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# UNIVERSITI SAINS MALAYSIA

First Semester Examination  
2015/2016 Academic Session

December 2015 / January 2016

## EBS 336/3 – Analytical Chemistry [Kimia Analitis]

Duration : 3 hours  
[Masa : 3 jam]

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Please ensure that this examination paper contains THIRTEEN printed pages and ONE page APPENDIX before you begin the examination.

*[Sila pastikan bahawa kertas peperiksaan ini mengandungi TIGA BELAS muka surat beserta SATU muka surat LAMPIRAN yang bercetak sebelum anda memulakan peperiksaan ini.]*

This paper consists of SEVEN questions.

*[Kertas soalan ini mengandungi TUJUH soalan.]*

**Instruction:** Answer FIVE questions. If a candidate answers more than five questions only the first five questions answered in the answer script would be examined.

**Arahan:** Jawab LIMA soalan. Jika calon menjawab lebih daripada lima soalan hanya lima soalan pertama mengikut susunan dalam skrip jawapan akan diberi markah.]

The answers to all questions must start on a new page.

*[Mulakan jawapan anda untuk semua soalan pada muka surat yang baru.]*

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

*[Anda dibenarkan menjawab soalan sama ada dalam Bahasa Malaysia atau Bahasa Inggeris.]*

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

*[Sekiranya terdapat sebarang percanggahan pada soalan peperiksaan, versi Bahasa Inggeris hendaklah digunapakai.]*

1. [a] What do you understand by the term masking in a chemical separation method and in what condition do you need to add a masking agent?

*Apakah yang anda faham tentang istilah "masking" dalam kaedah pemisahan kimia dan dalam keadaan yang bagaimana agen masking diperlukan?*

(15 marks/markah)

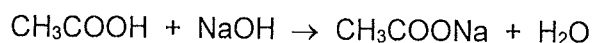
- [b] Calculate the concentrations of the various species at equilibrium in a solution prepared by diluting 1.40 moles of KCN and 0.10 moles of  $\text{Cd}(\text{NO}_3)_2$  to 1 liter with water.

*Kirakan kepekatan pelbagai spesi-spesi yang wujud pada keseimbangan dalam satu larutan yang telah disediakan dari pencairan 1.40 moles KCN dan 0.10 moles  $\text{Cd}(\text{NO}_3)_2$  dengan 1 liter air.*

(25 marks/markah)

- [c] Calculate the pH of the buffer prepared by mixing 500 mL of 0.200 M acetic acid with 1.00 g of sodium hydroxide. The  $K_a$  for acetic acid is  $1.75 \times 10^{-5}$ .

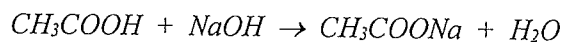
Given the chemical equation:



Formula weight for NaOH = 40.0 g/mole

*Kirakan pH bagi satu larutan tampan yang disediakan dengan mencampurkan 500 mL 0.200 M asid asetik dengan 1.00 g natrium hidroksida. Nilai  $K_a$  bagi asid asetik adalah  $1.75 \times 10^{-5}$ .*

*Diberikan tindak balas kimia:*



*Berat molekul NaOH = 40.0 g/mol*

(30 marks/markah)

- [d] 0.3017 g sample of uranium ore was dissolved and pre-reduced to convert all of the uranium to  $U^{4+}$ . This solution required 28.37 mL of 0.01374 M  $KMnO_4$  for titration to  $UO_2^{2+}$  in 0.1 M  $H_2SO_4$ . Calculate the percentage of  $U_3O_8$  in the sample.

Given: At. wt of U = 238 and O = 16 g/mole

*0.3017 g sampel bijih uranium dilarutkan dan melalui pra-penurunan untuk menukarkan kesemua uranium kepada  $U^{4+}$ . Larutan ini memerlukan 28.37 mL 0.01374 M  $KMnO_4$  bagi pentitratan kepada  $UO_2^{2+}$  in 0.1 M  $H_2SO_4$ . Kirakan peratus  $U_3O_8$  dalam sampel.*

*Diberikan: Jisim atom U = 238 dan O = 16 g/mol*

(30 marks/markah)

2. [a] Sodium hydroxide is added to a solution containing magnesium (II) so that a precipitate of  $Mg(OH)_2$  forms, and the pH of the aqueous solution is 11.0. From the solubility product of  $Mg(OH)_2$  ( $K_{sp} = 1.82 \times 10^{-11}$ ), calculate the molar concentration of the magnesium ion in solution.

