UNIVERSITI SAINS MALAYSIA

Second Semester Examination
2007/2008 Academic Session

KAA 505 – Separation Techniques
[Kaedah Pemisahan]

Duration : 3 hours
[Masa : 3 jam]

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

Instructions:

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

Begin your answer on a new page.

You may answer either in Bahasa Malaysia or in English.

If a candidate answers more than five questions, only the first five answers will be graded.

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

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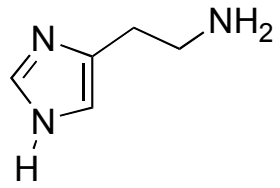
1. (a) Histamine ($pKa_1 = 6.9$; $pKa_2 = 10.4$) is a chemical messenger that is released in minute quantities ($\mu g\ L^{-1}$ levels) during an allergy attack and can lead to multiple biological effects including asthma, watery eyes and running nose. A student wishes to determine its concentration in a biological fluid.

(i) Describe a suitable liquid phase microextraction technique to treat the sample.

(ii) Describe a HPLC method for the determination of the analyte.

Provide justifications for your answers wherever possible.



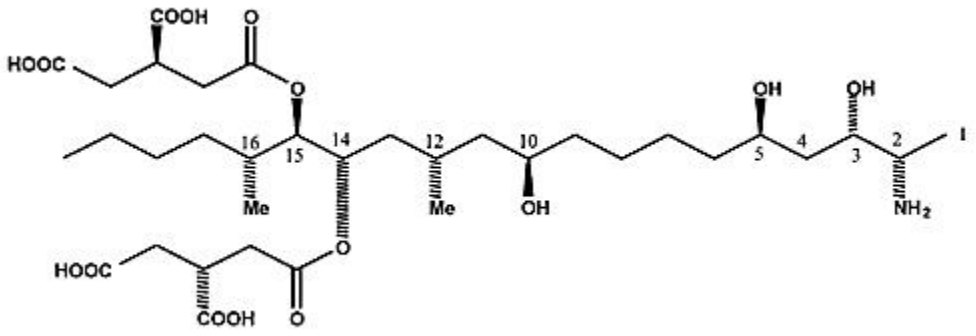
Histamine

(15 marks)

(b) What are the main functions of the modulator in comprehensive two dimensional GC (GC x GC) technique?

(5 marks)

2. (a) Fumonisins belong to a family of food-borne carcinogenic mycotoxins that were first isolated in 1988. Describe a GC method for the determination of fumonisin B1 (**I**) in an animal feed sample.



(I)

(10 marks)

(b) Discuss the effects of pH of the mobile phase in reversed phase HPLC. By providing a specific example, explain how pH can be used to modify the retention time.

(10 marks)

3. (a) Describe the operating principles for the following CE detectors:

(i) Chemiluminescence.

(ii) Capacitively coupled contactless conductivity (C4D).

(10 marks)

(b) List the limitations and capabilities of the chemiluminescence and C4D detectors.

(5 marks)

(c) Explain why the retention time of aniline becomes progressively longer when the pH of the buffer is made more basic.

(5 marks)

4. (a) Discuss the major advances in the following fields:

(i) Highly selective sorbents for solid phase extraction.

(ii) GC x GC.

(15 marks)

(b) In the determination of active ingredients in certain pharmaceutical tablets, pretreatment of samples is sometimes not required when CE technique is used but it will be mandatory for HPLC separations. Explain.

(5 marks)

5. (a) Describe the operational principles of the evaporative light scattering detector (ELSD).

(5 marks)

(b) What major innovations are required for the ELSD?

(5 marks)

(c) Describe an active area of research in CE. Explain how research in this area will help to overcome problems of the current technique.

(10 marks)

6. A typical electropherogram for the separation of 20 mg L^{-1} racemic citalopram **(II)** is shown below.

(a) Suggest the conditions that give rise to the successful separation.

(4 marks)

(b) How can the efficiency be further improved?

(3 marks)

(c) How can the electroosmotic flow (EOF) be determined?

