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# UNIVERSITI SAINS MALAYSIA

First Semester Examination  
Academic Session 2012/2013

January 2013

## **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 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, the English version shall be used.

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

1. [a] The basicity constant,  $K_b$  for ammonia is  $1.75 \times 10^{-5}$  at  $25^\circ \text{C}$ . Calculate the pH and pOH for a  $1.00 \times 10^{-3} \text{ M}$  solution of ammonia.

*Pemalar basa,  $K_b$  bagi ammonia is  $1.75 \times 10^{-5}$  pada  $25^\circ \text{C}$ . Kirakan pH dan pOH bagi  $1.00 \times 10^{-3} \text{ M}$  larutan ammonia.*

(30 marks/markah)

- [b] Calculate pH of the solution after the addition of 50 mL and 60.0 mL of titrant.

*Kirakan pH larutan selepas penambahan 50 mL dan 60.0 mL titrant.*

(40 marks/markah)

- [c] Four-tenth gram samples of paint from a bridge, analyzed for the lead content by a precise method ( $<1\%$  rsd), gives a relative sampling precision,  $R$ , of 5%. What weight of sample should be taken to improve this to 2.5%?

*Empat persepuluh dari sampel cat dari satu jambatan, yang telah dianalisis bagi kandungan plumbum oleh satu kaedah yang persis ( $<1\%$  rsd), memberikan kepersisan pensampelan relatif,  $R$ , sebanyak 5%. Apakah berat sampel yang harus diambil untuk memperbaiki nilai ini kepada 2.5%?*

(30 marks/markah)

2. [a] Potassium dichromate is a high-purity material that is used as an oxidising agent in the volumetric determination of iron. However, it is frequently standardized by titrating with a known amount of iron (II) prepared from high purity iron wire or electrolytic iron. This is because the color of the iron (III) product of the titration tends to mask the indicator color used to detect the end of the titration causing a slight error.

A solution of 0.1012 M is prepared and standardized with the following results: 0.1017, 0.1019, 0.1016, 0.1015 M. Is the supposition that the titration values are statistically different from the actual prepared concentration is valid?

*Kalium dikromat adalah satu bahan yang tinggi ketulenannya yang digunakan sebagai agen pengoksidaan dalam penentuan volumetrik bagi ferum. Walaubagaimanapun, ia kerap dipiawaikan dengan mentitratkannya dengan amaun Fe (II) yang diketahui yang disediakan dari dawai ferum berketulanan tinggi atau ferum hasil dari proses elektrolitik. Ini adalah kerana warna produk ferum (III) dari pentitratan boleh mengaburi warna zat penunjuk yang digunakan untuk menentukan takat akhir menyebabkan sedikit ralat.*

*Satu larutan 0.1012 M disediakan dan dipiawaikan dengan keputusannya adalah seperti berikut: 0.1017, 0.1019, 0.1016, 0.1015 M. Adakah tanggapan bahawa nilai-nilai pentitratan ini berbeza secara statistik dari kepekatan sebenar itu boleh diterima?*

(45 marks/markah)

- [b] What is the molar solubility of  $PbI_2$ , in g/L, if the solubility product is  $7.1 \times 10^{-9}$ ?

Given that: At. Wt Pb = 207.2 and I = 126.9 g/mol

*Apakah molar keterlarutan bagi  $PbI_2$  dalam g/L, jika hasil darab keterlarutan adalah  $7.1 \times 10^{-9}$ ?*

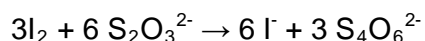
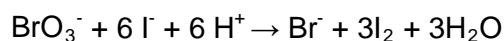
*Diberikan: JAR bagi Pb = 207.2 dan I = 126.9 g/mol*

(30 marks/markah)

- [c] A solution of  $Na_2S_2O_3$  is standardized iodometrically against 0.1262 g of high purity  $KBrO_3$ , requiring 44.97 mL  $Na_2S_2O_3$ . What is the molarity of the  $Na_2S_2O_3$ ?

Given: At wt Br = 79.9, K = 39.1 and O = 16 g/mol

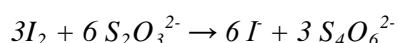
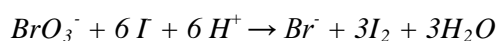
The Reactions are:



*Satu larutan  $Na_2S_2O_3$  telah dipiawaikan secara iodometri dengan 0.1262 g  $KBrO_3$  tulen, memerlukan sebanyak 44.97 mL  $Na_2S_2O_3$ . Apakah kemolaran  $Na_2S_2O_3$ ?*

*Diberikan, JAR bagi Br = 79.9, K = 39.1 dan O = 16 g/mol*

*Tindakbalas:*



(25 marks/markah)

3. [a] A and B reacts 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 keseimbangannya adalah  $5.0 \times 10^6$ . Jika 0.40 mol A dan 0.70 mol B dicampurkan dalam 1 L, apakah kepekatan A, B dan C selepas tindakbalas?*

*(40 marks/markah)*

- [b] Calculate the volume of concentrated ammonia and the weight of ammonium chloride that you have to take to prepare 100 mL of a buffer at pH 10.00 if the final concentration of salt is to be 0.200 M.

