MALAYSIA

First Semester Examination
Academic Session 2008/200

November 2008

KAA 501 – Quality Control In Chemistry
[Kawalan Mutu Dalam Kimia]

Duration : 3 hours
[Masa : 3 jam]

Please check that this examination paper consists of **TWENTY** printed pages before you begin the examination.

Instructions:

Answer **FIVE** (5) questions.

SECTION A, questions 1, 2, 3 are **COMPULSORY**.

SECTION B, select and answer any **TWO** (2) questions from No. 4 – 7.

Begin the answers to each question on a new page.

You may answer the questions either in Bahasa Malaysia or in English.

If a candidate answers more than five questions, only the answers to the first five questions in the answer sheet will be graded.

Appendix: AQL Sampling Table based on the Mil-STD-105D

Cumulative standard normal distribution (values of the probability ϕ corresponding to the value Z_ϕ of a standard normal random variable)

Plackett and Burman Design for 11 factors

Constants for the construction of control charts

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SECTION A

1. A chemist is studying a newly developed polymer to be used in removing toxic wastes from water. Experiments were conducted at 5 different temperatures. The response recorded was the percentage of impurities removed by the treatment. The data are given in the following table;

| Temperature | | | | | |
|-----------------------------------|------|------|------|------|------|
| 1 | 2 | 3 | 4 | 5 | |
| 40 | 36 | 49 | 47 | 55 | |
| 45 | 42 | 51 | 49 | 60 | |
| 42 | 38 | 53 | 51 | 62 | |
| 48 | 39 | 53 | 52 | 63 | |
| 50 | 37 | 52 | 50 | 59 | |
| 51 | 40 | 50 | 51 | 61 | |
| n_i | 6 | 6 | 6 | 6 | 6 |
| T_i | 276 | 232 | 308 | 300 | 360 |
| \bar{X}_i | 46.0 | 38.7 | 51.3 | 50.0 | 60.0 |
| $\sum_i \sum_j X_{ij}^2 = 74.268$ | | | | | |

- (i) Are there significant differences in the percentage of impurities removed among the five temperatures? (Assume normality of each set of data)
- (ii) Use Bonferroni t test to determine which differences exist at significance level of 5 %.
- (20 marks)
2. A manufacturer receives large batches of components daily and decides to institute an acceptance sampling scheme. Three alternative plans are considered.

- Plan A: A sample of 30 is inspected and the batch is accepted if no non-conforming items are found, otherwise the batch is rejected.
- Plan B: A sample of 30 is inspected and the batch is accepted if not more than one non-conforming component is found, otherwise the batch is rejected.
- Plan C: A sample of 60 is inspected and the batch is accepted if not more than one non-conforming component is found, otherwise the batch is rejected.

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- (a) For each plan, calculate the probability of accepting batches containing 2, 5 and 10% non-conforming. (8 marks)
- (b) Sketch on the same axes the operating characteristics of each plan. (4 marks)
- (c) Which plan would be the most appropriate in each of the following circumstances?
- (i) There should be a high probability of accepting batches containing 2% non-conforming.
- (ii) There should be a high probability of rejecting batches containing 10% non-conforming. (4 marks)
- (d) Discuss the factors to be considered when deciding which of the plans to use. (4 marks)

(Given: The binomial formula $P(d \leq c) = \sum_{d=0}^c [n! / d!(n-d)!] p^d (1-p)^{n-d}$)

3. (a) Briefly explain the following terms commonly used in validation and statistical process control.
- (i) Sensitivity.
- (ii) Specificity
- (iii) Process capability index. (8 marks)

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- (b) A robustness test was done on a HPLC method to determine active main compound (MC) and for the detection of two related compounds (RC1) and (RC2) in a pharmaceutical sample. This study was done using Plackett Burmann Experimental design for 11 factors (N=12) that includes 8 factors and 3 dummy factors (see Table 1 in appendix). One of the results of the study for MC was based on asymmetry factor (Asf). The results of the study are given in the following table. Identify the factors that have significant effect on the determination of MC from the perspective of asymmetry factor by using Dong's algorithm and dummy effects at $\alpha = 0.05$.

