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UNIVERSITI SAINS MALAYSIA

First Semester Examination  
Academic Session 2008/2009

November 2008

**KAE 345 – Special Topics In Analytical Chemistry**  
**[Tajuk Khusus dalam Kimia Analisis]**

Duration: 3 hours  
*[Masa : 3 jam]*

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

**Instruction:-**

Answer any **FIVE** (5) questions.

This paper consists of **SEVEN** (7) questions in two sections **SECTION A AND B**, answer at least **TWO** (2) questions from each section.

Answer each question on a new page.

You may answer 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.

**SECTION A : STATISTICS**

1. (a) Describe the difference between null and alternative hypothesis.  
(4 marks)
- (b) If in repeated titrations of 5.00 mL of 0.10 M HCl with 0.10 M NaOH, the results are normally distributed, the mean being 5.00 mL alkali and the standard deviation 0.04 mL. What proportion and percentage of titrations would the volume of alkali be expected
- (i) to exceed 4.95 mL?  
(ii) between 4.90 and 5.05 mL?
- (6 marks)

- (c) The phosphorus content was measured for three different soil locations. Five replicate determinations were made on each soil sample. A partial ANOVA table follows:

Variation Source	Sum of squares	Degree of freedom	Mean squares	F value
Between soils				
Within soils			0.0081	
Total	0.374			

- (i) Fill in the missing entries in the ANOVA table.  
(ii) State the null and alternative hypotheses.  
(iii) Do the three soils differ in phosphorus content at the 95 % confidence level?
- (10 marks)
2. (a) An ion-selective electrode (ISE) determination of sulphide from sulphate reducing bacteria was compared with a gravimetric determination. The results obtained were expressed in milligrams of sulphide.

No.	mg sulphide	
	ISE method	Gravimetric method
1	108	105
2	12	16
3	152	113
4	3	0
5	106	108
6	11	11
7	128	141
8	12	11
9	160	182
10	128	118

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- (i) Determine the equation for the least squares regression line to estimate values by the gravimetric method (Y) from values by the ISE method (X).
- (ii) Estimate the 95 % confidence limit for the slope and intercept.
- (iii) Comment on the suitability of the ISE method for this sulphide determination.

Given:

Estimate the random errors in the y-direction:

$$S_{Y/X} = \sqrt{\frac{\sum (Y_i - \hat{Y}_i)^2}{n - 2}}$$

Standard deviation of slope:

$$S_b = \frac{S_{Y/X}}{\sqrt{\sum (X_i - \bar{X})^2}}$$

Standard deviation of intercept:

$$S_a = S_{Y/X} \sqrt{\frac{\sum X_i^2}{n \sum (X_i - \bar{X})^2}}$$

(10 marks)

- (b) Under statistical control, a process produces a powder with a mean Fe content of 6.70 % (w/w) with a standard deviation of 0.18 %. Samples of size 4 are routinely analysed weekly by the quality control laboratory. The following sequence of results was obtained for the mean Fe percentage:

6.68, 6.71, 6.68, 6.72, 6.82, 6.54, 6.59, 6.58, 6.71, 6.48, 6.57, 6.61, 6.64, 6.56, 6.69, 6.64, 6.69, 6.57, 6.54, 6.56, 6.55, 6.50, 6.55, 6.51, 6.42.

Construct a Shewhart control chart for the above results and comment on the chart.

(10 marks)

...4/-

3. (a) A customer purchased two tank cars of a residue amine product. The total nitrogen analyses are listed below. Based on hypothesis testing, do the two tank cars have equivalent total nitrogen contents at  $P = 0.05$ ?

% Total Nitrogen	
Car 1	Car 2
9.23	9.18
9.28	9.01
9.17	9.09
9.24	9.20
9.20	9.00
9.24	
9.19	
9.21	

(8 marks)

- (b) Four laboratories (A - D) made five determinations of ethyl acetate content on samples from the same drum of material. The results obtained by the laboratories are listed below. Give comments on the average values obtained by the four laboratories at 95 % confidence level.