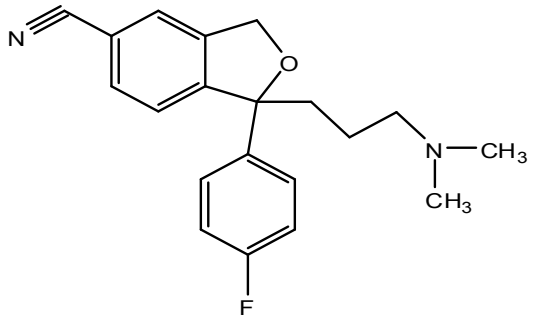
(4 marks)

(d) Describe how the racemates can be separated using HPLC instead.

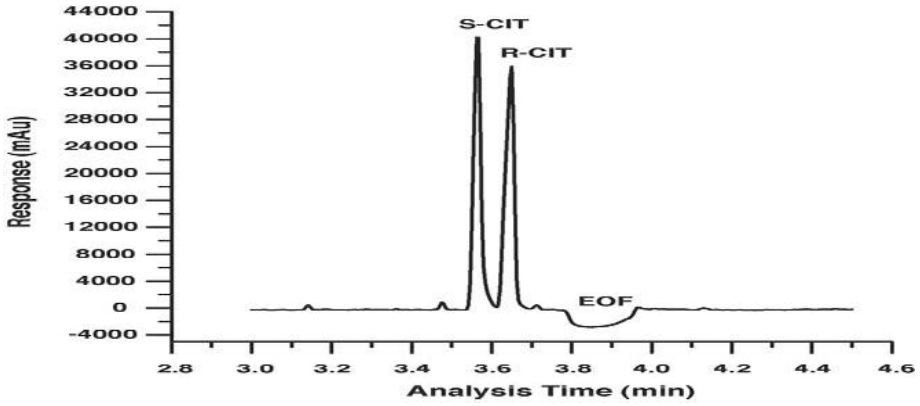
(5 marks)

(e) Compare and contrast the separations between the two techniques (i.e., CE and HPLC).

(4 marks)



(II)

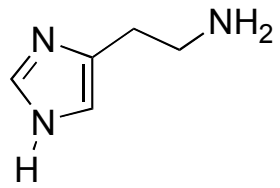


1. (a) Histamina (pKa_1 = 6.9; pKa_2 = 10.4) adalah satu pengutus kimia yang diterbitkan dengan kuantiti kecil (paras μg L^{-1}) semasa serangan alahan dan boleh mengakibatkan pelbagai kesan biologi termasuklah lelah, mata berair dan selsema. Seorang pelajar ingin menentukan kepekatannya di dalam bendalir biologi.

(i) Terangkan satu kaedah pengekstrakan mikro fasa cecair untuk merawat sampel.

(ii) Terangkan keadaan HPLC untuk penentuan analit.

Berikan justifikasi bagi jawapan anda dimana boleh.



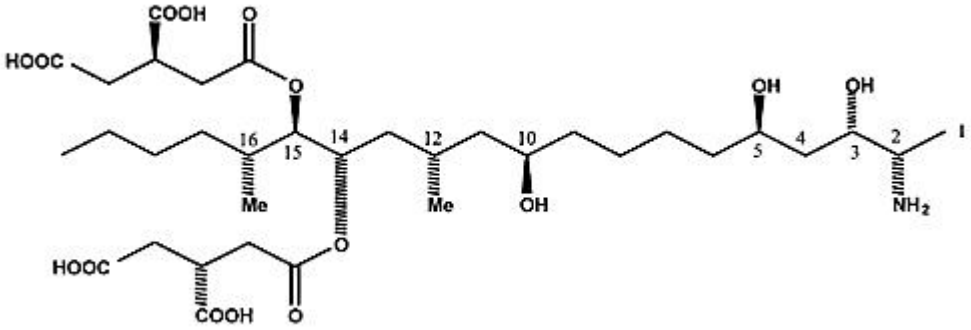
Histamine

(15 markah)

(b) Apakan fungsi utama pemodulasi bagi kaedah kromatografi gas dua dimensi komprehensif (GC x GC)?

