
UNIVERSITI SAINS MALAYSIA

Second Semester Examination
2009/2010 Academic Session

April/May 2010

KAT 244 – Separation Methods
[Kaedah Pemisahan]

Duration : 3 hours
[Masa : 3 jam]

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

Instructions:-

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

Answer to 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.

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

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1. (a) Using schematic presentations, explain what happens when two pure immiscible solvents are in contact in the presence and in the absence of the solute molecules. Choose your own compound as a model.
- (10 marks)
- (b) Solid phase extraction is considered a bridging technique between solvent extraction and chromatographic technique, while counter current extraction is viewed to be a precursor to the chromatography. Explain.
- (10 marks)
2. (a) The partition isotherm is not always ideal. Show all possible isotherms and the conditions that cause deviations and suggest how to overcome.
- (10 marks)
- (b) Five grams of compound A is dissolved in 90 mL of water. The partition coefficient for compound A between chloroform and water is 15.
- (i) How much of compound A will be in chloroform if you extract it from water once with 90 mL of chloroform?
- (ii) How much of compound A will be in chloroform if you extract it from water with three sequential extractions using 30 mL of chloroform each time, and then combine the chloroform extracts?
- (iii) Suggest an economical way and safety in handling for optimum extraction with zero risk.
- (10 marks)

3. (a) Discuss the basics in the separation of closely related compounds by solvent extraction and chromatographic methods. Compare and contrast between the two methods.
- (10 marks)
- (b) Secondary chemical equilibrium are important in solvent extraction and also in reversed phase liquid chromatography. Discuss about this equilibrium and explain how could they be utilized to enhance the selectivity in the separation by both techniques.
- (10 marks)
4. (a) In reversed phase high performance liquid chromatography, HPLC, ion pairing reagents can be used to facilitate retention of ionic substances or substances that undergo protonation or dissociation within the experimental pH range.
- (i) How is ion pair chromatography (IPC) carried out?
 - (ii) What major factors affect the retention?
 - (iii) Compare ion pairing technique to ion chromatography and conventional RP-HPLC (reversed phase HPLC). What are the advantages?
- (10 marks)
- (b) What types of analytes have ion pair chromatography become particularly important for? What are the prerequisites for the successful use of ion pair chromatography with these compounds?
- (10 marks)

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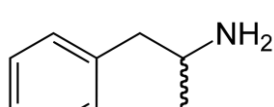
5. (a) Detectors in chromatography can be classified into universal, selective and specific. Describe these terms. Give an example of a detector for each of the mentioned classes. Suggest a sample/analyte for which each selected detector is best suited for. Justify your choice.

(10 marks)

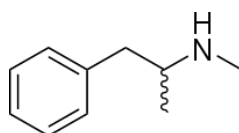
- (b) Propose your solution to quantitatively determine a mixture of two selected compounds via external standard and internal standard method. Then describe the implications and advantages of using these standard addition methods.

(10 marks)

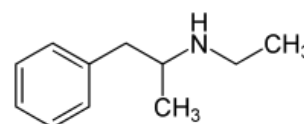
6. The use of narcotic substances such as amphetamine (I), methamphetamine (II) and ethylamphetamine (III) illegally has become a challenging task to the clinical and forensic laboratories. A common way of monitoring the abuse of these substances is through sensitive quantitation of urine samples.



(I)



(II)



(III)

- (i) Describe a suitable procedure to determine these substances using gas chromatography. Discussion should include: Sample preparation, internal standard, injection mode, detection principle, etc.
- (ii) Describe briefly at least one alternative technique for the separation of these substances?

(20 marks)

7. Suggest at least one appropriate set of chromatographic separation techniques that can be used to solve the following analytical problems. Describe both the sampling and the sample clean-up/enrichment procedures, a suitable analytical separation technique, and a detection principle. Choose any two of the following.
- (i) Suspected leakage of petroleum products from a petrol station into a ground drinking water reservoir.
 - (ii) Drug metabolites, in human serum.
 - (iii) Ethanol in human whole blood.
 - (iv) Butylacetate and xylene in workplace air in a car painting facility.
 - (v) Polyaromatic hydrocarbons from diesel exhaust in a bus garage.

(20 marks)

TERJEMAHAN

Jawab **LIMA** soalan sahaja.

Jawab tiap-tiap 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.