Determination	Ethyl acetate (%)			
	Laboratory			
	A	B	C	D
1	73	74	68	71
2	75	74	69	72
3	73	75	69	72
4	75	74	70	71
5	73	74	69	73

(12 marks)

4. (a) Explain what the normal distribution curve is and how it can be related to the error? Also explain briefly the difference between normal distribution and the standardized normal distribution.

(6 marks)

- (b) Four standard solutions were prepared, each calculated to contain 16.00 % w/w of chloride. Three titration methods (A, B and D), each with a different technique of endpoint determination, were used to analyze each standard solution. The order of the experiments was randomized. The results for the chloride found are shown below.

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Method	Percentage Cl <sup>-</sup> (% w/w)			
	Solution 1	Solution 2	Solution 3	Solution 4
A	16.03	16.05	16.02	16.12
B	16.13	16.13	15.94	15.97
C	16.09	16.15	16.12	16.10

Test whether there are any significant differences between

- (i) the concentration of chloride in the different solutions, and
- (ii) the results obtained by the different methods at 5 % level of significance.

(14 marks)

### **SECTION B: FORENSIC**

5. (a) A Forensic chemist compares the obvious and the inconspicuous writing features in the questioned document with the writing features in the exemplar writing to find the combinations of significant similarities and differences. Comment on this statement.
 

(5 marks)
- (b) Explain the steps taken by the forensic chemist towards reporting whether the signature on the above document is genuine or an imitation, and identifying who wrote the signature or other writing on the document?
 

(7 marks)
- (c) Fingerprint having loop pattern are dominant in 65 percent of the population; the whorl pattern are dominant in approximately 30 percent; and the arch pattern are dominant in approximately 5 percent. In addition to that several minutiae are also unique to the owner of the finger. Elaborate how the finding of the patterns of fingerprint can be revealed and related to the criminal.
 

(8 marks)
6. DEOXYRIBONUCLEIC ACID (DNA) IDENTIFICATION ACT 2008, has been tabled to the Malaysian Parliament recently. The act is for the establishment of a Forensic DNA Databank Malaysia, the taking of DNA samples, forensic DNA analysis, the use of DNA profiles and information in relation thereto, and for matters connected therewith.
  - (a) At least two clauses in the act were strongly objected by the parliamentarians. Give your comments.
 

(5 marks)
  - (b) Elaborate on the significance of the act to be enacted in Malaysia and relate its relevance to you as a chemist and suitable tests to be performed in your laboratory.
 

( 15 marks)

7. (a) The Dangerous Drugs Act was passed in 1952, several amendments have taken place. The major reason for the changing, dropping and adding of certain clauses is to cope with the advancement of the knowledge of the clandestine operators. Comment on the statement and provide appropriate examples.

(5 marks)

- (b) Explain how to determine the relevant compounds in the sample seized from the premise suspected to produce illicit drugs for testifying in the court. Hint: create your hypothetical clandestine laboratory.

( 15 marks)

**TERJEMAHAN**

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**Arahan:-**

Jawab **LIMA** soalan sahaja.

Kertas soalan ini mengandungi **TUJUH** soalan dalam **DUA** Bahagian, **BAHAGIAN A DAN B**. Pilih sekurang-kurang **DUA** soalan daripada setiap bahagian.

Jawab setiap soalan pada muka surat yang baru.

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

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

**BAHAGIAN A: STATISTIK**

1. (a) Jelaskan perbezaan antara hipotesis nol dan hipotesis alternatif.

(4 markah)

- (b) Jika pentitratan berulang-ulang 5.00 mL 0.10 M HCl dengan 0.10 M NaOH, keputusan yang didapati adalah tertabur secara normal dengan purata 5.00 mL dan sisihan piawai 0.04 mL NaOH. Berapakah kebarangkalian pentitratan yang akan memberikan isipadu NaOH

- (i) melebihi 4.95 mL?  
(ii) diantara 4.90 dan 5.05 mL?

