
UNIVERSITI SAINS MALAYSIA

Peperiksaan Semester Pertama
Sidang Akademik 2006/2007
*First Semester Examination
Academic Session 2006/2007*

Oktober/November 2006

EBS 336/3 - Kimia Analitis
EBS 336/3 - Analytical Chemistry

Masa : 3 jam
Time : 3 hours

Sila pastikan bahawa kertas peperiksaan ini mengandungi DUA BELAS muka surat beserta SATU muka surat (Lampiran) yang bercetak sebelum anda memulakan peperiksaan.

Kertas soalan ini mengandungi TUJUH soalan.

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

Mulakan jawapan anda untuk setiap soalan pada muka surat yang baru.

Semua soalan mesti dijawab dalam Bahasa Malaysia.

Please check that this examination paper consists of TWELVE pages of printed material and ONE page APPENDIX before you begin the examination.

This paper contains SEVEN questions.

Answer any FIVE questions. If a candidate answers more than five questions, only the first five answers will be examined and awarded marks.

Answer to any question must start on a new page.

All questions must be answered in Bahasa Malaysia.

1. [a] Cadangkan satu kaedah untuk menjalankan analisis berikut:
- (i) Kandungan klorida dalam satu larutan garam
 - (ii) Kandungan aluminium dalam satu bijih mengandungi aluminium
- (30 markah)
- [b] Kirakan pH selepas penambahan 25.0, 50.0 mL dan 60.0 ml isipadu titrant dalam pentitratan 50.0 mL 0.100 M asid asetik dengan 0.100 M NaOH. **Diberikan:** $K_a(\text{CH}_3\text{COOH}) = 1.75 \times 10^{-5}$
- (30 markah)
- [c] Kehadiran kuprum dalam air sisa berhampiran dengan loji perawatan kumbahan ditentukan pada mulanya dengan cara pemisahan dan pengkonsentran melalui pengekstrakan pelarut dengan mengekstrak masuk ke dalam metilena klorida menggunakan pengkelet dithiozon pada pH 3. Ini diikuti dengan proses penyejatan pelarut, pengabuan (*ashing*) bahan pengkelet untuk menyingkirkan bahagian organik dan mentitratkan kuprum dengan EDTA. Setiapnya tiga 1-L bahagian sampel diekstrakkan dengan 25-mL bahagian metilena klorida dan ekstrak ini dicampurkan dalam satu kelalang volumetrik 100-mL dan dicairkan ke isipadu. Satu 50-mL alikuot disejatkan, diabukan dan dititratkan. Jika larutan EDTA memberikan titer CaCO_3 sebanyak 2.69 mg/mL and 2.67 mL diperlukan untuk pentitratan kuprum, apakah kepekatan kuprum dalam air sisa dalam bahagian per juta (ppm)?
- Diberikan:** Berat formula $\text{CaCO}_3 = 100.1 \text{ g/mol}$, Berat atom Cu = 63.5 g/mol
- (40 markah)

2. [a] Bezakan antara had keyakinan, julat keyakinan dan paras keyakinan.
(10 markah)
- [b] Kepekatan kuprum dalam satu sampel bijih adalah 3% (wt/wt). Berapakah sampel yang harus dianalisiskan untuk memperolehi peratus sisihan piawai relatif sebanyak 5% dalam keputusan analitis pada paras keyakinan 95%, jika kepersisan pensampelan adalah 0.15% (wt/wt)?
Sila rujuk **Jadual-Jadual** dalam Lampiran.
(20 markah)
- [c] Sampel replikat bagi satu aloi argentum telah dianalisiskan dan ditentukan kandungan Ag sebanyak: 95.67, 95.61, 95.71 dan 95.60 % Ag.
Kirakan:
(i) Sisihan piawai
(ii) Sisihan piawai bagi purata
(iii) Sisihan piawai relatif bagi purata (dalam peratus) bagi keputusan individu.
(20 markah)

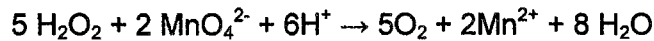
- [d] Satu lengkuk kalibrasi bagi penentuan fosforus dalam satu sampel sisa disediakan dengan melakukan tindakbalas satu larutan piawai fosfat dengan molibdenum (VI) dan penurunan kompleks asid fosfomolibdik tersebut untuk menghasilkan warna biru ciri. Absorban A yang diukur diplotkan melawan kepekatan fosforus. Dari data berikut, tentukan garisan "*linear least squares*" dan kirakan kepekatan fosforus dalam sampel sisa tersebut. Gunakan kertas graf untuk melukis dengan jitu garisan lurus tersebut.

