
UNIVERSITI SAINS MALAYSIA

Second Semester Examination
Academic Session 2006/2007

April 2007

KAT 244 – Separation Methods
[Kaedah Pemisahan]

Duration: 3 hours
[Masa: 3 jam]

Please check that this examination paper consists of EIGHT printed material before you begin the examination.

Read all instructions carefully before you begin.

Answer any **FIVE** questions.

Answer to each question on a new page.

Only the first FIVE questions answered in the answer book will be marked.

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

...2/-

1. (a) What are (i) accelerated solvent extraction and (ii) microwave-assisted extraction?
(5 marks)
- (b) The distribution ratio between 3 M HCl and tri-n-butylphosphate for PdCl_2 is 2.3. What percent PdCl_2 will be extracted from 25.0 mL of a 7.0×10^{-4} M solution into 10.0 mL tri-n-butylphosphate?
(5 marks)
- (c) Ninety percent of a metal chelate is extracted when equal volumes of aqueous and organic phases are used. What will be the percent extracted if the volume of the organic phase is doubled?
(5 marks)
- (d) Arsenic(III) is 70% extracted from 7 M HCl into an equal volume of toluene. What percentage will remain unextracted after three individual extractions with toluene?
(5 marks)
2. (a) A gas chromatographic peak had a retention time of 65 s. The base width obtained from intersection of the baseline with the extrapolated sides of the peak was 5.5 s. If the column was 1 m in length, what was the height, H, in cm plate^{-1} ?
(4 marks)
- (b) Describe the principles of the following gas chromatography (GC) detectors:
(i) Thermal conductivity.
(ii) Flame ionization.
(iii) Electron capture.
Compare the above detectors with respect to sensitivity and types of compounds that can be detected.
(9 marks)
- (c) What is the van Deemter equation? Define terms. How do the Huber and Knox equations differ from the van Deemter equation?
(7 marks)
3. (a) It is desired to just resolve two gas chromatographic peaks with retention times of 85 and 100 s, respectively, using a column that has an H value of $1.5 \text{ cm plate}^{-1}$ under the operating conditions. What is the length of the columns required? Assume the two peaks have the same base width.
(6 marks)
...3/-

- (b) Differentiate wall coated open tubular (WCOT) and support coated open tubular (SCOT) columns. (4 marks)
- (c) How does temperature programming improve separations? (5 marks)
- (d) What is required for fast GC analysis? (5 marks)
4. (a) Describe how high performance liquid chromatography (HPLC) differs from conventional liquid chromatography. (8 marks)
- (b) Describe two commonly used detectors in liquid chromatography and their bases of operation. (5 marks)
- (c) If the optimum volumetric flow rate for a 4.6 mm i.d. column is 1.5 mL min^{-1} with $5 \mu\text{m}$ particles, what would it be for a 2.1 mm i.d. column with the same particles? How much solvent would be consumed for a 10 min separation for each column? (5 marks)
- (d) Why are silica particles endcapped in bonded reversed-phase particles? (2 marks)
5. (a) Briefly describe the differences between microporous particles, perfusion particles, and nonporous particles. What are their unique features or uses? (9 marks)
- (b) What is a guard column and why is it used? (5 marks)
- (c) Why is mobile-phase gradient elution used in HPLC? (6 marks)
6. (a) What are some commonly used nonpolar bonded phases for reversed-phase HPLC and polar bonded phases for normal-phase chromatography? (5 marks)
- (b) What advantages do narrow-bore columns have in HPLC? (5 marks)
- ...4/-

- (c) Briefly discuss common interfaces/ionizers for liquid chromatography – mass spectrometry (LC-MS)?
(10 marks)
7. (a) Describe the principle of size exclusion chromatography. What is the exclusion limit?
(5 marks)
- (b) Describe the factors that affect the selectivity of ion exchange resins.
(5 marks)
- (c) Describe the principle of capillary electrophoresis. What are its advantages?
(5 marks)
- (d) Alkali metal ions can be determined volumetrically by passing a solution of them through a cation exchange column in the hydrogen form. They displace an equivalent amount of hydrogen ions that appear in the effluent and can be titrated. How many millimoles of potassium ion are contained in a liter of solution if the effluent obtained from a 5.00 mL aliquot run through a cation exchange column requires 26.7 mL of 0.0506 M NaOH for titration?
(5 marks)

