



UNIVERSITI SAINS MALAYSIA

Final Examination
2016/2017 Academic Session

May/June 2017

JIK 226 – Analytical Chemistry I
[Kimia Analitis I]

Duration : 3 hours
[Masa : 3 jam]

Please ensure that this examination paper contains **ELEVEN** 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 **SEBELAS** 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) Briefly describe the following concepts/items, use appropriate diagrams/equations/formulas/examples if needed:
- (i) Relative standard deviation
 - (ii) Q-test in data analysis
 - (iii) Confidence level in *t*-test
 - (iv) Semi-permeable membrane used in dialysis
 - (v) Isocratic elution in high performance liquid chromatography

Terangkan dengan ringkas konsep / perkara berikut, gunakan gambar rajah/persamaan/formula/ccontoh-contoh yang sesuai jika perlu:

- (i) *Sisihan piawai relatif*
- (ii) *Ujian-Q dalam analisa data*
- (iii) *Tahap keyakinan dalam ujian-t*
- (iv) *Membran separa telap yang digunakan dalam dialisis*
- (v) *Elusi isokratik dalam kromatografi cecair berkeupayaan tinggi*

(10 marks/markah)

- (b) An environmental safety officer was conducting a study on the concentration level of lead (Pb) at a solid waste disposal site. Six samples were collected at one critical location in weekly intervals. An atomic absorption spectrometer was used in the determination. The data obtained for two weeks are as follow:

Seorang pegawai keselamatan persekitaran telah menjalankan suatu kajian mengenai tahap kepekatan plumbum (Pb) di suatu tapak pelupusan sisa pepejal. Enam sampel telah dikumpulkan di satu lokasi kritikal dalam selang seminggu. Suatu spektrometer penyerapan atom telah digunakan dalam penentuan itu. Data yang diperolehi selama dua minggu adalah seperti berikut:

Sample No. (Week 6/2015)	Pb level (ppb)	Sample No. (Week 7/2015)	Pb level (ppb)
6A	47.3	7A	42.7
6B	48.3	7B	44.2
6C	49.9	7C	45.7
6D	44.8	7D	45.0
6E	52.0	7E	46.1
6F	46.2	7F	44.8

- (i) Determine whether the Pb level for sample no. 7A can be accepted or should be rejected based on statistical consideration.
- (ii) Calculate the standard deviation and relative standard deviation of the Pb determination for the two weeks.
- (iii) By using an appropriate statistical test, determine whether there is a significant difference in the Pb concentration levels in those two weeks.
- (iv) Suggest a way to improve the precision of the determination.
- (i) *Tentukan sama ada tahap Pb bagi sampel no. 7A boleh diterima atau perlu dikeluarkan berdasarkan pertimbangan statistik*
- (ii) *Kira sisihan piawai dan sisihan piawai relatif untuk penentuan Pb dalam dua minggu itu.*

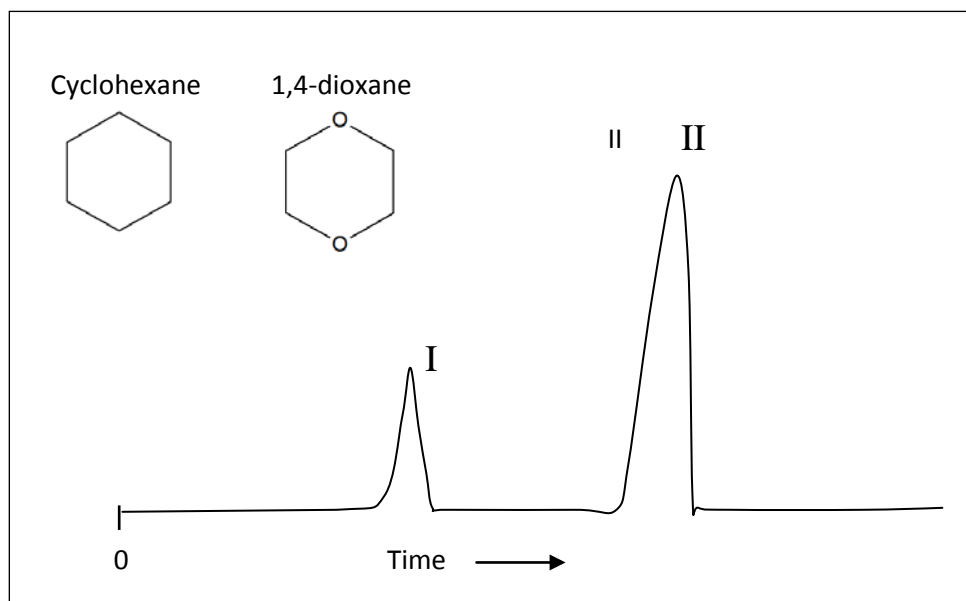
(iii) Dengan menggunakan ujian statistik yang sesuai, tentukan sama ada terdapat perbezaan yang ketara dalam tahap kepekatan Pb untuk kedua-dua minggu itu.

