
UNIVERSITI SAINS MALAYSIA

First Semester Examination
2010/2011 Academic Session

November 2010

KAT 347 – Electroanalytical Methods
[Kaedah Elektroanalisis]

Duration : 3 hours
[Masa : 3 jam]

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

Instructions:

The paper has **SEVEN** questions in two **SECTION A** and **B**.

Answer **FIVE** questions only with at least **TWO** questions from each section.

Answer each question on a new page.

You may answer the questions 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.

Section A – Potentiometry

1. (a) A membrane is the crucial part of an ion selective electrode (ISE). Describe two types of conventional membranes and their mechanisms that generate the responses.
(10 marks)
- (b) Explain why is glass electrode still unrivalled by other electrodes for solution pH measurements?
(10 marks)
2. (a) What is the significance of selectivity coefficient to an ISE? How would you determine its value?
(12 marks)
- (b) A nitrate ISE and a reference electrode were dipped into a solution of nitrate with an activity of 1.00×10^{-3} M. The potential of nitrate ISE was found to be -122.4 mV. Both electrodes were then dipped into a solution containing both nitrate and chloride, each of which had an activity of 1.00×10^{-3} M. The potential of the nitrate ISE in this solution was -124.8 mV. The temperatures of both solutions were 25 °C. Calculate the selectivity coefficient of the nitrate ISE over chloride.
(8 marks)
3. (a) Describe one way to prepare a membrane for the potentiometric measurement of either Ag^+ or Pb^{2+} . What is its major interfering ion and how is it minimised?
(10 marks)
- (b) Discuss the major sources of error in potentiometric measurements.
(10 marks)

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4. (a) Data in the following table were obtained with a cadmium ISE and used for calibration plot of cadmium in an environmental water sample. The ionic strength of each solution was 0.3 M, and the reference electrode was a saturated calomel electrode (SCE).

[Cd ²⁺], M	1.00x10 ⁻¹	8.75x10 ⁻³	1.00x10 ⁻³	3.16x10 ⁻⁵	3.16x10 ⁻⁶	sample
E, V	-0.115	-0.150	-0.175	-0.213	-0.242	-0.200

Plot a graph on semi-log paper and determine the concentration of cadmium in the sample. Give an example of source of cadmium pollution in environmental waters.

(10 marks)

- (b) List down the differences between a coated wire electrode, or sometime called a solid state electrode, and a chemically modified electrode.

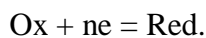
(10 marks)

Section B – Voltammetry

5. (a) What is a double layer? How does this affect the performance of an analysis? Suggest ways to overcome this.

(10 marks)

- (b) The data in the following table were obtained using dropping mercury electrode against an SCE at 25 °C on the rising portion of a reversible cathodic wave which obeys the general equation,



E, V	-0.600	-0.612	-0.620	-0.627	-0.639	-0.650
I, μA	0.91	2.01	3.20	4.44	6.66	8.25

The diffusion current (I_d) of the wave was 10.0 μA. Calculate the number of electrons (n) transferred and the half-wave potential ($E_{1/2}$) in the electrode process.

(10 marks)

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6. (a) Voltammetric analysis is a diffusion-controlled technique. How do you improve diffusion in an electrochemical system? Briefly explain one experiment to check whether this has been attained. (10 marks)
- (b) Why is differential pulse voltammetry becoming the most favoured technique for quantitative analyses nowadays? (10 marks)
7. (a) What is reversibility in voltammetry? Why is it significant to the analysis? (10 marks)
- (b) Describe the basic principle and application of either one of these techniques: cyclic voltammetry or stripping voltammetry (10 marks)

TERJEMAHAN

Arahan:

Kertas ini mengandungi **TUJUH** soalan dalam dua **BAHAGIAN A** dan **B**.

Jawab **LIMA** soalan sahaja dengan sekurang kurang **DUA** soalan dari setiap bahagian.

Jawab setiap soalan pada muka surat yang baru.

Anda dibenarkan menjawab soalan ini 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.