| Factors | Asf (MC) |
|--------------|----------|
| pH | 0.204 |
| Column | -0.432 |
| Dummy 1 | -0.065 |
| Temp | -0.103 |
| % B begin | -0.147 |
| % B end | -0.013 |
| Dummy 2 | -0.003 |
| Flow | -0.146 |
| Wavelength | 0.067 |
| Buffer conc. | 0.029 |
| Dummy 3 | -0.011 |

(12 marks)

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SECTION B

4. (a) Consider the data given in the following table that relates to the analysis of Pb in mg/Kg^{-1} of composites guava leaf samples. The purpose of the analysis was to evaluate the lead air pollution due to the automobiles. Provide a conclusion of the comparison on the effect of air pollution on guava from both districts.

| Composite sample | Ipoh Guava | Tapah Guava |
|------------------|------------|-------------|
| 1 | 2.10 | 2.94 |
| 2 | 2.82 | 1.98 |
| 3 | 2.17 | 2.38 |
| 4 | 1.99 | 2.65 |
| 5 | 2.22 | 2.77 |
| 6 | 3.09 | 1.85 |
| 7 | 2.47 | 1.95 |
| 8 | 2.52 | 2.70 |
| 9 | 2.80 | 2.68 |
| 10 | 2.10 | 2.06 |

(10 marks)

- (b) Discuss the systematic and random effects in sampling and the measures which can be adopted to reduce them.
- (10 marks)
5. For two variables x and y on a large population, we assume there is a linear model:

$$Y = \beta_0 + \beta_1 X + \varepsilon_x \quad \text{where } \varepsilon_x \approx N(0, \sigma^2)$$

We take a sample of 50 (x, y) points and obtain: $\sum x = 8$, $\sum y = 2$, $\sum xy = 0.71$, $\sum x^2 = 2.3$, $\sum y^2 = 0.23$

- (i) Find the regression line and estimate σ .
- (ii) What percentage of the Y values is due to the linear model?
- (iii) Using regression line coefficient, perform a test whether there is a meaningful linear relationship between X and Y .

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- (iv) Give a 95 % confidence interval for the slope of the regression line.

(20 marks)

6. (a) Describe systematically the process of constructing Ishikawa or cause and effect diagram in solving the problem of too often downtime for an analytical instrument in your laboratory.

(5 marks)

- (b) In a control chart of an analytical process, a pattern of shifting means is observed. Provide possible causes of this observed pattern.

(5 marks)

- (c) The specification of a chemical process is set at 197.5 ± 27.5 . At present, the reject rate due to exceeding the Upper Specification Limit (USL) is 1.5 % and the scrap rate due to falling below the Lower Specification Limit (LSL) is 0.5 %. Assume the process parameter has a normal distribution, find the process performance index

(10 marks)

7. (a) A method for detection of phosphate in lake water provided an estimated detection limit of 0.045 mgL^{-1} . An analyst had decided to verify the method detection limit. A standard was prepared with PO_4^{3-} concentration of 0.175 mg L^{-1} . This solution was analyzed 9 times over the course of several days. The results are given in the table below.

| Replicates | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|----------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Results, mgL^{-1} | 0.194 | 0.166 | 0.174 | 0.149 | 0.183 | 0.153 | 0.144 | 0.173 | 0.190 |

- (i) Construct a box plot and use it to identify any possible outliers.
- (ii) Determine method detection limit (MDL), limit of quantification (LOQ) and percent mean recovery.

(12 marks)

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- (b) There are several quality systems that are well accepted by industries and laboratories namely ISO 9001, ISO 14000 and ISO/IEC 17025. ISO/IEC 17025 was a modified version of ISO G25. Provide a short explanation of the differences between the scheme accredited for ISO 17025 and ISO G25 and the key differences between accreditation and certification.

(8 marks)