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

1. (a) Secara skematik terangkan apakah yang akan terjadi apabila dua pelarut yang tak bercampur bersentuhan dengan atau tanpa kehadiran molekul solut.
- (10 markah)
- (b) Pengekstrakan fasa pepejal disifatkan sebagai teknik penghubung diantara pengekstrakan pelarut dan kromatografi, sedangkan pengekstrakan arus lawan dilihat sebagai pemula kepada kromatografi. Terangkan.
- (10 markah)
2. (a) Isoterma pembahagian tidak selalunya ideal. Tunjukkan semua isoterma yang mungkin dan keadaan yang menyebabkan penyimpangan dan cadangkan bagaimana dielakkan.
- (10 markah)
- (b) Lima gram sebatian A dilarutkan didalam 90 mL air. Pekali pembahagian bagi sebatian A diantara kloroform dan air adalah 15.
- (i) Berapa banyak sebatian A yang akan berada didalam kloroform jika anda mengekstraknya daripada air sekali dengan 90 mL kloroform?
- (ii) Berapa banyak sebatian A yang akan berada di dalam kloroform jika anda mengekstraknya di dalam air dengan secara tiga kali berturutan menggunakan kloroform 30 mL setiap kali, dan kemudian satukan ekstrak kloroform?
- (iii) Cadangkan jalan yang menjimatkan dan selamat dalam pengendalian bagi pengekstrakan optimum tanpa risiko.
- (10 markah)

3. (a) Bincangkan asas-asas pemisahan bagi sebatian yang hampir serupa dengan kaedah pengekstrakan pelarut dan kromatografi. Banding dan bezakan di antara kedua-dua kaedah.

(10 markah)

- (b) Keseimbangan kimia sekunder adalah penting didalam pengestrakan dan kromatografi cecair fasa terbalik. Bincangkan mengenai keseimbangan ini dan terangkan bagaimana dapat dimanfaatkan untuk meningkatkan kepilihan pemisahan bagi kedua-dua teknik.

(10 markah)

4. (a) Didalam kromatografi cecair prestasi tinggi, HPLC, reagen pasangan ion dapat digunakan bagi membolehkan penahanan sebatian ion atau sebatian yang mengalami pemprotonan atau perceraian di dalam julat pH eksperimen.

(i) Bagaimana kromatografi ion (IPC) di laksanakan?

(ii) Apakah faktor utama yang mempengaruhi penahanan?

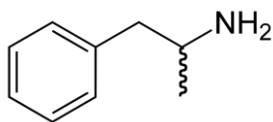
(iii) Bandingkan teknik pasangan ion dengan kromatografi ion biasa, RP-HPLC (HPLC- fasa terbalik). Apakah kelebihanannya?

(10 markah)

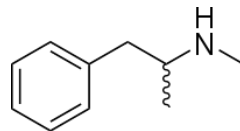
- (b) Apakah jenis analit yang membuat kromatografi pasangan ion menjadi penting?

(10 markah)

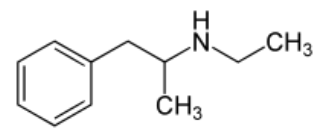
5. (a) Pengesanan kromatografi dikelaskan kepada universal, kepilahan dan spesifik. Terangkan istilah-istilah tadi. Berikan contoh satu pengesanan bagi setiap kelas yang dinyatakan. Cadangkan satu sampel/analit yang mana pengesanan yang dipilih adalah bersesuaian. Beri alasan kukuh terhadap pilihan anda. (10 markah)
- (b) Cadangkan penyelesaian anda bagi penentuan kuantitatif suatu campuran dua sebatian yang dipilih melalui kaedah piawai luaran dan dalaman. Seterusnya terangkan implikasi dan kelebihan menggunakan kaedah penambahan piawai tersebut. (10 markah)
6. Penggunaan bahan narkotik seperti amfetamina (I), metamfetamina (II) dan etilamfetamina (III) secara haram merupakan tugas mencabar kepada makmal klinikal dan forensik. Cara yang biasa digunakan bagi mengawasi penyalahgunaan bahan tersebut adalah melalui pengkuantitatifan yang peka terhadap sampel air kencing.



(I)



(II)



(III)

- (i) Terangkan tatacara yang sesuai bagi menentukan bahan-bahan tersebut dengan kromatografi gas. Perbincangan mestilah mengandungi: Penyediaan sampel, piawai dalaman, mod penyuntikan, asas pengesanan dll.
- (ii) Secara ringkas terangkan teknik alternatif bagi pemisahan bahan tersebut.

(20 markah)

7. Cadangkan sekurang-kurangnya satu set teknik pemisahan kromatografi yang sesuai digunakan bagi menyelesaikan masalah analisis berikut. Terangkan tatacara pensampelan dan pembersihan/pengkayaan, teknik pemisahan analisis bersesuaian dan prinsip pengesanan. Pilih dua yang berikut.
- (i) Kemungkinan kebocoran produk petroleum daripada stesen minyak ke dalam tadahan air minum bawah tanah.
 - (ii) Metabolit dadah didalam serum manusia.
 - (iii) Etanol di dalam darah manusia.
 - (iv) Butilasetat dan xilena di udara tempat kerja suatu kemudahan mengecat kereta.
 - (v) Hidrokarbon poliaromatik daripada ekzos enjin disel di dalam garaj bas.

(20 markah)