
UNIVERSITI SAINS MALAYSIA

Final Examination
2015/2016 Academic Session

May/June 2016

JIK 226 – Analytical Chemistry I
[Kimia Analitis I]

Duration : 3 hours

[Masa : 3 jam]

Please ensure that this examination paper contains **THIRTEEN** printed pages before you begin the examination.

Answer **FIVE** questions. Answer the questions in English. You may also answer the questions in Bahasa Malaysia, but not a mix of both languages.

All answers must be written in the answer booklet provided.

Each question is worth 20 marks and the mark for each sub question is given at the end of that question.

In the event of any discrepancies in the exam questions, the English version shall be used.

*Sila pastikan bahawa kertas peperiksaan ini mengandungi **TIGA BELAS** muka surat yang bercetak sebelum anda memulakan peperiksaan ini.*

*Jawab **LIMA** soalan. Jawab soalan-soalan dalam Bahasa Inggeris. Anda juga dibenarkan menjawab soalan dalam Bahasa Malaysia, tetapi campuran antara kedua-dua bahasa ini tidak dibenarkan.*

Setiap jawapan mesti dijawab di dalam buku jawapan yang disediakan.

Setiap soalan bernilai 20 markah dan markah subsoalan diperlihatkan di penghujung subsoalan itu.

Sekiranya terdapat sebarang percanggahan pada soalan peperiksaan, versi Bahasa Inggeris hendaklah diguna pakai.

Answer any **FIVE** questions.

Jawab mana-mana LIMA soalan.

1. (a) Explain the **DIFFERENCES** in the pair of concepts/items given below. Use appropriate diagrams/equations/formulas/examples if needed.
- (i) Standard deviation and relative standard deviation
 - (ii) Q-test and *t*-test
 - (iii) Mean value and certified value of an ion in a standard solution
 - (iv) Significant figures and significant difference
 - (v) Osmosis and dialysis processes

Terangkan PERBEZAAN dalam pasangan konsep / perkara yang diberikan di bawah. Gunakan gambar rajah / persamaan / formula / contoh yang sesuai jika diperlukan.

- (i) *Sisihan piawai dan sisihan piawai relatif*
- (ii) *Ujian Q dan Ujian-t*
- (iii) *Nilai min dan nilai yang disahkan untuk suatu ion dalam larutan piawai*
- (iv) *Angka bererti dan perbezaan bererti*
- (v) *Proses osmosis dan dialisis*

(10 marks/markah)

- (b) A student was conducting a study on the concentration level of Cd in water samples collected from a factory waste effluent over a two-day period. There was no significant change in weather conditions over the two day period. Atomic emission spectroscopy was used to determine the Cd concentration in the samples:

Seorang pelajar telah menjalankan suatu kajian tentang tahap kepekatan Cd dalam sampel air yang diambil dari efluen sisa kilang dalam tempoh dua hari. Tidak ada perubahan ketara dalam keadaan cuaca dalam tempoh dua hari berkenaan. Spektroskopi pemancaran atom telah digunakan untuk menentukan kepekatan Cd dalam sampel-sampel tersebut:

Sample No.	Cd level (ppm)	Sample No.	Cd level (ppm)
1000	6.3	1016	9.7
1002	7.8	1018	9.5
1004	8.9	1020	8.8
1006	8.4	1022	7.9
1008	9.0	1024	8.6
1010	9.2		
1012	9.3		
1014	9.9		

- (i) Calculate the average Cd level contained in the waste effluent.
- (ii) Calculate the standard deviation and relative standard deviation of the determination.
- (iii) The Cd level in sample no. 1000 appeared to be off compared to the other 12 samples. Should that be value be retained or rejected? Justify your answer.

- (i) *Kirakan min/purata tahap Cd yang terkandung dalam efluen sisa buangan itu.*
- (ii) *Kira sisihan piawai dan sisihan piawai relatif untuk penentuan itu*
- (iii) *Tahap Cd dalam sampel no. 1000 kelihatan seperti tersasar berbanding dengan 12 sampel yang lain. Wajarkah nilai itu dikekalkan atau dikeluarkan? Beri justifikasi kepada jawapan anda.*

(10 marks/markah)

2. (a) Describe the differences between

- (i) Gas solid chromatography and gas liquid chromatography

(4 marks)

- (ii) Open tubular column and packed column

(8 marks)

Terangkan perbezaan antara

- (i) *kromatografi pepejal gas dan kromatografi cecair gas*

(4 markah)

- (ii) *turus tubul terbuka dan turus padat*

(8 markah)

(b) (i) State FOUR examples of carrier gas

- (ii) Based on the answers in b(i), discuss about the carrier gases in gas chromatography.