*Natrium hidroksida ditambahkan kepada satu larutan yang mengandungi magnesium (II) untuk membentuk mendakan  $Mg(OH)_2$  dan pH larutan akuas adalah 11.0. Dari hasil darab keterlarutan  $Mg(OH)_2$  ( $K_{sp} = 1.82 \times 10^{-11}$ ), kirakan kepekatan molar ion magnesium dalam larutan.*

(30 marks/markah)

- [b] A 0.2000 g sample of a metal alloy is dissolved and the tin is reduced to tin (II). Titration of the Sn (II) requires 22.20 mL of 0.1000 N  $K_2Cr_2O_7$  where the tin is oxidized from tin (II) to tin (IV). Calculate the % of tin in the metal alloy.

Given: Atomic weight of tin = 118.71 g/mole

*Suatu sampel aloi logam dengan berat 0.2000 g telah dilarutkan dan kandungan Sn diturunkan kepada Sn (II). Pentitratan Sn (II) memerlukan 22.20 mL 0.1000 N  $K_2Cr_2O_7$  di mana timah itu dioksidakan kembali dari Sn (II) to Sn (IV). Kirakan % timah dalam aloi logam tersebut.*

*Diberikan: Berat atom timah = 118.71 g/mol*

(30 marks/markah)

- [c] A new method for the determination of copper in the waste water containing cyanide from a gold processing plant is being developed. The results of several samples are compared with a standard method. From the following data below, determine by the t test if there is a statistically significant difference between the two methods for analyzing a range of sample concentrations. It has been ascertain that the two methods have comparable precisions.

*Satu kaedah baru bagi penentuan kuprum dalam air sisa mengandungi sianida dari satu loji pemprosesan emas telah dibangunkan. Keputusan analisis dari beberapa sampel telah dibandingkan dengan kaedah piawai. Dari data berikut, dengan menggunakan ujian t, tentukan samada wujud perbezaan statistik yang bermakna antara dua kaedah ini dalam menganalisis satu julat kepekatan sampel. Kedua-dua kaedah telah dibuktikan mempunyai kepersisian yang sama.*

**Table 1 - The two sets of results for a number of individual samples****Jadual 1 - Dua set keputusan bagi sampel-sampel individu**

Sample <i>Sampel</i>	Your method <i>Kaedah anda</i> (mg/L)	Standard method <i>Kaedah piawai</i> (mg/L)
A	10.2	10.5
B	12.7	11.9
C	8.6	8.7
D	17.5	16.9
E	11.2	10.9
F	11.5	11.1

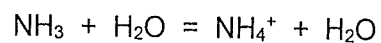
Refer to Table A as in Appendix 1.

*Rujuk kepada Jadual A dalam Lampiran 1.*

(40 marks/markah)

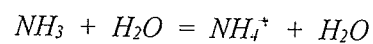
3. [a] Calculate the pH of a 0.0750 M solution of ammonia in water. The  $K_b$  for ammonia is  $1.75 \times 10^{-5}$ .

Given the chemical equilibrium:



*Kirakan pH satu larutan 0.0750 M ammonia dalam air. Nilai  $K_b$  bagi ammonia adalah  $1.75 \times 10^{-5}$ .*

*Diberikan keseimbangan kimia:*



(30 marks/markah)

- [b] A 50.00 mL aliquot of hard water sample is titrated with 15.00 mL of 0.0100 M EDTA using an indicator. A second 50.00 mL aliquot is made strongly alkaline with sodium hydroxide. A precipitate results. An indicator (II) is then added to the solution. The sample then requires 10.00 mL of 0.0120 M EDTA for titration. What is the total hardness (as  $\text{CaCO}_3$ ) of the water?