(iv) Cadangkan satu cara untuk memperbaiki kepersisan penentuan itu.

(10 marks/markah)

2. A mixture of cyclohexane and dioxane is eluted on a non-polar gas chromatography column. The chromatogram obtained is shown below:

Suatu campuran sikloheksana dan dioksana dielusikan dalam suatu turus kromatografi gas tidak berkutub. Kromatogram yang dihasilkan ditunjukkan di bawah:



(a) Define

(i) Non-polar column

(ii) Elution

(iii) Eluate

Takrifkan

(i) Turus tidak berkutub

(ii) Elusi

(iii) Eluat

(6 marks/markah)

- (b) Identify the compounds that are represented by peak I and peak II. Explain your answer.

Kenalpasti sebatian-sebatian yang diwakili oleh puncak I dan puncak II. Terangkan jawapan anda.

(4 marks/markah)

- (c) The area of peak I is measured as 3 cm^2 and the area measured for peak II is 7 cm^2 . The detector response is the same for both substances. Calculate the mol % of compound I in the mixture.

Keluasan puncak I diukur sebagai 3 cm^2 dan keluasan bagi puncak II ialah 7 cm^2 . Gerak balas pengesanan adalah sama bagi kedua-dua sebatian. Kira % mol sebatian I dalam campuran berkenaan.

(2 marks/markah)

- (d) Differentiate between open tubular column and packed column.

Bezakan antara turus tubul terbuka dan turus padat.

(8 marks/markah)

3. (a) An HPLC analysis was conducted for the determination of ascorbic acid in a syrup drink. A 9.5 ppm acetonitrile internal standard was introduced both into the sample and a standardised solution of 205 ppm of ascorbic acid. Diode-array detector was used at each λ_{\max} for the absorptions of both and the information are shown in the table below.

Satu analisa HPLC telah dijalankan untuk penentuan asid askorbik dalam minuman sirap. Asetonitril sebagai piawai dalaman yang berkepekatan 9.5 ppm telah dimasukkan ke dalam sampel dan larutan piawai yang mengandungi 205 ppm asid askorbik. Pengesan diod tatasusunan digunakan pada setiap penyerapan λ_{\max} bagi kedua-duanya dan maklumat berkaitan ditunjukkan dalam jadual berikut:

	Internal standard	Ascorbic acid
Sample	20242	56899
205 ppm ascorbic acid standard	27556	78914

- (i) Explain the functions of internal standard in the analysis.
(ii) List the types of detectors used in HPLC besides diode-array.
(iii) Calculate the concentration of ascorbic acid in that syrup drink.

- (i) *Jelaskan fungsi piawai dalaman bagi analisis ini.*
(ii) *Senaraikan jenis pengesan yang digunakan dalam HPLC selain diod tatasusunan.*
(iii) *Kira kepekatan asid askorbik dalam minuman sirap tersebut.*

(15 marks/markah)

- (b) Predict the order of elution from an HPLC column packed with C₁₈ hydrocarbon for acetamide, 1,2-dichloroethane and acetone. Justify your answer.

Ramalkan tertib elusi daripada HPLC yang berturus padat dengan hidrokarbon C₁₈ untuk asetamida, 1,2-dikloroetana dan aseton. Beri kewajaran jawapan anda itu.

(5 marks/markah)

4. (a) Isoelectric focusing separates proteins according to the net charge of the protein.

- (i) Briefly describe the basis of separation.
 (ii) Define isoelectric point.
 (iii) Describe the mechanism of determining the protein charge.

Pemfokusan isoelektrik mengasingkan protein mengikut cas bersih protein tersebut.

- (i) *Jelaskan dengan ringkas tentang asas pengasingan.*
 (ii) *Takrifkan titik isoelektrik.*
 (iii) *Jelaskan mekanisme bagi penentuan cas protein.*

(10 marks/markah)

- (b) The determination of relative molecular weight of a protein can be analysed using electrophoresis. Describe the principles behind:

- (i) 1D
 (ii) 2D gel electrophoresis of proteins.