(8 marks)

- (i) *Nyatakan EMPAT contoh gas pembawa*

- (ii) *Berdasarkan jawapan di b(i), bincangkan tentang gas-gas pembawa dalam kromatografi gas.*

(8 markah)

3. (a) Define

- (i) elution
- (ii) eluate
- (iii) eluent

Takrifkan

- (i) *elusi*
- (ii) *eluat*
- (iii) *eluen*

(6 marks/markah)

(b) State ALL aspects that need to be considered for selecting a column.

Nyatakan SEMUA aspek yang perlu dipertimbangkan dalam memilih sebatang turus.

(4 marks/markah)

(c) Discuss briefly about guard column and analytical column.

Bincangkan secara ringkas turus pengawal dan turus analitikal.

(5 marks/markah)

- (d) Figure 1 shows the HPLC chromatogram of mixture of various nucleosides. The information of chromatogram is listed in Table 1.

Rajah 1 menunjukkan kromatogram HPLC bagi campuran pelbagai nukleotida. Maklumat dalam kromatogram disenaraikan dalam Jadual 1.

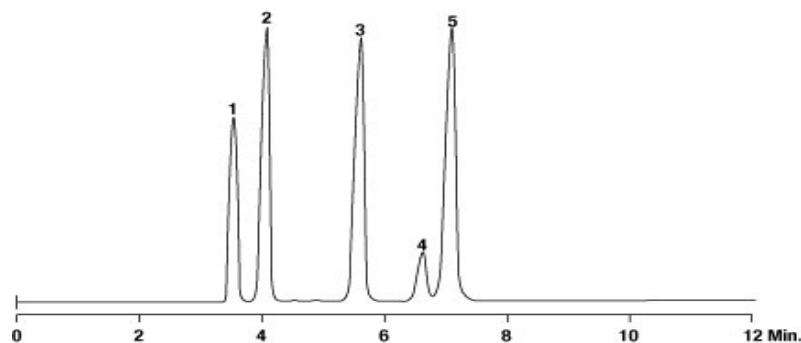


Figure 1: HPLC chromatogram of mixture of nucleosides

Table 1 - Retention time and peak width information of mixture of nucleosides

Peak identification	Retention time, min	Peak width, min
1. Cytidine	3.87	0.94
2. Uridine	4.06	1.05
3. Xanthosine	5.63	1.30
4. Guanosine	6.75	1.53
5. Adenosine	7.25	1.65

The resolution between the guanosine and adenosine is relatively low. Suggest THREE adjustments on HPLC system in order to improve the separation efficiency between guanosine and adenosine.

Resolusi antara guanosina dan adenosina agak rendah. Cadangkan TIGA pelarasan pada sistem HPLC bagi meningkatkan kecekapan pemisahan antara guanosina dan adenosina.

(5 marks/markah)

4. (a) Explain the benefits of using sodium dodecyl sulphate (SDS) in PAGE.
Terangkan faedah-faedah menggunakan sodium dodesil sulfat (SDS) dalam PAGE.
- (5 marks/markah)
- (b) Describe the concept of one-dimensional SDS-PAGE in the separation process in vaccine production.
Terangkan konsep satu dimensi SDS-PAGE dalam proses pemisahan bagi pengeluaran vaksin.
- (5 marks/markah)
- (c) Based on Figure 2 below:
- (i) Name the separation method, (1 marks)
- (ii) Based on the answer in c(i), discuss the principles. (9 marks)

Berdasarkan Rajah 2 di bawah:

- (i) *Namakan kaedah pemisahan tersebut,* (1 markah)
- (ii) *Berdasarkan jawapan di c(i), bincangkan prinsipnya.* (9 markah)