Given: Molarity of concentrated ammonia is 14.8 M.

Formula wt.  $\text{NH}_4\text{Cl} = 53.5 \text{ g/mol}$ ,  $K_b (\text{NH}_3) = 1.75 \times 10^{-5}$

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

*Diberikan: Kemolaran ammonia pekat adalah 14.8 M.*

*Berat formula  $\text{NH}_4\text{Cl} = 53.5 \text{ g/mol}$ ,  $K_b (\text{NH}_3) = 1.75 \times 10^{-5}$*

*(40 marks/markah)*

- [c] Distinguish between iodimetry and iodometry.

*Bezakan di antara iodimetri dan iodometri.*

*(20 marks/markah)*

4. [a] Calculate the titer of 0.100 M EDTA in mg  $\text{CaCO}_3/\text{mL}$ .

*Kirakan titer bagi 0.100 M EDTA dalam mg  $\text{CaCO}_3/\text{mL}$ .*

*(20 marks/markah)*

- [b] A solution (100 mL) containing 325 ppm  $\text{K}^+$  is analyzed by precipitating it as the tetraphenyl borate,  $\text{K}(\text{C}_6\text{H}_5)_4\text{B}$ , dissolving the precipitate in acetone solution and measuring the concentration of tetraphenyl borate ion,  $(\text{C}_6\text{H}_5)_4\text{B}^-$ , in the solution. If the acetone solution is 250 mL, what is the concentration of the tetraphenyl borate in ppm?

Given: At wt B = 10.8, C = 12; K = 39 and H = 1.0 g/mol

*Satu larutan (100 mL) mengandungi 325 ppm  $\text{K}^+$  dianalisa dengan memendakkannya sebagai tetrafenil borat,  $\text{K}(\text{C}_6\text{H}_5)_4\text{B}$ , kemudian melarutkan mendakan dalam larutan aseton dan mengukur kepekatan ion tetrafenil borat,  $(\text{C}_6\text{H}_5)_4\text{B}^-$ , dalam larutan. Jika larutan aseton adalah 250 mL, apakah kepekatan tetrafenil borat dalam ppm?*

*Diberikan: JAR bagi B = 10.8, C = 12; K = 39 dan H = 1.0 g/mol*

*(40 marks/markah)*

- [c] Replicate samples of a silver alloy are analyzed and determined to contain 95.67, 95.61 and 95.60% Ag.

Calculate the:

- (i) standard deviation of the mean
- (ii) relative standard deviation of the mean (in percent) of the individual results

*Sampel replikat satu aloi perak telah dianalisis dan ditentukan mengandungi 95.67, 95.61 dan 95.60% Ag.*

*Kirakan:*

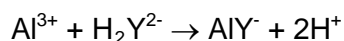
- (i) *sisihan piawai bagi purata*
  - (ii) *sisihan piawai relatif bagi purata (dalam peratus) keputusan individu*
- (40 marks/markah)*

5. [a] A soda ash sample is analyzed in the laboratory by titration with standard hydrochloric acid. The analysis is performed in triplicate with the following results: 93.50, 93.58 and 93.43 % Na<sub>2</sub>CO<sub>3</sub>. Within what range are you confident that the true value lies? Refer to Table A as in Appendix.

*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 % Na<sub>2</sub>CO<sub>3</sub>. Carikan julat di mana anda yakin bahawa nilai sebenarnya berada? Rujuk pada Jadual A dalam Lampiran.*

*(25 marks/markah)*

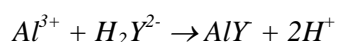
[b] Aluminum is determined by titrating with EDTA:



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*

(50 marks/markah)

[c] Calculate the percentage of copper by mass in a sample of rock containing 0.50% by mass of chalcopyrite, assuming this is the only source of copper in the rock.

Given: At. wt. : Cu = 63.55; Fe = 55.85; S = 32.07g/mol)

*Kirakan peratus jisim kuprum dalam satu sampel batuan yang mengandungi 0.50 % berat kalkopirit, dengan menganggapkan ia adalah satu-satunya sumber kuprum dalam batuan.*

*Diberikan: At. wt. : Cu = 63.55; Fe = 55.85; S = 32.07g/mol)*

(25 marks/markah)



6. [a] Define what monochromators are and explain why monochromators are needed in analytical equipments.

Berikan definisi monokromator dan jelaskan mengapa monokromator diperlukan dalam peralatan analitis.

(10 marks/markah)

- [b] With the aid of a table, compare THREE types of monochromators typically used in analytical equipments.

*Dengan bantuan jadual, bandingkan TIGA jenis monokromator yang lazimnya digunakan dalam peralatan analitis.*

(15 marks/markah)

- [c] Describe in detail how instrumental factors may contribute to deviations in Beer's Law.

