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# UNIVERSITI SAINS MALAYSIA

First Semester Examination  
Academic Session 2010/2011

November 2010

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

Duration : 3 hours  
[Masa : 3 jam]

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

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

This paper consists of SEVEN questions.

*[Kertas soalan ini mengandungi TUJUH soalan.]*

**Instruction:** Answer **FIVE** questions. If 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, the English version shall be used.

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

...2/-

1. [a] Name the **two** types of indicators used in the detection of end point in precipitation reactions involving the determination of chloride ion. Distinguish between the two indicators and write a brief description of the mechanisms involve with the use of each indicator. Write the chemical equations where necessary.

*Namakan **dua** jenis zat penunjuk yang digunakan dalam pengesanan takat akhir dalam tindakbalas pemendakan yang melibatkan penentuan ion klorida. Bezakan antara dua zat penunjuk tersebut dan tuliskan secara ringkas mekanisme yang terlibat dengan penggunaan setiap zat penunjuk. Tuliskan persamaan tindakbalas di mana perlu.*

(30 marks/markah)

- [b] A soda ash sample is analyzed in the lab by titration with standard hydrochloric acid. The analysis is performed in triplicate with the following results: 93.50, 93.58 and 93.43 %  $\text{Na}_2\text{CO}_3$ . Within what range are you confident that the true value lies? Refer to Table A as in Appendix I.

*Satu sampel abu soda dianalisis di makmal menggunakan kaedah pentitratan dengan asid hidroklorik. Analisis dijalankan sebanyak tiga kali dengan keputusan berikut: 93.50, 93.58 dan 93.43 %  $\text{Na}_2\text{CO}_3$ . Carikan julat di mana anda yakin bahawa nilai sebenarnya berada? Rujuk pada Jadual A dalam Lampiran I.*

(20 marks/markah)

- [c] The tin and zinc contents of a brass sample are analyzed with the following results: (i) Zn: 33.27, 33.37 and 33.34% and (ii) Sn: 0.022, 0.025 and 0.026%. Calculate the standard deviation and the coefficient of variation for each analysis.

*Kandungan timah dan zink satu sampel loyang memberikan keputusan analisis seperti berikut: (i) Zn: 33.27, 33.37 dan 33.34% dan (ii) Sn: 0.022, 0.025 dan 0.026%. Kirakan sisihan piawai dan pekali variasi bagi setiap analisis.*

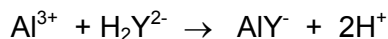
(20 marks/markah)

- [d] The following replicate molarities were obtained when standardizing a solution: 0.1067, 0.1071, 0.1066 and 0.1050. Can one of the results be discarded as due to accidental error at the 95% confidence level? Refer to Table B as in Appendix I.

*Bacaan-bacaan replikat kemolaran berikut diperolehi hasil dari pempiawaan satu larutan: 0.1067, 0.1071, 0.1066 dan 0.1050. Tunjukkan samada salah satu keputusan ini boleh disingkirkan disebabkan oleh ralat kecuaiian pada paras keyakinan 95%? Rujuk pada Jadual B dalam Lampiran I.*

(30 marks/markah)

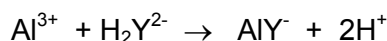
2. [a] Aluminum is determined by titrating with EDTA:



A 1.00 g sample requires 20.5 mL EDTA for titration. The EDTA was standardized by titrating 25.0 mL of a 0.100 M  $\text{CaCl}_2$  solution, requiring 30.0 mL EDTA. Calculate the percent  $\text{Al}_2\text{O}_3$  in the sample.

Given the atomic wt for Al = 27, O = 16 g/mol.

*Aluminum ditentukan melalui pentitratan dengan EDTA:*



*Satu sampel dengan berat 1.00 g memerlukan sebanyak 20.5 mL EDTA bagi pentitratan. Larutan EDTA ini telah dipiawaikan dengan mentitratkan 25.0 mL satu larutan 0.100 M  $\text{CaCl}_2$  yang memerlukan sebanyak 30.0 mL EDTA. Kirakan peratus  $\text{Al}_2\text{O}_3$  dalam sampel.*

*Diberikan JMR bagi Al = 27, O = 16 g/mol.*

(35 marks/markah)

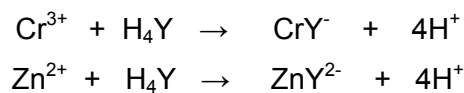
- [b] Chromium (III) is slow to react with EDTA ( $\text{H}_4\text{Y}$ ) and is therefore determined by back-titration. A mineral sample containing chromium (III) as impurity is analyzed by treating a 2.63 g sample with 5.00 mL of 0.0103 M EDTA. Following the reaction, the unreacted EDTA is back-titrated with 1.32 mL of 0.0122M zinc solution. What is the percent chromium chloride in the mineral sample?