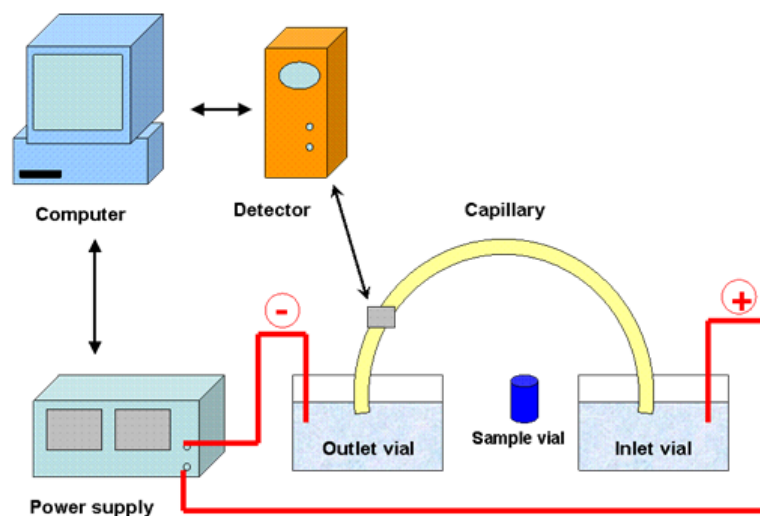


Figure 2 - Adapted from <http://theses.ulaval.ca/archimede/fichiers/24330/apa.html>

5. (a) A commercially available standard solution is been certified to contain 5.00 ppm Zn. In a laboratory exercise, five students were asked to determine the Zn concentration in that solution using atomic absorption spectroscopic technique (AAS). Another five students were asked to determine the Zn concentration using flame atomic emission spectroscopic technique (AES). The results obtained are given below:

Suatu larutan piawai yang boleh diperolehi secara komersial disahkan mengandungi 5.00 ppm Zn. Dalam satu latihan makmal, lima pelajar diminta untuk menentukan kepekatan Zn dalam larutan itu menggunakan teknik spektroskopi penyerapan atom (AAS). Lima pelajar lain diminta menentukan kepekatan Zn itu menggunakan teknik spektroskopi pemancaran atom (AES). Keputusan yang diperolehi diberikan di bawah:

	Zn concentration, via AAS		Zn concentration, via AES
Student 1	4.10	Student 6	5.85
Student 2	4.16	Student 7	6.05
Student 3	4.02	Student 8	5.85
Student 4	4.02	Student 9	6.15
Student 5	4.02	Student 10	5.95

By using an appropriate statistical test, show whether

- (i) the Zn concentration levels obtained via AAS and
- (ii) the Zn concentration levels obtained via AES are significantly different (or NOT significantly different) from the certified value.

Dengan menggunakan ujian statistik yang sesuai, tunjukkan sama ada

- (i) *tahap kepekatan Zn yang diperolehi melalui AAS dan*
 (ii) *tahap kepekatan Zn yang diperolehi melalui AES berbeza dengan ketara (atau TIDAK berbeza dengan ketara) daripada nilai yang disahkan.*

(10 marks/markah)

- (b) A student was conducting a study on the concentration level of Cr in water samples collected from a factory waste effluent from January 2nd. - January 4th. 2015. Another student was conducting the same study from January 9th. – January 10th. 2015. Atomic absorption spectroscopy was used by both students to determine the Cr concentration:

Seorang pelajar telah menjalankan kajian mengenai tahap kepekatan Cr dalam sampel air dari efluen sisa satu kilang mulai 2 Januari - 4 Januari 2015. Seorang lagi pelajar telah menjalankan kajian yang sama dari 9 Januari - 10 Januari 2015. Spektroskopi penyerapan atom telah digunakan oleh kedua-dua pelajar untuk menentukan kepekatan Cr:

Sample Taken on 2 - 3 Jan.	Cr level (ppm)	Sample Taken on 9-10 Jan.	Cr level (ppm)
1a	2.5	1b	4.3
2a	2.8	2b	3.5
3a	3.1	3b	3.8
4a	2.1	4b	4.2
5a	3.0	5b	3.8
6a	3.2	6b	4.0