Bahagian A – Potensiometri

1. (a) Membran ialah suatu bahagian yang penting dalam suatu elektrod pemilih ion (ISE). Huraikan dua jenis membran yang lazim dan mekanisme masing-masing dalam menjana gerakbalas.
(10 markah)
- (b) Terangkan mengapakah elektrod kaca masih lagi tiada saingan daripada elektrod-elektrod lain bagi menyukat pH larutan?
(10 markah)
2. (a) Apakah kepentingan pekali kepilihan kepada suatu ISE? Bagaimanakah anda menentukan nilainya?
(12 markah)
- (b) Suatu ISE nitrat dan suatu elektrod rujukan telah di celupkan kedalam suatu larutan nitrat dengan keaktifan 1.00×10^{-3} M. Keupayaan ISE nitrat didapati -122.4 mV. Kedua elektrod kemudian dicelup kedalam suatu larutan yang mengandungi ion nitrat dan klorida dengan keaktifan masing-masing 1.00×10^{-3} M. Keupayaan ISE nitrat dalam larutan ini ialah -124.8 mV. Suhu kedua larutan ialah 25 °C. Kira pekali kepilihan ISE nitrat terhadap klorida.
(8 markah)
3. (a) Huraikan satu cara penyediaan suatu membran bagi penyukatan potensiometri samada Ag^+ atau Pb^{2+} . Apakah ion ganggu utama dan bagaimanakah meminimumkannya?
(10 markah)
- (b) Bincangkan punca-punca utama ralat dalam penyukatan potensiometri.
(10 markah)

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4. (a) Data dalam jadual dibawah didapati menerusi ISE kadmium dan digunakan bagi plot tentukan kadmium dalam suatu sampel air persekitaran. Kekuatan ion setiap larutan ialah 0.3 M, dan elektrod rujukan ialah elektrod kalomel tepu (SCE).

[Cd ²⁺], M	1.00x10 ⁻¹	8.75x10 ⁻³	1.00x10 ⁻³	3.16x10 ⁻⁵	3.16x10 ⁻⁶	sample
E, V	-0.115	-0.150	-0.175	-0.213	-0.242	-0.200

Plot suatu graf diatas kertas semi-log dan tentukan kepekatan kadmium dalam sampel. Berikan satu contoh punca pencemaran kadmium dalam air persekitaran.

(10 markah)

- (b) Senarai turunkan perbezaan diantara suatu elektrod dawai tersalut, atau kekadang disebut suatu elektrod keadaan pepejal, dan suatu elektrod terubahsuai secara kimia.

(10 markah)

Bahagian B – Voltammetri

5. (a) Apakah suatu lapisan ganda dua? Bagaimanakah ini memberi kesan terhadap prestasi suatu analisis? Cadangkan cara-cara mengatasinya.

(10 markah)

- (b) Data dalam jadual dibawah diperolehi menggunakan elektrod titisan merkuri melawan suatu SCE pada 25 °C terhadap bahagian menaik suatu gelombang katodik berbalik yang menurut persamaan umum,



E, V	-0.600	-0.612	-0.620	-0.627	-0.639	-0.650
I, μA	0.91	2.01	3.20	4.44	6.66	8.25

Arus bauran (I_d) gelombang ialah 10.0 μA . Kira jumlah elektron (n) yang dipindahkan dan keupayaan setengah gelombang ($E_{1/2}$) dalam proses elektrod tersebut.

(10 markah)

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6. (a) Analisis voltammetri ialah suatu teknik terkawal bauran. Bagaimanakah anda memperbaiki bauran dalam suatu sistem elektrokimia? Dengan ringkas terangkan satu eksperimen bagi mengesahkan samada ini sudah dicapai.
(10 markah)
- (b) Mengapakah semasa ini voltammetri denyut pembezaan semakin disukai bagi analisis kuantitatif?
(10 markah)
7. (a) Apakah kebalikan dalam voltammetri? Mengapakah ianya penting terhadap analisis?
(10 markah)
- (b) Nyatakan prinsip asas dan kegunaan bagi salah satu antara teknik berikut: voltammetri berkitar atau voltammetri pelucutan.
(10 markah)

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