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Appendix 1

$$Z_2 = \frac{(x_2 - \bar{x})}{s}$$

Where

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

$$Mz = 0.6745 [X_i - X_{avg}] / MAD$$

$$R_{2+1} = \frac{|x^{(2)} - \bar{x}^{(2)}|}{s^{(2)}}$$

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

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

$$s_{pooled}^2 = \frac{SS_1 + SS_2}{n_1 + n_2 - 2}$$

$$= \frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{n_1 + n_2 - 2}$$

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

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

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

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

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

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

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 | $n^* - 1$ | $\sum_i \sum_j (y_{ij} - \bar{y} \bullet \bullet)^2$ | |

$$\text{Recovery}(\%) = \frac{\bar{x}_s - \bar{x}}{x_{add}} \times 100\%$$

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

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

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

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

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

$$E_x = t_{critical} \text{ multiply } (SE)_e$$

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

$$s_o = 1.5 \cdot \text{median}_i |E_i|$$

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

$$T = \frac{(\bar{x}_1 - \bar{x}_2) - (\mu_1 - \mu_2)}{SE_{\bar{x}_1 - \bar{x}_2}}$$

$$|\bar{x}_1 - \bar{x}_2|_{least} = t_{critical} \times s \times \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}$$

$$HSD = q \sqrt{\frac{MS_{within}}{n}}$$

$$q_{obs} = \frac{\bar{X}_B - \bar{X}_A}{SE_{Tukey}}$$

$$SE_{Tukey} = \sqrt{\frac{s_w^2}{n}}, \text{ for equal sample sizes}$$

$$SE_{Tukey} = \sqrt{\frac{9.7652}{6}} = 1.28$$

$$SE_{\bar{x}_1 - \bar{x}_2} = s \times \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}$$

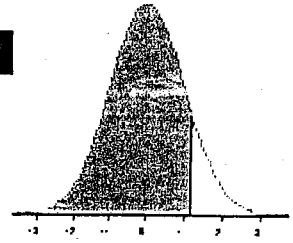
$$|\bar{x}_j - \bar{x}_j| > t_{\alpha/2} \sqrt{MSW \left(\frac{1}{n_j} + \frac{1}{n_j} \right)}$$

$$\text{or } \frac{|\bar{x}_1 - \bar{x}_2|}{\sqrt{MSW \left(\frac{1}{n_j} + \frac{1}{n_j} \right)}} > t_{\alpha/2}$$

$$S_{obs} = \frac{|\sum c_i X_i|}{SE_{Scheffe-subsets}}$$

$$SE_{Scheffe-subsets} = \sqrt{\left(s_w^2 \right) \left(\sum \frac{c_i^2}{n_i} \right)}$$

Student's t-distribution table



| df | p | | | | | | | | | | |
|-----|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| | 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 |

Critical Values of the F Distribution
($\alpha = .05$)

| 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 |

The critical values for q corresponding to alpha = .05 (top) and alpha = .01 (bottom)