P (ppm)	A (Abs)
1.00	0.205
2.00	0.410
3.00	0.615
4.00	0.820
Sampel Sisa	0.625

(50 markah)

3. [a] Bezakan antara satu teknik, kaedah, tatacara dan protokol.
(20 markah)
- [b] (i) Satu 2.60 g sampel sisa telah dianalisiskan dan didapati mengandungi 3.6 μg zink. Apakah kepekatan zink dalam sisa dalam unit ppm dan ppb?
(20 markah)
- (ii) Kepekatan zink dalam satu sampel adalah 1 ppm. Apakah kepekatan zink ini dalam unit meq/L.
Diberikan: Berat atom Zinc: 65.4 g/mol
(20 markah)

- [c] Dalam larutan berasid, kalium permanganat bertindakbalas dengan H_2O_2 membentuk Mn^{2+} :



Dalam larutan neutral, ia bertindakbalas dengan MnSO_4 membentuk MnO_2 :



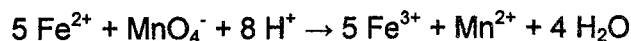
Kirakan bilangan millimeters 0.100 M KMnO_4 yang akan bertindakbalas dengan 50.0 mL 0.200 M H_2O_2 dan dengan 50.0 mL 0.200 M MnSO_4 .

(40 markah)

4. [a] Satu larutan kalium permanganat disediakan dengan melarutkan sebanyak 4.68 g KMnO_4 dalam air dan mencairkan kepada isipadu 500 mL. Berapakah milliliters yang akan bertindakbalas dengan ferum dalam 0.500 g satu bijih yang mengandungi sebanyak 35.6 % Fe_2O_3 ?

Diberikan: Berat formula $\text{KMnO}_4 = 158.0 \text{ g/mol}$, $\text{Fe}_2\text{O}_3 = 159.7 \text{ g/mol}$

Persamaan Tindakbalas:



(30 markah)

- [b] Ketulenan satu 0.287 g sampel $\text{Zn}(\text{OH})_2$ telah ditentukan dengan mentitratkannya dengan satu larutan piawai HCl . Pentitratan ini memerlukan sebanyak 37.8 mL. Larutan HCl ini telah dipiawai dengan memendakkan AgCl dalam 25.0 mL alikuot dan menimbang mendakan ini. Berat mendakan AgCl yang diperolehi adalah sebanyak 0.462 g. Apakah ketulenan $\text{Zn}(\text{OH})_2$?

Diberikan: Berat formula $\text{AgCl} = 143 \text{ g/mol}$, $\text{Zn}(\text{OH})_2 = 99.4 \text{ g/mol}$

(40 markah)

- [c] Zink ditentukan dengan memendakkan dan menimbanginya sebagai $Zn_2Fe(CN)_6$.

Diberikan:

Berat atom Zn = 65.4 g/mol, Fe = 56 g/mol, C = 12 g/mol, N = 14 g/mol

- (i) Apakah berat zink yang terkandung dalam satu sampel yang menghasilkan mendakan sebanyak 0.348 g?
 (ii) Apakah berat mendakan yang akan terbentuk dari 0.50 g zink?

(30 markah)

5. [a] A dan B bertindakbalas seperti berikut: $A + B = 2C$.

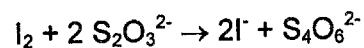
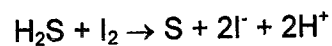
Pemalar keseimbangannya adalah 5.0×10^{-6} . Jika 0.40 mol A dan 0.70 mol B dicampurkan dalam 1 L, apakah kepekatan A, B dan C selepas tindakbalas?

(30 markah)

- [b] Kandungan sulfur dalam sampel keluli ditentukan dengan menukarkan ia kepada gas H_2S dan menjerap gas ini dalam 10.0 mL of 0.005 M I_2 dan mentitratkan balik I_2 berlebihan dengan 0.002 M Na_2SO_3 .
 Jika 2.6 mL Na_2O_3 diperlukan untuk pentitratan, berapakah milligram sulfur yang terkandung dalam sampel?

Diberikan: Berat atom S = 32.06 g/mol

Persamaan Tindakbalas:



(30 markah)

- [c] 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 NH_4Cl = 53.5 g/mol, $K_b(NH_3) = 1.75 \times 10^{-5}$

(40 markah)