(5 markah)

2. (a) Fumonisin merupakan suatu famili mikotoksin karsinogen makanan yang telah pertama kalinya dipencilkan pada 1988. Terangkan satu kaedah GC bagi penentuan fumonisin B1 (**I**) di dalam sampel makanan ternakan.



(I)

(10 markah)

(b) Bincangkan kesan pH fasa gerak bagi HPLC fasa terbalik. Dengan memberikan satu contoh spesifik, terangkan bagaimana pH boleh digunakan untuk mengubah masa penahanan.

(10 markah)

3. (a) Terangkan prinsip operasi bagi pengesan CE berikut:

(i) Pendarcahaya.

(ii) Gandingan kapasitif kekonduksian tanpa sentuh (C4D).

(10 markah)

(b) Senaraikan keupayaan dan penghadan bagi pengesan pendarcahaya dan C4D.

(5 markah)

(c) Terangkan mengapa masa penahanan bagi anilina menjadi semakin lama apabila pH tampan dijadikan semakin berbes.

(5 markah)

4. (a) Bincangkan perkembangan utama di dalam bidang berikut:

(i) Penjerap berkepilihan tinggi bagi pengekstrakan fasa pepejal.

(ii) GC x GC.

(15 markah)

(b) Di dalam penentuan bahan aktif di dalam tablet farmaseutik, prapengolahan sampel kadang-kadang tidak diperlukan jika kaedah CE digunakan tetapi ia adalah perlu untuk pemisahan HPLC. Terangkan.

(5 markah)

5. (a) Terangkan prinsip operasi pengesan sejatan penyerakan cahaya (ELSD).

(5 markah)

(b) Apakah inovasi utama yang diperlukan untuk pengesan ELSD?

(5 markah)

(c) Terangkan bidang penyelidikan yang aktif di dalam CE. Jelaskan bagaimana penyelidikan di dalam bidang ini akan membantu untuk mengatasi masalah kaedah kini.

(10 markah)

6. Satu elektroferogram tipikal bagi pemisahan 20 mg L^{-1} rasemat citalopram **(II)** ditunjuk di bawah.

(a) Cadangkan keadaan yang menghasilkan pemisahan yang berjaya.

(4 markah)

(b) Bagaimanakah kecekapan boleh di baiki selanjutnya?

(3 markah)

(c) Bagaimanakah aliran elektroosmotik (EOF) boleh ditentukan?

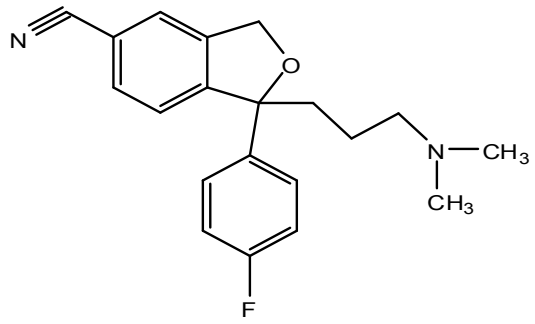
(4 markah)

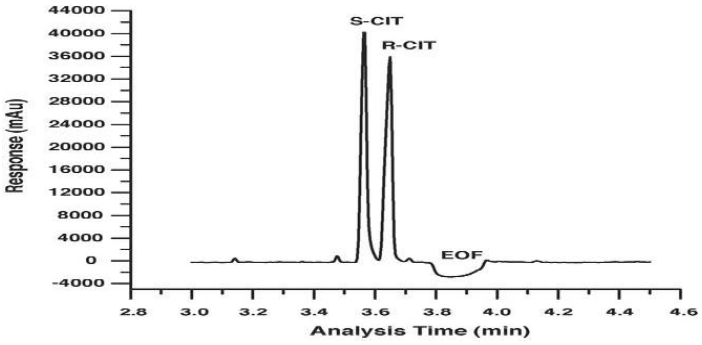
(d) Terangkan bagaimana rasemat boleh dipisahkan menggunakan HPLC.

(5 markah)

(e) Banding dan bezakan pemisahan di antara kedua kaedah (iaitu, CE dan HPLC).

(4 markah)





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