| df for Error Term | k= Number of Treatments | | | | | | | | |
|-------------------|-------------------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|---------------|
| | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| 5 | 3.64 5.70 | 4.60 6.98 | 5.22 7.80 | 5.67 8.42 | 6.03 8.91 | 6.33 9.32 | 6.58 9.67 | 6.80 9.97 | 6.99 10.24 |
| 6 | 3.46 5.24 | 4.34 6.33 | 4.90 7.03 | 5.30 7.56 | 5.63 7.97 | 5.90 8.32 | 6.12 8.61 | 6.32 8.87 | 6.49 9.10 |
| 7 | 3.34 4.95 | 4.16 5.92 | 4.68 6.54 | 5.06 7.01 | 5.36 7.37 | 5.61 7.68 | 5.82 7.94 | 6.00 8.17 | 6.16 8.37 |
| 8 | 3.26 4.75 | 4.04 5.64 | 4.53 6.20 | 4.89 6.62 | 5.17 6.96 | 5.40 7.24 | 5.60 7.47 | 5.77 7.68 | 5.92 7.86 |
| 9 | 3.20 4.60 | 3.95 5.43 | 4.41 5.96 | 4.76 6.35 | 5.02 6.66 | 5.24 6.91 | 5.43 7.13 | 5.59 7.33 | 5.74 7.49 |
| 10 | 3.15 4.48 | 3.88 5.27 | 4.33 5.77 | 4.65 6.14 | 4.91 6.43 | 5.12 6.67 | 5.30 6.87 | 5.46 7.05 | 5.60 7.21 |
| 11 | 3.11 4.39 | 3.82 5.15 | 4.26 5.62 | 4.57 5.97 | 4.82 6.25 | 5.03 6.48 | 5.20 6.67 | 5.35 6.84 | 5.49 6.99 |
| 12 | 3.08 4.32 | 3.77 5.05 | 4.20 5.50 | 4.51 5.84 | 4.75 6.10 | 4.95 6.32 | 5.12 6.51 | 5.27 6.67 | 5.39 6.81 |
| 13 | 3.06 4.26 | 3.73 4.96 | 4.15 5.40 | 4.45 5.73 | 4.69 5.98 | 4.88 6.19 | 5.05 6.37 | 5.19 6.53 | 5.32 6.67 |
| 14 | 3.03 4.21 | 3.70 4.89 | 4.11 5.32 | 4.41 5.63 | 4.64 5.88 | 4.83 6.08 | 4.99 6.26 | 5.13 6.41 | 5.25 6.54 |
| 15 | 3.01 4.17 | 3.67 4.84 | 4.08 5.25 | 4.37 5.56 | 4.59 5.80 | 4.78 5.99 | 4.94 6.16 | 5.08 6.31 | 5.20 6.44 |
| 16 | 3.00 4.13 | 3.65 4.79 | 4.05 5.19 | 4.33 5.49 | 4.56 5.72 | 4.74 5.92 | 4.90 6.08 | 5.03 6.22 | 5.15 6.35 |
| 17 | 2.98 4.10 | 3.63 4.74 | 4.02 5.14 | 4.30 5.43 | 4.52 5.66 | 4.70 5.85 | 4.86 6.01 | 4.99 6.15 | 5.11 6.27 |
| 18 | 2.97 4.07 | 3.61 4.70 | 4.00 5.09 | 4.28 5.38 | 4.49 5.60 | 4.67 5.79 | 4.82 5.94 | 4.96 6.08 | 5.07 6.20 |
| 19 | 2.96 4.05 | 3.59 4.67 | 3.98 5.05 | 4.25 5.33 | 4.47 5.55 | 4.65 5.73 | 4.79 5.89 | 4.92 6.02 | 5.04 6.14 |
| 20 | 2.95 4.02 | 3.58 4.64 | 3.96 5.02 | 4.23 5.29 | 4.45 5.51 | 4.62 5.69 | 4.77 5.84 | 4.90 5.97 | 5.01 6.09 |
| 24 | 2.92 3.96 | 3.53 4.55 | 3.90 4.91 | 4.17 5.17 | 4.37 5.37 | 4.54 5.54 | 4.68 5.69 | 4.81 5.81 | 4.92 5.92 |
| 30 | 2.89 3.89 | 3.49 4.45 | 3.85 4.80 | 4.10 5.05 | 4.30 5.24 | 4.46 5.40 | 4.60 5.54 | 4.72 5.65 | 4.82 5.76 |
| 40 | 2.86 3.82 | 3.44 4.37 | 3.79 4.70 | 4.04 4.93 | 4.23 5.11 | 4.39 5.26 | 4.52 5.39 | 4.63 5.50 | 4.73 5.60 |
| 60 | 2.83 3.76 | 3.40 4.28 | 3.74 4.59 | 3.98 4.82 | 4.16 4.99 | 4.31 5.13 | 4.44 5.25 | 4.55 5.36 | 4.65 5.45 |

TERJEMAHAN

Arahan:

Jawab **LIMA** (5) soalan.

BAHAGIAN A, soalan-soalan 1, 2 dan 3 **PERLU** dijawab.

BAHAGIAN B, pilih dan jawab **DUA** (2) soalan dari soalan-soalan 4 – 7.

Anda perlu mulakan setiap soalan pada muka surat baru.

Anda boleh menjawab sama ada dalam Bahasa Melayu atau Bahasa Inggeris.

Jika calon menjawab lebih daripada lima soalan, hanya lima soalan pertama mengikut susunan dalam skrip jawapan akan diberi markah.

...15/-

BAHAGIAN A

1. Seorang ahli kimia telah mengkaji suatu polimer yang baru dihasilkan untuk digunakan dalam pemecatan bahan bertoksik daripada air. Eksperimen dilakukan pada 5 suhu yang berbeza. Gerakbalas yang dirakamkan ialah peratus bahan cemar yang dipekat melalui pengolahan tersebut. Data diberikan di dalam jadual berikut.