*Kromium (III) bertindak balas secara perlahan dengan EDTA ( $\text{H}_4\text{Y}$ ). Dengan itu, ia ditentukan menggunakan kaedah pentitratan balik. Suatu sampel mineral mengandungi kromium (III) dianalisis dengan merawat 2.63 g sampel dengan 5.00 mL 0.0103 M EDTA. Selepas tindakbalas, EDTA yang tidak bertindakbalas dititratkan balik dengan 1.32 mL of 0.122 M larutan zink. Apakah peratus kromium klorida dalam sampel?*

...5/-

Reaction equation:

*Persamaan tindakbalas:*



Given: MWt.for  $\text{CrCl}_3 = 158.4 \text{ mg/mmol}$

*Diberikan: JMR bagi  $\text{CrCl}_3 = 158.4 \text{ mg/mmol}$*

(35 marks/markah)

[c] Calculate the equivalent weights of the following substances:

- (i)  $\text{NH}_3$
- (ii)  $\text{KMnO}_4$  [Mn(VII) is reduced to  $\text{Mn}^{2+}$ ]

*Kirakan berat ekuivalen bagi bahan-bahan berikut:*

- (i)  $\text{NH}_3$
- (ii)  $\text{KMnO}_4$  [ Mn(VII) diturunkan kepada  $\text{Mn}^{2+}$  ]

Given the At. wt of N = 14 and H = 1.0, K= 39, Mn = 54.9 g/mol

*Diberikan JMR bagi N= 14 dan H = 1.0, K= 39, Mn = 54.9 g/mol*

(30 marks/markah)

3. [a] Calculate the normality of a solution containing 5.267 g/L  $K_2Cr_2O_7$  (the Cr is reduced to  $Cr^{3+}$ ).

Given the At. wt of K = 39, Cr = 52 and K= 39 g/mol

*Kirakan kenormalan satu larutan mengandungi 5.267 g/L  $K_2Cr_2O_7$  (Cr diturunkan kepada  $Cr^{3+}$ ).*

*Diberikan JMR bagi K = 39, Cr = 52 dan K= 39 g/mol*

(25 marks/markah)

- [b] A 0.500 g sample is analyzed spectrophotometrically for manganese by dissolving it in acid and transferring to a 250 mL flask and diluting to volume. Three aliquots are analyzed by transferring 50mL portions with a pipet to 500 mL Erlenmeyer flasks and reacting with an oxidizing agent, potassium peroxydisulfate, to convert the manganese to permanganate. After reaction, these are quantitatively transferred to 250mL volumetric flasks, diluted to volume and measured spectrophotometrically. By comparison with standards, the average concentration in the final solution is determined to be  $1.25 \times 10^{-5}$  M. What is the percent manganese in the sample?

Given the At. Wt. of Mn = 54.9 g/mol.

Sebanyak 0.50 g sampel dianalisis secara spektrofotometri untuk penentuan kandungan mangan dengan pelarutan dalam asid dan pencairan ke 250 ml kelalang volumetrik. Tiga 50-mL sampel alikuot diambil dan dimasukkan ke dalam 500 mL kelalang Erlenmeyer dan ditindakbalaskan dengan agen pengoksidaan, kalium peroksidisulfat, untuk menukarkan mangan kepada permanganat.

Selepas tindakbalas, kesemua kandungan dalam kelalang Erlenmeyer dipindahkan secara kuantitatif ke dalam kelalang volumetrik 250mL, dicairkan ke tanda dan pengukuran spektrofotometri dijalankan. Perbandingan dengan larutan piawai memberikan purata kepekatan dalam larutan akhir sebanyak  $1.25 \times 10^{-5}$  M. Berapakah peratus mangan dalam sampel?

Diberikan JMR bagi Mn = 54.9 g/mol.