Penentuan jisim molekul relatif suatu protein boleh dianalisa melalui elektroforesis. Jelaskan prinsip di sebalik:

- (i) 1D
 (ii) elektroforesis gel protein 2D.

(10 marks/markah)

5. (a) Separation of many species using extraction is generally preferred over precipitation. Discuss two factors that lead to that preference.

Pengasingan banyak spesies menggunakan pengekstrakan secara amnya lebih diutamakan berbanding kaedah pemendakan. Bincangkan dua faktor yang membawa kepada keutamaan itu.

(5 marks/markah)

- (b) Liquid-solid extraction has been used for many extraction processes involving solid samples.

- (i) Draw an apparatus that can be used for continuous liquid-solid extraction process. Label all the parts and clearly indicate where the solid sample and solvent are placed.
- (ii) Discuss two advantages of liquid-solid extraction.

Pengekstrakan cecair-pepejal telah digunakan untuk banyak proses pengekstrakan melibatkan sampel pepejal.

- (i) *Lukiskan suatu radas yang boleh digunakan untuk proses pengekstrakan cecair-pepejal secara berterusan. Labelkan semua bahagian dan tunjukkan dengan jelas tempat di mana sampel pepejal dan pelarut diletakkan.*
- (ii) *Bincangkan dua kelebihan pengekstrakan cecair-pepejal.*

(10 marks/markah)

(c) Read the following excerpt:

Baca petikan di bawah:

*“Lithium is found in trace amounts in many places, but it is being produced commercially mainly by two methods. One is through mining and processing, a relatively expensive method that produces the metal used mostly for manufacturing glass, ceramics and batteries. The more economical and significant method is through **evaporation** of lithium-containing brines (saturated solutions), found mostly in salt flats in the highland areas in South America and western China. Lithium reservoirs have been formed over millions of years in highland bowls, after rivers and hot springs washed over lithium-laden rocks and leached the mineral from them. Producers drill wells into the salt flats and pump the brine into evaporation ponds. With the removal of water, the lithium content in the brine in the form of **lithium carbonate** increases to a level where it can be collected and shipped to a chemical plant for processing.*

As you can see from the above excerpt, a separation process doesn't have to be very complicated. Propose a way to get lithium metal from lithium carbonate.

Seperti yang anda boleh lihat dari petikan di atas, suatu proses pemisahan tidaklah begitu rumit. Cadangkan suatu cara untuk mendapatkan logam litium daripada litium karbonat.

(5 marks/markah)

6. (a) Describe in detail the process involved in the extraction of caffeine from an aqueous solution containing the compound. Your description must include the following:

- (i) the steps involved in the extraction process including sample preparation
- (ii) the apparatus used
- (iii) solvent(s) used, and
- (iv) problems anticipated in the process and ways to solve those problems.

Terangkan secara terperinci proses yang terlibat dalam pengekstrakan kafein dari suatu larutan akueus yang mengandungi sebatian tersebut. Huraian anda hendaklah meliputi perkara-perkara berikut:

- (i) langkah-langkah yang terlibat dalam proses pengekstrakan termasuk penyediaan sampel*
- (ii) radas yang digunakan*
- (iii) pelarut(-pelarut) yang digunakan, dan*
- (iv) masalah yang dijangka berlaku dalam proses dan cara-cara untuk menyelesaikan masalah itu.*

(10 marks/markah)

(b) A major function of human kidney is to clean the blood from 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 the haemodialysis process using appropriate diagram and/or schematics.

Suatu fungsi utama buah pinggang manusia adalah untuk membersihkan darah daripada toksin dan kotoran. Walau bagaimanapun, dalam kes-kes yang mana buah pinggang telah berhenti berfungsi dengan berkesan, hemodialisis digunakan untuk melakukan proses pembersihan. Terangkan secara terperinci proses hemodialisis menggunakan gambar rajah dan/atau skema yang sesuai.

(10 marks/markah)

Table 1 - *t*-values for various confidence intervals

No. of determination	<i>t</i> -value at various confidence intervals			
	80%	90%	95%	99%
2	3.08	6.31	12.71	63.66
3	1.89	2.92	4.30	9.93
4	1.64	2.35	3.18	5.84
5	1.53	2.13	2.78	4.60
6	1.48	2.02	2.57	4.03
7	1.44	1.94	2.45	3.71
8	1.42	1.90	2.36	3.45
9	1.40	1.86	2.31	3.36
10 - 30	1.38	1.83	2.26	3.25

Table 2 - Q values 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