| Suhu, °C | | | | |
|-----------------------------------|------|------|------|------|
| 1 | 2 | 3 | 4 | 5 |
| 40 | 36 | 49 | 47 | 55 |
| 45 | 42 | 51 | 49 | 60 |
| 42 | 38 | 53 | 51 | 62 |
| 48 | 39 | 53 | 52 | 63 |
| 50 | 37 | 52 | 50 | 59 |
| 51 | 40 | 50 | 51 | 61 |
| n_i | 6 | 6 | 6 | 6 |
| T_i | 276 | 232 | 308 | 300 |
| $X_{i\text{-bar}}$ | 46.0 | 38.7 | 51.3 | 50.0 |
| $\sum_i \sum_j X_{ij}^2 = 74.268$ | | | | |

- (i) Apakah terdapat perbezaan yang ketara dalam peratus bahan cemar yang dipekat dikalangan lima suhu pengolahan tersebut? (Anggap kenormalan bagi setiap graf)
- (ii) Guna ujian Benferroni untuk menentukan perbezaan yang wujud pada paras signifikan 5 %.

(20 marks)

2. Seorang pekilang menerima kelompok komponen yang besar tiap-tiap hari and ia memutuskan untuk memulakan skema pensampelan penerimaan. Tiga rancangan alternatif dicadangkan.

Rancangan A: Sebanyak 30 sampel diperiksa dan kelompok itu diterima jika tidak ada item yang tidak mematu. Sebaliknya, kelompok itu ditolak.

Rancangan B: Sebanyak 30 sampel diperiksa dan kelompok itu diterima jika tidak melebihi satu item yang tidak mematu ditemui. Sebaliknya, kelompok itu ditolak.

Rancangan C: Sebanyak 60 sampel diperiksa dan kelompok itu diterima jika tidak melebihi satu item yang tidak mematu ditemui. Sebaliknya, kelompok itu ditolak.

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- (a) Bagi setiap rancangan, kirakan kebarangkalian untuk menerima kelompok yang mengandungi 2, 5 dan 10% item yang tidak mematuhi. (8 markah)
- (b) Lakarkan pada paksi yang sama lengkungan ciri-ciri pengoperasian bagi setiap rancangan. (4 markah)
- (c) Rancangan yang manakah yang paling sesuai bagi setiap keadaan yang berikut:
- (v) Terdapat kebarangkalian yang tinggi untuk menerima kelompok yang mengandungi 2% item yang tidak mematuhi.
- (vi) Terdapat kebarangkalian yang tinggi untuk menolak kelompok yang mengandungi 10% item yang tidak mematuhi. (4 markah)
- (d) Bincangkan faktor yang dipertimbangkan ketika memilih rancangan yang digunakan. (4 markah)

(Diberikan: Formula binomial $P(d \leq c) = \sum_{d=0}^c [n! / d!(n-d)!] p^d (1-p)^{n-d}$)

3. (a) Terangkan dengan ringkas sebutan-sebutan berikut yang dipakai dalam pengesahan dan kawalan proses statistik.
- (i) Kepekaan
- (ii) Kespesifikan
- (iii) Indeks kecekapan proses (8 marks)

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- (b) Satu kajian ketegapan telah dilakukan ke atas satu kaedah HPLC (Kromatografi cecair prestasi tinggi) bagi menentukan satu sebatian utama (MC) dan untuk mengesan dua sebatian yang berkaitan (RC1 dan RC(2) dalam suatu sampel farmasi. Kajian ini dilakukan dengan menggunakan rekabentuk eksperimen Plackett Burmann bagi 11 faktor (N=12) yang melibatkan 8 faktor dan 3 faktor dumi (lihat Jadual 1 yang dibekalkan dalam appendiks). Satu daripada keputusan kajian diberikan dalam jadual berikut. Kenalpastikan faktor-faktor yang memberikan kesan signifikan dalam penentuan MC berdasarkan faktor asimetri dengan menggunakan algorithma Dong dan kesan faktor dumi pada $\alpha=0.05$.

| Faktor | Asf (MC) |
|------------------|----------|
| pH | 0.204 |
| Turus | -0.432 |
| Dum 1 | -0.065 |
| Suhu | -0.103 |
| % B mula | -0.147 |
| % B akhir | -0.013 |
| Dum 2 | -0.003 |
| aliran | -0.146 |
| Jarak gelombang | 0.067 |
| Kepeka. Penimbal | 0.029 |
| Dum 3 | -0.011 |

(12 markah)