(40 marks/markah)

[c] Calculate the molarity of:

- (i) 70% HClO<sub>4</sub>, specific gravity 1.668
- (ii) 85 % H<sub>3</sub>PO<sub>4</sub>, specific gravity 1.689

Assuming that the density and specific gravity is equal.

Kirakan kemolaran bagi:

- (i) 70% HClO<sub>4</sub>, graviti spesifik 1.668
- (ii) 85 % H<sub>3</sub>PO<sub>4</sub>, graviti spesifik 1.689

Mengandaikan ketumpatan dan graviti spesifik adalah sama.

(35 marks/markah)

4. [a] Briefly explain the principles of adsorption indicators.

*Terangkan secara ringkas prinsip zat penunjuk penjerapan.*

(25 marks/markah)

- [b] A and B react as follows:  $A + B = 2C$ . The equilibrium constant is  $5.0 \times 10^6$ . If 0.40 mol of A and 0.70 mol of B are mixed in 1 L, what are the concentrations of A, B and C after reaction?

*A dan B bertindakbalas seperti berikut:  $A + B = 2C$ . Pemalar keseimbangan diberikan sebagai  $5.0 \times 10^6$ . Jika 0.40 mol A dan 0.70 mol B dicampurkan dalam 1 L, apakah kepekatan A, B dan C selepas tindakbalas?*

(25 marks/markah)

- [c] Hydrogen sulfide,  $H_2S$ , dissociates stepwise, with dissociation constants of  $9.1 \times 10^{-8}$  and  $1.2 \times 10^{-15}$ , respectively. Write the overall dissociation reaction and the overall equilibrium constant.

*Hidrogen sulfida,  $H_2S$ , terurai secara berperingkat, dengan pemalar penguraian  $9.1 \times 10^{-8}$  dan  $1.2 \times 10^{-15}$ , masing-masing. Secara keseluruhan, tuliskan persamaan tindakbalas penguraian dan pemalar keseimbangan.*

(25 marks/markah)

- [d] Calculate the equilibrium concentrations of A and B in a 0.10 M solution of weak electrolyte AB with an equilibrium constant of  $3.0 \times 10^{-6}$ .

Given:  $AB = A + B$

*Kirakan kepekatan pada keseimbangan bagi A dan B dalam satu larutan elektrolit lemah AB dengan pemalar keseimbangan  $3.0 \times 10^{-6}$ .*

*Diberikan:  $AB = A + B$*

(25 marks/markah)

...9/-

5. [a] Calculate the volume of concentrated ammonia and the weight of ammonium chloride required to prepare 100 mL of a buffer at pH 10.00 if the final concentration of salt is to be 0.200 M.

*Kirakan isipadu ammonia pekat dan berat ammonium klorida yang diperlukan untuk menyediakan 100 mL satu larutan tampan pada pH 10.00 jika kepekatan akhir garam adalah 0.200 M.*

(35 marks/markah)

- [b] Calculate the pH of a solution prepared by mixing 5.0 mL of 0.10M NH<sub>3</sub> with 10.0 mL of 0.020 M HCl. Given: K<sub>b</sub> (NH<sub>3</sub>) = 1.75 x 10<sup>-5</sup>.

*Kirakan pH satu larutan yang disediakan dengan mencampurkan 5.0 mL 0.10 M NH<sub>3</sub> dengan 10.0 mL 0.020 M HCl. Diberikan K<sub>b</sub> (NH<sub>3</sub>) = 1.75 x 10<sup>-5</sup>.*

(35 marks/markah)

- [c] Ammonium ions can be analyzed gravimetrically by precipitating with H<sub>2</sub>PtCl<sub>6</sub> as (NH<sub>4</sub>)<sub>2</sub>PtCl<sub>6</sub> and then igniting the precipitate to platinum metal, which is weighed. Calculate the percent ammonia in a 1.00 g sample that yields 0.100 g Pt by this method.