Given: Formula wt of  $\text{CaCO}_3$  is 100.1 g/mole

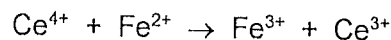
*Sebanyak 50.00 mL aliquot sampel air keras dititratkan dengan 15.00 mL 0.0100 M EDTA menggunakan zat penunjuk (I). Alikuot kedua sebanyak 50.00 mL dijadikan sangat beralkali dengan penambahan natrium hidroksida di mana satu mendakan terbentuk. Zat penunjuk (II) kemudian ditambahkan kepada larutan. Larutan memerlukan 10.00 mL 0.0120 M EDTA pentitratan. Apakah keliatan total air (dalam sebutan  $\text{CaCO}_3$ )?*

*Diberikan: Berat formula  $\text{CaCO}_3$  adalah 100.1 g/mol*

(40 marks/markah)

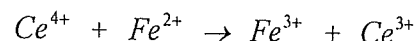
- [c] A 1.0000 g sample of limonite iron ore ( $2 \text{Fe}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ ) is dissolved, reduced to  $\text{Fe}^{2+}$  and titrated with 20.00 mL of 0.200 N cerium (IV). Calculate the percentage of  $2 \text{Fe}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$  (formula wt. of 373.38 g/mole) in the sample.

Given the oxidation and reduction equation:



*Sebanyak 1.0000 g sampel bijih besi limonit ( $2 \text{Fe}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ ) telah dilarutkan dan ferum diturunkan kepada  $\text{Fe}^{2+}$  dan dititratkan dengan 20.00 mL 0.200 N cerium (IV). Kirakan peratus  $2 \text{Fe}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$  (berat formula 373.38 g/mol) dalam sampel.*

*Diberikan persamaan pengoksidaan dan penurunan:*



(30 marks/markah)

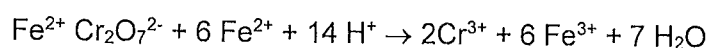
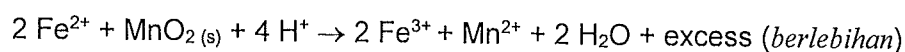
4. [a] Explain the principles of back titration.

*Terangkan prinsip pentitratan balik.*

(25 marks/markah)

- [b] Manganese dioxide can be determined by reduction with excess ferrous sulphate. The excess iron (II) is then back titrated with potassium dichromate as shown in the chemical equation below:

*Manganese dioksida boleh ditentukan secara penurunan menggunakan ferus sulfat berlebihan. Ferum (II) yang berlebihan ini dititratkan balik dengan kalium dikromat seperti dalam persamaan kimia berikut:*



Calculate the %  $\text{MnO}_2$ .

Given:

Weight of sample = 200 mg,

Volume of Iron (II) added = 50.0 mL of 0.1 000 M,

Volume of dichromate from back-titration = 16.07 mL of 0.0230 M

Formula wt. of  $\text{MnO}_2$  = 86.94 g /mole

*Kirakan %  $\text{MnO}_2$*

*Diberikan:*

*Berat sampel = 200 mg,*

*Isipadu Iron (II) ditambah = 50.0 mL of 0.1 000 M,*

*Isipadu dikromat dari pentitratan balik = 16.07 mL of 0.0230 M*

*Berat formula  $\text{MnO}_2$  = 86.94 g /mole*

(35 marks/markah)

- [c] The following are analysis data from different sets of measurement for the percentages of Ag recovered by the ion-exchange method from different electroplating wastes solutions:

*Berikut adalah data analisis dari set pengukuran yang berbeza bagi peratus perolehan Ag dari kaedah penukaran ion dari sisa larutan penyaduran.*

Set	% Ag
1	19.76 %, 19.58%, 19.82 %
2	31.56%, 31.85%, 31.90 %, 31.42 %
3	24.17 %, 24.08%, 23.97 %
4	30.01 %, 29.66%, 30.34%, 29.83%

Calculate the pooled standard deviation and the relative standard deviation (ppt) of the method used.

*Kirakan sisihan piawai terkumpul dan sishan piawai relatif (ppt) bagi kaedah yang digunakan.*

(40 marks/markah)

5. [a] You and your friend are in the analytical lab doing a UV-Visible experiment. Your friend asks you about white light and why we see a solution containing copper ion is blue in color. Explain briefly why we 'see' objects as colored? Briefly describe electromagnetic radiation.

*Anda dan rakan anda berada dalam makmal analitikal dan sedang melakukan eksperimen UV-ternampakkan. Rakan anda bertanya pada anda tentang cahaya putih dan mengapa kita melihat larutan yang mengandungi ion kuprum adalah berwarna biru. Terangkan secara ringkas mengapa kita melihat sesuatu objek itu berwarna? Terangkan secara ringkas sinaran elektromagnet.*

(30 marks/markah)



- [b] The Beer-Lambert's law is the linear relationship between absorbance and concentration of an absorbing species. State the Beer-Lambert's law equation and define absorbance, transmittance and molar absorptivity.