(6 markah)

- (c) Kandungan fosforus telah ditentukan dalam tiga sampel tanah yang berbeza lokasinya. Lima kali penentuan telah dibuat bagi setiap sampel tanah itu. Jadual ANOVA separa telah didapati seperti di bawah.

Punca variasi	Jumlah kuasa dua	Darjah kebebasan	Purata kuasa dua	Nilai F
Antara tanah				
Dalam tanah			0.0081	
Total	0.374			

- (i) Isikan kesemua ruang yang diperlukan dalam jadual ANOVA di atas.  
(ii) Nyatakan hipotesis nol dan hipotesis alternatif.  
(iii) Adakah ketiga-tiga sampel tanah berbeza dalam kandungan fosforus pada paras keyakinan 95 %?

(10 markah)

2. (a) Penentuan sulfida dengan kaedah elektrod pemilih ion daripada bakteria penurun sulfat telah dibandingkan dengan kaedah gravimetri. Keputusan yang didapati dinyatakan dalam miligram sulfida.

No.	mg sulfida	
	Kaedah ISE	Kaedah gravimetri
1	108	105
2	12	16
3	152	113
4	3	0
5	106	108
6	11	11
7	128	141
8	12	11
9	160	182
10	128	118



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- (i) Tentukan persamaan garis regresi kuasa dua terkecil bagi menganggarkan nilai kaedah gravimetri (Y) daripada nilai kaedah ISE (X).
- (ii) Anggarkan nilai 95 % had keyakinan kecerunan dan perpotongan.
- (iii) Berikan ulasan tentang kesesuaian kaedah ISE bagi penentuan sulfida.

Diberikan:

Anggaran ralat rawak dalam arah-y:

$$S_{Y/X} = \sqrt{\frac{\sum (Y_i - \hat{Y}_i)^2}{n - 2}}$$

Sisihan piawai kecerunan:

$$S_b = \frac{S_{Y/X}}{\sqrt{\sum (X_i - \bar{X})^2}}$$

Sisihan piawai perpotongan:

$$S_a = S_{Y/X} \sqrt{\frac{\sum X_i^2}{n \sum (X_i - \bar{X})^2}}$$

(10 markah)

- (b) Di bawah kawalan statistik, satu proses menghasilkan debu yang mengandungi purata kandungan Fe adalah 6.70 % (w/w) dengan sisihan piawai adalah 0.18 %. Sebanyak 4 sampel dianalisis secara rutin setiap minggu oleh makmal kawalan mutu. Keputusan di bawah telah diperolehi bagi purata peratus Fe:

6.68, 6.71, 6.68, 6.72, 6.82, 6.54, 6.59, 6.58, 6.71, 6.48, 6.57, 6.61, 6.64, 6.56, 6.69, 6.64, 6.69, 6.57, 6.54, 6.56, 6.55, 6.50, 6.55, 6.51, 6.42.

Lakarkan carta kawalan Shewhart bagi keputusan di atas dan berikan ulasan mengenai carta yang diperolehi.

(10 markah)

3. (a) Seorang pembeli telah membeli dua tangki kereta berisi hasil amina. Jumlah nitrogen yang dianalisis disenaraikan seperti di bawah. Berdasarkan ujian hipotesis, adakah kedua-dua tangki kereta itu mempunyai kandungan jumlah nitrogen yang sama pada  $P = 0.05$ ?

% Jumlah Nitrogen	
Kereta 1	Kereta 2
9.23	9.18
9.28	9.01
9.17	9.09
9.24	9.20
9.20	9.00
9.24	
9.19	
9.21	

(8 markah)

- (b) Empat makmal (A – D) telah melakukan lima penentuan kandungan etil asetat ke atas satu sampel dari satu bekas yang sama. Peratus kandungan etil asetat yang didapati oleh makmal berkenaan di senaraikan seperti di bawah. Berikan penjelasan berhubung dengan purata kandungan dari makmal-makmal berkenaan pada aras keyakinan 95 %.