*Terangkan dengan terperinci bagaimana faktor peralatan boleh menyumbang kepada sisihan dalam Hukum Beer.*

(10 marks/markah)

- [d] Distinguish the types and the known operational characteristics of the sources of continuous radiation in a spectrometer, for visible, ultraviolet, infrared regions and fluorescence spectrometry.

*Bezakan jenis-jenis dan ciri-ciri operasi bagi sumber radiasi berterusan dalam sebuah spektrofotometer bagi bahagian sinar nampak, ultra-ungu, inframerah dan spektrometri pendarfluor.*

(20 marks/markah)

- [e] A sample in a 2 cm cell was determined using a spectrometer. The sample allows 80% transmission of light at a certain wavelength. If the sample's absorptivity is  $2.0 \text{ cm}^{-1} \text{ g}^{-1} \text{ L}$  at the measured wavelength, calculate the concentration of the sample.

*Satu sampel dalam 2 cm sel ditentukan dengan menggunakan satu spektrometer. Sampel tersebut membenarkan penghantaran 80% cahaya pada jarakgelombang tertentu. Jika absorptiviti bahan ini adalah  $2.0 \text{ cm}^{-1} \text{ g}^{-1} \text{ L}$  pada jarakgelombang yang diukur, kirakan kepekatan bahan tersebut.*

(15 marks/markah)

- [f] Chloroaniline in a sample is determined as amine picrate, which absorbs strongly at the wavelength of 359 nm ( $\epsilon = 1.25 \times 10^4 \text{ cm}^{-1} \text{ mol}^{-1} \text{ L}$ ). A sample of 0.0265 g was reacted with picric acid and was diluted to 1 L. The absorbance reading is 0.368, in a 2 cm cell. With the provided information, calculate the percentage of chloroaniline in the sample. ( $MW_{\text{chloroaniline}} = 127.6 \text{ g/mol}$ ).

*Kloroanilin dalam satu sampel ditentukan sebagai amina pikrat yang menyerap kuat pada jarakgelombang 359 nm ( $\epsilon = 1.25 \times 10^4 \text{ cm}^{-1} \text{ mol}^{-1} \text{ L}$ ). Sebanyak 0.0265 g sampel ditindakbalaskan dengan asid pikrik dan dicairkan kepada 1 L. Bacaan absorbans larutan adalah 0.368 dalam sel 2 cm. Dengan maklumat tersebut, kirakan peratus kloroanilin dalam sampel ini. ( $BM_{\text{kloroanilin}} = 127.6 \text{ g/mol}$ ).*

(30 marks/markah)

7. [a] The burner is an essential component in an atomic absorption spectrophotometer (AAS). With an aid of a table, compare TWO different types of burners used in AAS in terms of design and operation, listing down as well the advantages and disadvantages of each burner.

*Pembakar ialah komponen yang penting dalam sesebuah spektrofotometer penyerapan atom (SPA). Dengan bantuan jadual, bandingkan DUA jenis pembakar berbeza yang digunakan dalam SPA dari segi rekabentuk dan operasi, serta senaraikan kelebihan serta kekurangan setiap pembakar tersebut.*

(20 marks/markah)

- [b] Explain why the use of organic solvents may be advantageous in AAS analysis.

*Terangkan mengapa penggunaan pelarut organik mungkin boleh memberikan kelebihan dalam analisis SPA.*

(20 marks/markah)

- [c] Justify the need for using 'internal standard' and 'standard addition calibration' techniques in improving precision and minimizing errors in analytical methods.

*Berikan justifikasi penggunaan teknik-teknik 'piawai dalaman' dan 'kalibrasi piawai penambahan' bagi meningkatkan kepersisan dan meminimakan sisihan dalam kaedah analitis.*

(20 marks/markah)

- [d] A sample of chemical X is analyzed for element Y by flame emission spectrometry. Using the method of standard additions, 2 aliquots of 0.5 mL, each added with 5.0 mL of water were prepared. A 10  $\mu$ L portion of 0.005 M Y-chloride (with a molecular formula of YCl) was added to second aliquot. The net emission signals in arbitrary units for the aliquots were 32.1 and 58.6, respectively. Determine the concentration of Y in chemical X.

*Sampel bahan kimia X telah dianalisa untuk unsur Y dengan menggunakan spektometri emisi pembakaran. Dengan menggunakan kaedah piawaian penambahan, 2 alikuat 0.5 mL, setiap satunya ditambahkan dengan 5.0 mL air telah disediakan. Sebanyak 10 mL 0.005 M klorida-Y (dengan formula molekul YCl) telah ditambahkan kepada alikuat kedua. Isyarat emisi dalam unit arbitrari bagi alikuat tersebut ialah 32.1 dan 58.6, masing-masing. Tentukan kepekatan Y dalam bahan kimia X.*

(40 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*

$\nu$	<b>Confidence Level</b> (Paras Keyakinan)			
	<b>90 %</b>	<b>95 %</b>	<b>99 %</b>	<b>99.5 %</b>
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

\*  $\nu = N - 1 = \text{degrees of freedom}$