*Hukum Beer- Lambert's adalah hubungan linear antara keserapan dan kepekatan spesies yang menyerap. Nyatakan persamaan hukum Beer-Lambert's dan tentukan definisi keserapan (absorbans), kehantaran dan molar keberserapan.*

(20 marks/markah)

- [c] The absorbance of an iron thiocyanate solution containing 0.00500 mg Fe/mL was reported as 0.4900 at 540 nm.
- Calculate the specific absorptivity, including units, of iron thiocyanate on the assumption that a 1.00 cm cuvette was used.
  - What will be the absorbance value if the solution is diluted to twice its original volume and the solution is placed in a 5.00 cm cuvette?

*Keserapan bagi satu larutan ferum tiosianat yang mengandungi 0.00500 mg Fe/mL dilaporkan adalah 0.4900 pada 540 nm.*

- Kirakan keberserapan tentu, termasuk unit, ferum tiosianat dengan menganggap bahawa 1.00 cm kuvet telah digunakan.*
- Apakah nilai keserapan jika larutan dicairkan dua kali ganda dari jumlah asalnya dan larutan diletakkan di dalam 5.00 cm kuvet?*

(30 marks/markah)

- [d] A 0.5000g steel sample is dissolved and the Mn in the sample is oxidized to permanganate by periodate using  $\text{Ag}^+$  as a catalyst. After the sample is diluted to 250.0 ml, the absorbance value is 0.393 at 540 nm in a 1.00 cm cell. Calculate the percentage of Mn in the steel. The molar absorptivity for permanganate at 540 nm is 2025.  
Given: Atomic weight of Mn = 54.94; Ag = 107.87 g/mole

*Satu sampel keluli dengan berat 0.5000g dilarutkan dan Mn dalam sampel dioksidakan kepada permanganat oleh periodat dengan menggunakan  $\text{Ag}^+$  sebagai pemangkin. Selepas sampel dicairkan kepada 250.0 ml, nilai keserapan adalah 0.393 pada 540 nm dalam sel 1.00 cm. Kirakan peratusan Mn dalam keluli. Molar keserapan untuk permanganat pada 540 nm adalah 2025.*

*Diberikan: Berat atom Mn = 54.94; Ag = 107.87 g/mol*

(20 marks/markah)

6. [a] What is the basic principle of atomic absorption spectroscopy and with the aid of a schematic diagram show the main components in AAS. What are the main interferences that can occur in atomic spectroscopy, and what can be done to alleviate them?

*Apakah prinsip asas spektroskopi penyerapan atom dan dengan bantuan gambarajah skema, tunjukkan komponen-komponen utama dalam AAS. Apakah gangguan-gangguan utama yang boleh berlaku dalam spektroskopi atom, dan apakah yang boleh dilakukan untuk mengurangkan gangguan tersebut?*

(40 marks/markah)

- [b] What are the differences between atomic absorption spectroscopy and flame emission spectroscopy?

*Apakah perbezaan-perbezaan antara spektroskopi penyerapan atom dan spektroskopi pancaran nyalaan?*

(20 marks/markah)

- [c] Data given below (Table 2) were obtained from Mn determination in an ore sample by AAS. Plot the calibration curve and calculate the Mn concentration in both sample 1 (X1) and sample 2 (X2) in ppm unit of concentration.

*Data berikut (Jadual 2) diperolehi dari penentuan Mn dalam satu sampel bijih dengan menggunakan AAS. Plot keluk penentukuran dan kirakan kepekatan Mn dalam kedua-dua sampel 1 (X1) dan sampel 2 (X2) dalam unit kepekatan ppm.*

**Table 2 - Analysis of Mn concentration in ore sample by AAS**

*Jadual 2 - Analisis kepekatan Mn dalam sampel bijih menggunakan AAS*

Standard Solution <i>Larutan Piawai</i>	Concentration Mn (ppm) <i>Kepekatan Mn (ppm)</i>	Absorbance <i>Keserapan</i>		
		1	2	3
1	0.00	0.000	0.0001	0.0000
2	2.00	0.053	0.052	0.051
3	3.8	0.104	0.105	0.104
4	5.8	0.160	0.160	0.163
5	8.00	0.220	0.220	0.220
6	9.60	0.260	0.261	0.262
<b>Ore Sample 1</b> <i>Sampel bijih 1</i>	X1	0.257	0.257	0.256
<b>Ore sample 2</b> <i>Sampel bijih 2</i>	X2	0.119	0.120	0.120

(40 marks/markah)

7. [a] With the aid of a sketch, explain in detail the typical setup to produce X-ray.