6. [a] 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 markah)
- [b] Apakah dia satu spektrofotometer atau spektrometer?
Dengan bantuan gambarajah, huraikan secara ringkas fungsi semua komponen peralatan yang ada dalam satu spektrofotometer.
(40 markah)
- [c] Takrifkan absorptiviti dan absorptiviti molar. Satu sebatian dengan berat formula 180 mempunyai absorptiviti sebanyak $286 \text{ cm}^{-1} \text{ g}^{-1} \text{ L}$. Apakah absorptiviti molarnya?
(30 markah)
7. [a] Lukiskan satu gambarajah skema satu peralatan penyerapan atom. Huraikan secara ringkas prinsip satu spektrofotometri penyerapan atom.
(30 markah)
- [b] Sumber gangguan dalam pengukuran penyerapan dan pemancaran boleh dikelaskan kepada gangguan spektral, kimia dan fizik. Bincangkan secara ringkas empat contoh gangguan-gangguan tersebut.
Mengapakah kepekatan yang tinggi larutan garam kalium kadang-kadang ditambahkan kepada larutan piawai dan larutan sampel dalam kaedah penyerapan atau pemancaran nyalaan?
(50 markah)
- [c] Dengan bantuan gambarajah-gambarajah, bandingkan antara operasi satu spektrofotometer bim tunggal dan spektrofotometer bim dubel.
(20 markah)

1. [a] *Suggest a method to accomplish the following analyses:*
- (i) *The Chloride content in a salt solution*
 - (ii) *The Aluminum content in an ore containing aluminum*
- (30 marks)
- [b] *Calculate the pH at 25.0, 50.0 mL and 60.0 ml addition of titrant in the titration of 50.0 mL of 0.100 M acetic acid with 0.100 M NaOH.*
- Given:** $K_a(\text{CH}_3\text{COOH}) = 1.75 \times 10^{-5}$
- (30 marks)
- [c] *The copper present in waste water near the discharge of a sewage treatment plant is determined by first separating and concentrating it by solvent extraction of its dithiozone chelate at pH 3 into methylene chloride and then evaporating the solvent, ashing the chelate to destroy the organic portion and titrating the copper with EDTA. Three 1-L portions of the sample are each extracted with 25 -mL portions of methylene chloride and the extracts are combined in a 100-mL volumetric flask and diluted to volume. A 50-mL aliquot is evaporated, ashed and titrated. If the EDTA solution has a CaCO_3 titer of 2.69 mg/mL and 2.67 mL is required for titration of the copper, what is the concentration of copper in the waste water in parts per million?*
- Given:** Formula wt. $\text{CaCO}_3 = 100.1 \text{ g/mol}$, At. Wt. $\text{Cu} = 63.5 \text{ g/mol}$
- (40 marks)
2. [a] *Distinguish between confidence limit, confidence interval and confidence level.*
- (10 marks)
- [b] *Copper in an ore sample is at a concentration of about 3 % (wt/wt). How many samples should be analyzed to obtain a percent relative standard deviation of 5% in the analytical result at 95% confidence level, if the sampling precision is 0.15 % (wt/wt)?*
- Please refer to Table in the Appendix*
- (20 marks)

[c] *Replicate samples of a silver alloy are analyzed and determined to contain 95.67, 95.61, 95.71 and 95.60 % Ag. Calculate*

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

(20 marks)

[d] *A calibration curve for the colorimetric determination of phosphorous in a waste sample is prepared by reacting standard solution of phosphate with molybdenum (VI) and reducing the phosphomolybdic acid complex to produce the characteristic blue color. The measured absorbance A is plotted against the concentration of phosphorous. From the following data, determine the linear least-squares line and calculate the phosphorus concentration in the waste sample. Use a graph paper to accurately draw the straight lines.*

<i>P (ppm)</i>	<i>A (Abs)</i>
1.00	0.205
2.00	0.410
3.00	0.615
4.00	0.820
<i>Sampel Sisa</i>	0.625

(50 marks)

3. [a] *Distinguish between a technique, a method, a procedure and a protocol.*

(20 marks)

[b] (i) *A 2.60 g sample of wastes was analyzed and found to contain 3.6 μg Zinc. What is the concentration of zinc in the waste in ppm and ppb?*

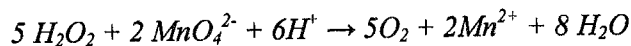
(20 marks)

- (ii) The concentration of zinc in a sample solution is about 1 ppm.
Express this as meq/L.

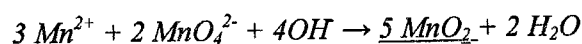
Given: At. Wt. Zinc : 65.4 g/mol

(20 marks)

- [c] In acid solution, potassium permanganate reacts with H_2O_2 to form Mn^{2+} :



In neutral solution it reacts with $MnSO_4$ to form MnO_2 :

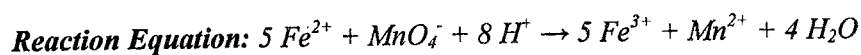


Calculate the number of millimeters of 0.10 M $KMnO_4$ that will react with 50.0 mL of 0.200 M H_2O_2 and with 50.0 mL of 0.200 M $MnSO_4$.

(40 marks)

4. [a] A potassium permanganate solution is prepared by dissolving 4.68 g $KMnO_4$ in water and diluting to 500 mL. How many milliliters of this react with the iron in 0.500 g of an ore containing 35.6 % Fe_2O_3 ?