Given the At wt for Pt = 195.1 dan NH<sub>3</sub> = 17 g/mol

Reaction equation: (NH<sub>4</sub>)<sub>2</sub>PtCl<sub>6</sub> → Pt + 2 NH<sub>4</sub>Cl + 2 Cl<sub>2</sub>

*Ion ammonium boleh dianalisis secara gravimetrik dengan memendakkannya bersama H<sub>2</sub>PtCl<sub>6</sub> sebagai (NH<sub>4</sub>)<sub>2</sub>PtCl<sub>6</sub> dan menukarkan kepada logam platinum dengan cara pencucuhan dan beratnya ditimbang. Kirakan peratus ammonia dalam 1.00 g sampel yang akan menghasilkan sebanyak 0.100 g Platinum menggunakan kaedah ini.*

*Diberikan JMR Pt = 195.1 dan NH<sub>3</sub> = 17 g/mol*

*Persamaan Tindakbalas: (NH<sub>4</sub>)<sub>2</sub>PtCl<sub>6</sub> → Pt + 2 NH<sub>4</sub>Cl + 2 Cl<sub>2</sub>*

(30 marks/markah)

6. [a] Briefly states the steps commonly employed in an analytical procedure.

*Nyatakan secara ringkas langkah-langkah yang digunakan dalam tatacara analitis.*

(25 marks/markah)

- [b] Calculate the pH and pOH of a solution obtained by mixing equal volumes of a strong acid solution of pH 3.00 and a strong base solution of pH 12.00.

*Kirakan pH dan pOH satu larutan dengan mencampurkan isipadu yang sama satu larutan asid kuat dengan pH 3.00 dan satu larutan biasa kuat dengan pH 12.00.*

(35 marks/markah)

- [c] An ore is analyzed for the manganese content by converting the manganese to  $Mn_3O_4$  and weighing it. If a 1.52 g sample yields  $Mn_3O_4$  weighing 0.126 g, what would be the percent  $Mn_2O_3$  in the sample? What is the percent Mn?

Given: the At. Wt. of Mn = 54.9 and O = 16 g/mol

*Satu bijih dianalisis untuk mengetahui kandungan mangan dengan menukarkannya kepada  $Mn_3O_4$  dan menimbang beratnya. Jika satu sampel dengan berat 1.52 g menghasilkan 0.126 g  $Mn_3O_4$ , apakah peratus  $Mn_2O_3$  dalam sampel? Berapakah pula peratus Mn?*

*Diberikan: JMR bagi Mn = 54.9 dan O = 16 g/mol.*

(40 marks/markah)

7. [a] In emission and absorption measurements, there are three classes of interferences: Spectral, chemical and physical. Briefly discuss.

*Dalam pengukuran pemancaran dan penyerapan, ada tiga kelas gangguan: Spektral, kimia dan fizikal. Huraikan secara ringkas.*

(35 marks/markah)

- [b] Iron is reacted with 1,10-phenantroline in an acid media to form a colored complex. If a  $2.00 \times 10^{-5}$  M solution absorbs 35.0 % of the radiation at 510 nm, what would be the:

- (i) Absorbance  
(ii) Transmittance and percent absorption for a  $6.00 \times 10^{-5}$  M solution?

*Ferum bertindakbalas dengan 1,10-phenantrolin dalam media berasid untuk membentuk satu kompleks berwarna. Jika satu larutan  $2.00 \times 10^{-5}$  M menyerap sebanyak 35.0 % daripada sinaran pada 510 nm, apakah:*

- (i) Absorban  
(ii) Transmitan dan peratus penyerapan bagi satu larutan dengan kepekatan  $6.00 \times 10^{-5}$  M?

(25 marks/markah)

- [c] The data given below are obtained from the determination of traces of Pb in an ore sample by using the atomic absorption method.  
Calculate the concentration of Pb in the ore sample in ppm.

*Data berikut diperolehi dari penentuan unsur surih plumbum dalam satu sampel bijih menggunakan kaedah penyerapan atom.*

*Kirakan kepekatan Pb dalam sampel bijih tersebut dalam ppm.*

Table 7.1: Atomic absorption data at various concentration of Pb in an ore sample

*Jadual 7.1: Data penyerapan atom pada pelbagai kepekatan Pb dalam satu sampel bijih*

Concentration of Pb / <i>Kepekatan Pb</i> (ppm)	Absorbance / <i>Absorban</i>
0.00	0.000
2.00	0.053
3.80	0.104
5.80	0.160
8.00	0.220
9.60	0.260
11.20	0.310
Ore sample / <i>Sampel bijih</i>	0.200

(40 marks/*markah*)

**APPENDIX I / LAMPIRAN I**

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			
	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

<sup>a</sup> $\nu = N - 1 =$  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		
	$Q_{90}$	$Q_{95}$	$Q_{99}$
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

<sup>a</sup>Adapted from D. B. Rorabacher, *Anal. Chem.*, 63 (1991) 139.