1. (a) Apakah (i) pengekstrakan pelarut dipercepatkan dan (ii) pengekstrakan berbantuan mikrogelombang?
(5 markah)
- (b) Nisbah taburan bagi PdCl_2 antara larutan 3 M HCl dan pelarut tri-n-butilfosfat ialah 2.3. Berapakah peratus PdCl_2 yang terekstrak daripada 25.0 mL larutan 7.0×10^{-4} M PdCl_2 ke dalam 10.0 mL tri-n-butilfosfat?
(5 markah)
- (c) Sebanyak 90% suatu logam kelat dapat diekstrak apabila isipadu fasa akueus dan fasa organik adalah sama. Berapakah peratus pengekstrakan apabila isipadu fasa organik digandakan (2 kali isipadu asal)?
(5 markah)
- (d) Sebanyak 70% arsenik(III) telah diekstrak daripada 7 M HCl ke dalam isipadu sama banyak toluena. Berapa peratuskah yang belum terekstrak selepas tiga kali pengekstrakan dengan toluena?
(5 markah)
2. (a) Suatu puncak kromatografi gas mempunyai masa penahanan 65 s. Lebar dasar puncak yang diperolehi daripada silangan garis dasar dengan garis sisi puncak yang diekstrapolasi ialah 5.5 s. Jika panjang turus 1 m, berapakah tinggi plat, H, dalam cm plat^{-1} ?
(4 markah)
- (b) Terangkan prinsip pengesanan kromatografi gas (GC) berikut:
- (i) Kekonduksian terma.
 - (ii) Pengionan nyala.
 - (iii) Penangkapan elektron.
- Bandingkan pengesanan-pengesanan di atas daripada segi kepekaan dan jenis-jenis sebatian yang dapat dikesan.
(9 markah)
- (c) Apakah persamaan van Deemter? Takrifkan sebutan-sebutan dalam persamaan ini. Bagaimanakah persamaan-persamaan Huber dan Knox berbeza daripada persamaan van Deemter?
(7 markah)

...7/-

3. (a) Dua puncak kromatografi gas dengan masa penahanan masing-masing 85 dan 100 saat hanya cukup-cukup dibejelaskan menggunakan satu turus yang mempunyai nilai H sebanyak 1.5 cm plat^{-1} di bawah keadaan operasi. Berapakah panjang turus yang diperlukan? Anggap kedua-dua puncak mempunyai lebar dasar yang sama. (6 markah)
- (b) Bezakan antara turus terbuka dinding tersalut (WCOT) dengan turus terbuka penyokong tersalut (SCOT). (4 markah)
- (c) Bagaimanakah pemrograman suhu dapat memperbaiki pemisahan? (5 markah)
- (d) Apakah yang diperlukan bagi analisis GC cepat? (5 markah)
4. (a) Nyatakan bagaimana kromatografi cecair keupayaan tinggi (HPLC) berbeza daripada kromatografi cecair konvensional. (8 markah)
- (b) Nyatakan dua pengesanan yang biasa digunakan dalam kromatografi cecair dan asas operasinya. (5 markah)
- (c) Jika kadar alir volumetri optimum bagi suatu turus yang mempunyai garispusat dalaman 4.6 mm dengan zarah $5 \mu\text{m}$ ialah 1.5 mL min^{-1} , berapakah kadar alir ini bagi suatu turus yang bergarispusat dalaman 2.1 mm dengan saiz zarah $5 \mu\text{m}$? Berapa banyakkah pelarut yang digunakan (dalam unit mL) bagi pemisahan selama 10 min bagi setiap turus? (5 markah)
- (d) Mengapakah zarah silika di "endcap" dalam zarah fasa terbalik terikat? (2 markah)
5. (a) Dengan ringkas nyatakan perbezaan antara zarah mikroliang, zarah "perfusion" dan zarah tak berliang? Apakah ciri-ciri unik atau kegunaan masing-masing? (9 markah)
- (b) Apakah turus pengawal dan mengapakah ia digunakan? (5 markah)
- (c) Mengapakah elusi kecerunan fasa bergerak digunakan dalam HPLC? (6 markah)
- ...8/-

6. (a) Berikan beberapa fasa terikat tak berkutub yang biasa digunakan bagi HPLC fasa terbalik. Apakah pula fasa-fasa terikat berkutub bagi kromatografi fasa normal? (5 markah)
- (b) Apakah kelebihan turus "narrow-bore" dalam HPLC? (5 markah)
- (c) Bincang dengan ringkas antaramuka/pengion yang biasa bagi kromatografi cecair – spektrometri jisim (LC-MS). (10 markah)
7. (a) Terangkan prinsip kromatografi penyisihan saiz. Apakah had penyisihan? (5 markah)
- (b) Terangkan faktor-faktor yang mempengaruhi kepilhan resin pertukaran ion. (5 markah)
- (c) Terangkan prinsip elektroforesis rerambut. Apakah kelebihanannya? (5 markah)
- (d) Ion-ion logam alkali dapat ditentukan secara volumetri dengan melakukan suatu larutan logam alkali ini melalui turus pertukaran kation dalam bentuk hidrogen. Ion-ion logam alkali ini bertukar tempat dengan ion hidrogen dan ion hidrogen dalam efluen dititratkan. Berapakah milimol ion kalium yang terkandung dalam seliter larutan jika efluen yang diperolehi daripada suatu alikuot 5.00 mL yang dilakukan melalui turus pertukaran kation memerlukan 26.7 mL 0.0506 M NaOH dalam pentitratan? (5 markah)

-oooOooo-