- (i) Calculate the average Cr level contained in the waste effluent for both studies.
 - (ii) Calculate the standard deviation and relative standard deviation of the determinations.
 - (iii) Are Cr levels determined by both students on the two different dates significantly different? Justify your answer.
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- (i) *Kirakan min/purata Cr yang terkandung dalam sisa efluen untuk kedua-dua kajian itu.*
 - (ii) *Kira sisihan piawai dan sisihan piawai relatif bagi penentuan itu.*
 - (iii) *Adakah tahap Cr yang ditentukan oleh kedua-dua pelajar pada dua tarikh berlainan itu berbeza dengan ketara atau tidak? Beri justifikasi kepada jawapan anda.*

(10 marks/markah)

6. (a) A major function of human kidney is to clean the blood from and toxins and impurities. However, in cases where the kidneys have ceased to function effectively, haemodialysis is used to do the cleaning process. Explain in detail haemodialysis process using appropriate diagram and/or schematics.
- Fungsi utama buah pinggang manusia adalah untuk membersihkan darah dari toksin dan kotoran. Walau bagaimanapun, dalam kes-kes di mana buah pinggang telah berhenti berfungsi secara berkesan, haemodialisis digunakan untuk melakukan proses pembersihan. Terangkan secara terperinci proses haemodialisis menggunakan gambar rajah dan/atau skema yang sesuai.*

(10 marks/markah)

- (b) For the following two cases, propose the most appropriate analytical method/technique that can be used to handle the requirements. However, you are limited to the analytical method/techniques that you have learned in this course only namely gas chromatography, liquid chromatography, high performance liquid chromatography, capillary electrophoresis, extraction, precipitation and dialysis. You must justify the method's selection and provide the details of how the proposed method/techniques is used for the given sample analysis:
- (i) A large number of soil and sand samples obtained from various quarries need to be tested for significant presence of silver. Your testing laboratory has to analyse at least 500 samples and the result is needed within 48 hours.
 - (ii) Human blood samples need to be analysed for the presence of methyl alcohol, ethyl alcohol, cannabis and amphetamine (a stimulant). Typically at least 10 samples need to be analysed every day and the concentration levels of those compounds have to be obtained.

Bagi dua kes berikut, cadangkan satu kaedah/teknik analisis yang paling sesuai yang boleh digunakan untuk menangani keperluan tersebut. Walau bagaimanapun, kaedah anda adalah terhadap kepada kaedah/teknik analisis yang telah anda pelajari dalam kursus ini sahaja iaitu kromatografi gas, kromatografi cecair, kromatografi cecair berprestasi tinggi, kapilari elektroforesis, pengekstrakan, pemendakan dan dialisis. Anda dikehendaki memberi justifikasi pemilihan kaedah berkenaan dan berikan perincian bagaimana kaedah/teknik itu digunakan untuk menganalisis sampel yang diberi:

- (i) *Sejumlah besar sampel tanah dan pasir yang diperolehi daripada beberapa kuari perlu diuji untuk kehadiran ketara logam perak. Makmal ujian anda perlu menganalisa sekurang-kurangnya 500 sampel dan keputusan diperlukan dalam tempoh 48 jam.*

- (ii) *Sampel darah manusia perlu dianalisa untuk kehadiran metil alkohol, etil alkohol, kanabis dan amphetamina (sejenis perangsang). Sekurang-kurangnya 10 sampel biasanya perlu dianalisis setiap hari dan tahap kepekatan sebatian-sebatian itu perlu diperolehi.*

(10 marks/markah)

Table 1 – *t*-value for confidence interval calculation

No. of determination	<i>t</i> -value at various confidence intervals		
	80%	90%	95%
2	3.08	6.31	12.71
3	1.89	2.92	4.30
4	1.64	2.35	3.18
5	1.53	2.13	2.78
6	1.48	2.02	2.57
7	1.44	1.94	2.45
8	1.42	1.90	2.36
9	1.40	1.86	2.31
10 - 30	1.38	1.83	2.26

Table 2 – Q value for data rejection

No. of replicates	Q value (90% confidence)
3	0.94
4	0.76
5	0.64
6	0.56
7	0.51
8	0.47
9	0.44
10 – 30	0.41