
UNIVERSITI SAINS MALAYSIA

Second Semester Examination
Academic Session 2003/2004

February/March 2004

KAA 505 – Separation Methods

Time : 3 hours

Please make sure this paper consists of **FOUR** printed pages before answering the questions.

Answer **FIVE** questions.

Only the first five questions answered by the candidate will be marked.

1. (a) Various processes take place on a column during a chromatographic separation that contribute to band broadening. Discuss the theoretical aspects of band broadening as it was developed for GC and how it can be extended to LC.

(10 marks)

- (b). Discuss the performance of columns 1, 2 and 3 for the separation of species A and B from the chromatograms below;

(6 marks)

- (c) If you are given column 3, suggest how separation can be improved.

(4 marks)

2. (a) You are assigned to equip an analytical laboratory for environmental pollution monitoring using GC. Propose the appropriate GC system (instrumentation) and the necessary chemicals, providing justifications for your choice.

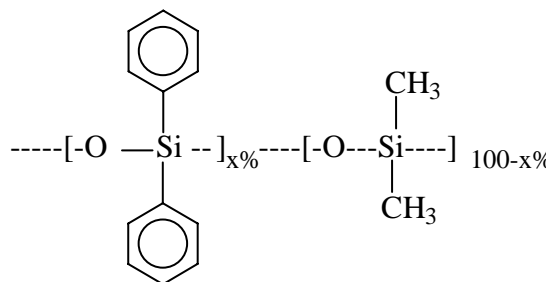
(10 marks)

- (b) Parallel and serial detectors have been proven to be suitable to certain applications. Nevertheless, due to the advances made in the interfacing technology that leads towards highly efficient hyphenated techniques, these systems become relatively impractical. Compare and contrast the dual detector systems and hyphenated techniques. Give an example to highlight the merits of each technique.

(10 marks)

...3/-

3. (a) Dimethyl polysiloxane is normally the choice as nonpolar stationary phase in GLC,. The polarity of dimethyl polysiloxane can be modified by introducing certain percentage of diphenyl siloxane to the dimethyl siloxane repeating units as shown below;



When $x=0$, nonpolar; $x=5$, low polarity and when $x=35$ or 65 , intermediate polarity columns are obtained. Give example of compound (or class of compounds) suitable for separation by nonpolar, low polarity and intermediate polarity stationary phase. Explain.

(10 marks)

- (b) Offer two major improvements to separation and quantitation after undergoing derivatization prior to gas chromatographic analysis.

(10 marks)

4. (a) Explain the choice of headspace technique for the determination of trace amount of volatile organochlorine in water.

(10 marks)

- (b) Compare and contrast how temperature programming in GC and gradient elution in LC improves separation.

(10 marks)

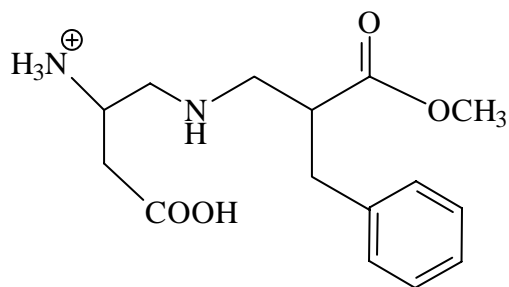
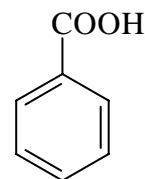
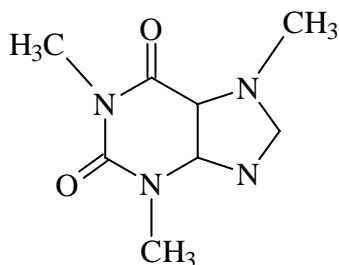
5. (a) Provide the advantages and disadvantages of using water as supercritical fluid.

(3 marks)

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- (b) Explain why the number of theoretical plates obtained from capillary electrophoresis is normally higher than chromatographic techniques.
(2 marks)
- (c) Discuss two specific applications of crown ethers as analytical reagents. What are some common strategies that can be implemented to regenerate these ligands?
(10 marks)
6. (a) Explain the main sample introduction modes in capillary electrophoresis.
(10 marks)
- (b) A soft drink containing aspartame (AS), benzoic acid (BA) and caffeine (K) has been separated by capillary electrophoresis method. If the separation was obtained by using buffer solution at pH 9.4, and based on the structure and pK_a values, predict the elution order of these analytes. Justify your answer.

(10 marks)

AS($pK_{a1} = 2.9$, $pK_{a2} = 7.4$)BA($pK_a = 4.2$)K($pK_a < 0$)

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