*Dengan bantuan lakaran, jelaskan dengan mendalam aturan tipikal bagi menghasilkan sinar-X.*

(30 marks/markah)

- [b] A crystal lattice is a regular three-dimension distribution of atoms in space. With aid of figures, name FIVE of the fourteen Bravais lattice structures.

*Sesuatukrystal ialah pengagihan secara lazim atom-atom dalam ruang. Dengan bantuan rajah, namakan LIMA dari empat belas struktur-struktur kristal Bravais.*

(15 marks/markah)

- [c] Analyses using XRF offer advantages such as (i) being versatile, (ii) accurate, (iii) reproducible, (iv) rapid analysis and (v) non-destructive. Specify exactly what each of the advantages (i)-(v) mean, and explain how each can be deemed as an advantage.

*Analisis-analisis menggunakan XRF memberikan kelebihan-kelebihan seperti (i) kepelbagaian, (ii) tepat, (iii) kebolehulangan, (iv) analisis pantas dan (v) tidak musnah. Jelaskan dengan spesifik apakah maksud kelebihan-kelebihan (i)-(v), dan terangkan bagaimana setiap satu tersebut boleh dianggap sebagai kelebihan.*

(25 marks/markah)

- [d] Elaborate on FOUR possible practices that ensure good accuracy for XRF analyses.

*Ulas dengan lanjut berkenaan EMPAT amalan-amalan mungkin yang dapat memastikan ketepatan yang tinggi bagi analisis-analisis XRF.*

*(20 marks/markah)*

- [e] Theoretically, detection of X-ray emission from virtually all elements is possible via XRF. However, practically, this is not possible. Discuss THREE main limitations of XRD in this case.

*Secara teoretikal, pengesanan penghasilan sinar-X dari hampir semua unsur adalah mungkin menggunakan XRF. Namun begitu, secara praktikal, ini tidak dapat dilakukan. Bincangkan TIGA keterbatasan XRD dalam kes ini.*

*(10 marks/markah)*

**APPENDIX****LAMPIRAN****Table A: Values of t for  $\nu$ , Degrees of freedom for various confidence level***Jadual A: Nilai-nilai t bagi darjah kebebasan,  $\nu$  pada pelbagai paras keyakinan*

$\nu$	Confidence Level			
	Paras Keyakinan			
	90 %	95 %	99 %	99.5 %
1	6.314	12.706	63.657	127.32
2	2.920	4.303	9.925	14.089
3	2.353	3.182	5.841	7.453
4	2.132	2.776	4.604	5.598
5	2.015	2.571	4.032	4.773
6	1.943	2.447	3.707	4.317
7	1.895	2.365	3.500	4.029
8	1.860	2.306	3.355	3.832
9	1.833	2.262	3.250	3.690
10	1.812	2.228	3.169	3.581
15	1.753	2.131	2.947	3.252
20	1.725	2.086	2.845	3.153
25	1.708	2.060	2.787	3.078
$\infty$	1.645	1.960	2.576	2.807

\*  $\nu = N - 1 =$  degrees of freedom**Table B: Rejection quotient, Q, at different confidence limits***Jadual B: Rejection quotient, Q, pada had keyakinan berbeza*

No. of Observations	Confidence Level		
	Paras Keyakinan		
	$Q_{90}$	$Q_{95}$	$Q_{99}$
3	0.941	0.970	0.994
4	0.765	0.829	0.926
5	0.642	0.710	0.821
6	0.560	0.625	0.740
7	0.507	0.568	0.680
8	0.468	0.526	0.634
9	0.437	0.493	0.598
10	0.412	0.466	0.568
15	0.338	0.384	0.475
20	0.300	0.342	0.425
25	0.277	0.317	0.393
30	0.260	0.298	0.372

\*Adapted from D.B. Rorabacher. Anal. Chem., 63 (1991) 139.