Given: Formula Wt. $KMnO_4 = 158.0$ g/mol, $Fe_2O_3 = 159.7$ g/mol



(30 marks)

- [b] The purity of a 0.287 g sample of $Zn(OH)_2$ is determined by titrating with a standard HCl solution, requiring 37.8 mL. The HCl solution was standardized by precipitating AgCl in a 25.0 mL aliquot and weighing (0.462 g AgCl obtained). What is the purity of the $Zn(OH)_2$?

Given : Formula Wt. AgCl = 143 g/mol, $Zn(OH)_2 = 99.4$ g/mol

(40 marks)

- [c] Zinc is determined by precipitating and weighing as $Zn_2Fe(CN)_6$.

Given: At. Wt. Zn = 65.4 g/mol, Fe = 56 g/mol, C = 12 g/mol, N = 14 g/mol

(i) What weight of zinc is contained in a sample that gives 0.348 g precipitate?

(ii) What weight of precipitate would be formed from 0.50 g of zinc?

(30 marks)

5. [a] A and B reacts as follows: $A + B = 2C$.

The equilibrium constant is 5.0×10^{-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?

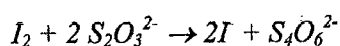
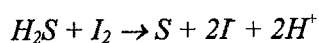
(30 marks)

[b] The sulfur content of a steel sample is determined by converting it to H_2S gas, absorbing the H_2S in 10.0 mL of 0.005 M I_2 and then back-titrating the excess I_2 with 0.002 M Na_2O_3 .

If 2.6 mL Na_2O_3 is required for the titration, how many milligrams of sulfur are contained in the sample?

Given: At. Wt. S = 32.06 g/mol

Reaction Equation:



(30 marks)

[c] 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. $NH_4Cl = 53.5$ g/mol, $K_b(NH_3) = 1.75 \times 10^{-5}$

(40 marks)

6. [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 mechanism involve with the use of each indicator. Please include chemical equations where necessary.
- (30 marks)
- [b] What is a spectrophotometer or a spectrometer?
With the help of a diagram, describe briefly the function of all instrument component present in a spectrophotometer.
- (40 marks)
- [c] Define absorptivity and molar absorptivity.
A compound of formula weight 180 has an absorptivity of $286 \text{ cm}^{-1} \text{ g}^{-1} \text{ L}$.
What is its molar absorptivity?
- (30 marks)
7. [a] Draw a schematic diagram of an atomic absorption instrument. Describe briefly the principles of an atomic absorption spectrophotometry.
- (25 marks)
- [b] The sources of interference in absorption and emission measurements can be classified into spectral, chemical and physical interferences. Discuss briefly all four examples of these type of interferences.
- Why is a high concentration of a potassium salt is sometimes added to standards and samples in flame absorption or emission methods?
- (50 marks)
- [c] With the help of diagrams, compare the operations of a single-beam spectrophotometer and a double-beam spectrophotometer.
- (25 marks)

LAMPIRAN**APPENDIX****Jadual 2.1****Values of t for ν Degrees of Freedom for Various Confidence Levels^a**

ν	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
∞	1.645	1.960	2.576	2.807

^a $\nu = N - 1 =$ degrees of freedom.**Jadual 2.2****Values of F at the 95% Confidence Level**

	$\nu_1 = 2$	3	4	5	6	7	8	9	10	15	20	30
$\nu_2 = 2$	19.0	19.2	19.2	19.3	19.3	19.4	19.4	19.4	19.4	19.4	19.4	19.5
3	9.55	9.28	9.12	9.01	8.94	8.89	8.85	8.81	8.79	8.70	8.66	8.62
4	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00	5.96	5.86	5.80	5.75
5	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.77	4.74	4.62	4.56	4.50
6	5.14	4.76	4.53	4.39	4.28	4.21	4.15	4.10	4.06	3.94	3.87	3.81
7	4.74	4.35	4.12	3.97	3.87	3.79	3.73	3.68	3.64	3.51	3.44	3.38
8	4.46	4.07	3.84	3.69	3.58	3.50	3.44	3.39	3.35	3.22	3.15	3.08
9	4.26	3.86	3.63	3.48	3.37	3.29	3.23	3.18	3.14	3.01	2.94	2.86
10	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02	2.98	2.85	2.77	2.70
15	3.68	3.29	3.06	2.90	2.79	2.71	2.64	2.59	2.54	2.40	2.33	2.25
20	3.49	3.10	2.87	2.71	2.60	2.51	2.45	2.39	2.35	2.20	2.12	2.04
30	3.32	2.92	2.69	2.53	2.42	2.33	2.27	2.21	2.16	2.01	1.93	1.84