	Peratus etil asetat (%)			
	Makmal			
	A	B	C	D
Penentuan				
1	73	74	68	71
2	75	74	69	72
3	73	75	69	72
4	75	74	70	71
5	73	74	69	73

(12 markah)

4. (a) Terangkan apakah dia keluk taburan normal dan bagaimana ianya dapat dihubungkan dengan ralat? Terangkan juga secara ringkas perbezaan antara taburan normal dengan taburan normal terpiawai.

(6 markah)

- (b) Empat larutan piawai telah disediakan dan setiap larutan ini dikira mengandungi 16.00 % w/w klorida. Tiga kaedah pentitratan (A, B dan C) digunakan untuk menganalisis setiap larutan piawai dan takat akhir setiap larutan ini ditentukan dengan kaedah yang berlainan. Tertib eksperimen adalah secara rawak. Keputusan kandungan klorida yang diperolehi ditunjukkan di bawah.

Kaedah	Peratus Cl <sup>-</sup> (% w/w)			
	Larutan 1	Larutan 2	Larutan 3	Larutan 4
A	16.03	16.05	16.02	16.12
B	16.13	16.13	15.94	15.97
C	16.09	16.15	16.12	16.10

Uji sama ada terdapat perbezaan yang bermakna antara

- (i) kepekatan klorida dalam larutan yang berlainan, dan
- (ii) kepekatan klorida yang didapati dengan menggunakan kaedah yang berbeza pada aras keertian 5 %.

(14 markah)

### **BAHAGIAN B: FORENSIK**

5. (a) Ahli kimia forensik membandingkan keadaan penulisan yang jelas nampak dan meragukan pada dokumen yang menjadi persoalan dengan keadaan tulisan dari contoh tulisan bagi mendapatkan gabungan kesamaan dan perbezaan yang ketara. Komen kenyataan ini.

(5 markah)

- (b) Terangkan langkah-langkah yang diambil oleh ahli kimia forensik bagi melaporkan samada tandatangan didokumen tersebut asli atau tiruan dan siapakah yang menulis dan menandatangani dokumen tersebut.

(7 markah)

- (c) Cap jari mempunyai pola 'loop' yang dominan didalam 65 peratus penduduk, sedangkan pola 'whorl' pula dominan didalam 30 peratus penduduk, sedangkan pola 'arch' pula 5 peratus. Tambahan kepada pola tersebut, terdapat beberapa minutiae yang juga unik kepada pemunya jari. Terangkan dengan jelas bagaimana mendapatkan pola capjari daripada tempat jenayah yang dapat dikaitkan dengan penjenayah.

(8 markah)

6. Akta Identifikasi Asid Deoksiribonukleik (DNA) 2008, telah dibentangkan kepada Parlimen Malaysia baru-baru ini. Akta tersebut adalah untuk menubuhkan Bankdata DNA Malaysia, mengambil sampel DNA, analisis forensik DNA, penggunaan profil DNA dan maklumat berkaitan dengan nya dan juga kejadian yang dapat dikaitkan.
- (a) Sekurang-kurangnya dua cerai yang dibantah keras oleh ahli parlimen. Berikan komen anda.  
(10 markah)
- (b) Terangkan dengan jelas pentingnya akta tersebut dikuatkuasa di Malaysia dan kaitkan relevannya kepada anda sebagai ahli kimia dan ujian-ujian sesuai untuk tujuan tersebut dimakmal anda.  
(10 markah)
7. (a) Sejak Akta Dadah Merbahaya diluluskan pada tahun 1952, berbagai perubahan berlaku. Sebab utama, penambahan dan pengurangan cerai tertentu adalah untuk mengatasi kemajuan pengetahuan pengusaha haram. Komen kenyataan ini dengan contoh bersesuaian.  
(5 markah)
- (b) Terangkan bagaimana menentukan sebatian yang relevan didalam sampel yang dirampas daripada tempat yang disyaki menghasilkan dadah haram untuk kesaksian dimahkamah. Bantuan: janakan makmal haram bayangan anda sendiri.  
(15 markah)