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BAHAGIAN B

4. (a) Pertimbangkan data yang diberikan dalam jadual berikut yang berkaitan dengan analisis Pb dalam mgL^{-1} terhadap sampel komposit daun jambu. Tujuan analisis ialah untuk menilai pencemaran plumbum dalam udara oleh kenderaan. Berikan satu keputusan terhadap perbandingan kesan pencemaran terhadap jambu daripada kedua-dua daerah tersebut.

| Sampel komposit | Jambu Ipoh | Jambu Guava |
|-----------------|------------|-------------|
| 1 | 2.1 | 2.94 |
| 2 | 2.82 | 1.98 |
| 3 | 2.17 | 2.38 |
| 4 | 1.99 | 2.65 |
| 5 | 2.22 | 2.77 |
| 6 | 3.09 | 1.85 |
| 7 | 2.47 | 1.95 |
| 8 | 2.52 | 2.70 |
| 9 | 2.80 | 2.68 |
| 10 | 2.10 | 2.06 |

- (b) Bincangkan kesan sistematik dan rambang dalam pensampelan dan langkah yang boleh diambil untuk mengurangkannya. (10 markah)
5. Untuk dua pembolehubah x dan y bagi satu populasi besar, kita menganggap ia satu model linear:

$$Y = \beta_0 + \beta_1 X + \varepsilon_x \quad \text{where } \varepsilon_x \approx N(0, \sigma^2)$$

Kita ambil satu sampel titik 50 (x, y) dan memperolehi $\sum x = 8$, $\sum y = 2$, $\sum xy = 0.71$, $\sum x^2 = 2.3$, $\sum y^2 = 0.23$

- (i) Cari garis regresi dan anggaran σ .
- (ii) Berapakah peratus nilai-nilai y hasil daripada model linear ini.
- (iii) Dengan menggunakan pekali garis regresi, lakukan satu ujian samada terdapat hubungan linear yang bermakna di antara x dan y .

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- (iv) Beri selang keyakinan 95 % untuk kecerunan garis regresi.
(20 markah)
6. (a) Berikan secara sistematik proses membina gambarajah Ishikawa atau kesan-sebab dalam menyelesaikan masalah kerosakkan sesuatu peralatan analisis dalam makmal anda.
(5 markah)
- (b) Dalam carta kawalan suatu proses analisis, satu pola anjakan purata telah diperhatikan. Berikan sebab-sebab yang mungkin terhadap pemerhatian pola ini.
(5 markah)
- (c) Spesifikasi suatu proses kimia ditetapkan pada 197.5 ± 27.5 . Pada masa kini, kadar tolak akibat melebihi had spesifikasi atas (*USL) ialah 1.5 % dan kadar tolak akibat jatuh di bawah had spesifikasi bawah ialah 0.5 %. Anggaph parameter proses ini mempunyai taburan normal, kiralah indeks prestasi proses.
(10 markah)
7. (a) Satu kaedah penentuan fosfat dalam air tasik memberikan anggaran had pengesanan 0.045 mgL^{-1} . Seorang juruanalisa membuat keputusan untuk menguji kebenaran had pengesanan kaedah ini. Satu piawai disediakan dengan kepekatan PO_4^{3-} pada 0.175 mg L^{-1} . Larutan ini dianalisis sebanyak 9 kali dalam tempoh beberapa hari. Keputusan diberikan di dalam jadual di bawah.

| Replikat | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Keputusan, mgL^{-1} | 0.194 | 0.166 | 0.174 | 0.149 | 0.183 | 0.153 | 0.144 | 0.173 | 0.190 |

- (i) Bina satu plot kotak dan gunakannya untuk mengenalpasti sebarang data terpencil yang mungkin wujud.
- (ii) Tentukan had pengesanan kaedah (MDL), had kuantifikasi (LOQ) dan peratus purata pemuliharaan.
(12 markah)

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- (b) Terdapat beberapa sistem kualiti yang diterima baik oleh industri dan makmal iaitu ISO 9001, ISO 14000 dan ISO/IEC 17025. ISO/IEC 17025 adalah versi ISO G25 yang telah dimodifikasikan. Berikan satu penerangan pendek tentang perbezaan di antara skema akreditasi ISO 17025 dan ISO G25 dan perbezaan utama di antara akreditasi dan perakuan.

(8 markah)

oooOOooo