

**PART A / BAHAGIAN A**

- (1). (a). Describe general rules for the use of electronic balance and distinguish zero point and rest point.

*Huraikan peraturan am untuk penggunaan imbangan elektronik dan bezakan titik sifar dan titik rehat*

(6 marks/markah)

- (b).  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}(\text{s})$  is a primary standard substance. A 2.3688 g of oxalic acid dihydrate were completely neutralized by 42.56 ml of NaOH solution.

*$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}(\text{s})$  ialah bahan piawai utama. Satu 2.3688 g asid oksalik dihidrat telah dineutralaskan sepenuhnya oleh 42.56 ml larutan NaOH.*

- (i). Define a primary standard substance.

*Takrifkan bahan piawai utama.*

(2 marks/markah)

- (ii) Define a standard solution.

*Takrifkan larutan piawai.*

(2 marks/markah)

- (iii) Write a balanced equation for the reaction above.

*Tulis persamaan seimbang bagi tindak balas di atas.*

(3 marks/markah)

- (iv) Calculate the molar concentration of the NaOH solution.

*Kira kepekatan molar larutan NaOH.*

(3 marks/markah)

Given/Diberi:

Atomic weight of /Berat atom H =1; C = 12; Na = 22.9, O =16 g/mol

- (c). Figure 1 show the procedure of ~0.1 M HCL solution standardization (prepared from 38 % HCL with density 1.19 g/mL).

*Rajah 1 menunjukkan prosedur ~0.1 M pempiawaian larutan HCL (disediakan daripada 38 % HCL dengan ketumpatan 1.19 g/mL).*

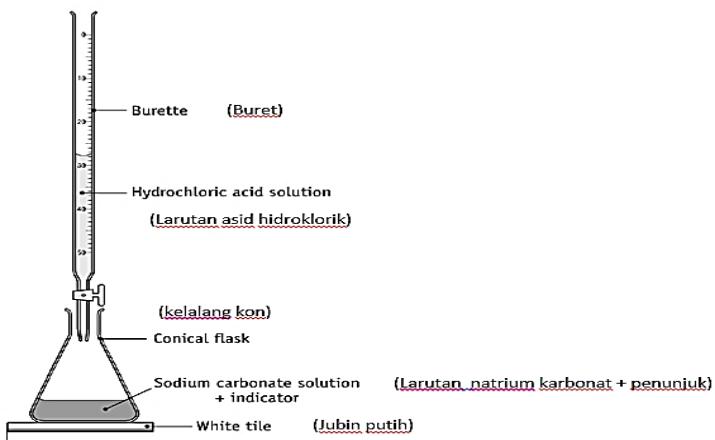


Figure 1: Titration procedure of ~0.1 M HCL solution standardization

*Figure 1: Prosedur pentitratan larutan pempiawaian ~0.1 M HCL*

Table 1: Chemicals and apparatus in the standardization of an acid

*Jadual 1: Bahan kimia dan radas dalam pempiawaian satu asid*

Chemicals (Bahan-bahan Kimia)	Dry sodium carbonate (prepared 0.1 M in 50 ml volumetrik flask), methyl orange indicator, ~0.1M HCL and disitilled water. <i>Natrium karbonat (disediakan 0.1M dalam 50 ml kelalang volumetrik), penunjuk metil jingga, ~0.1M HCL dan air suling.</i>
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Atomic weight/ Berat atom (g/mol): Na: 23; C: 41; O:16

- (i). WRITE a procedure of standardization of HCL using Na<sub>2</sub>CO<sub>3</sub> solution. Refer Table 1, for detail chemical and apparatus use.

*TULIS prosedur pempiawaian HCL dengan menggunakan larutan Na<sub>2</sub>CO<sub>3</sub>. Rujuk Jadual 1, untuk perincian penggunaan bahan kimia dan radas.*

(3 marks/markah)

- (ii). What are the specifications of secondary standard solution?

*Apakah spesifikasi larutan piawai sekunder?*

(2 marks/markah)

- (iii). Calculate weight of Na<sub>2</sub>CO<sub>3</sub> required if you want to prepare 100 ml, 0.1 M Na<sub>2</sub>CO<sub>3</sub> solution?

*Kira berat Na<sub>2</sub>CO<sub>3</sub> yang diperlukan jika anda ingin menyediakan 100 ml, 0.1 M larutan Na<sub>2</sub>CO<sub>3</sub>?*

(2 marks/markah)

- (iv). A 20.00 mL sample of HCl was titrated with the 0.1M NaOH solution. To reach the endpoint required 23.72 mL of the NaOH. Calculate the molarity of the HCl.

*Satu sampel 20.00 mL HCl telah dititrat dengan larutan 0.1M NaOH. Untuk mencapai titik akhir diperlukan 23.72 mL NaOH. Kira kemolaran HCl.*

(2 marks/markah)

- (2). (a). Explain the method of back-titration.

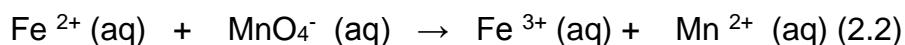
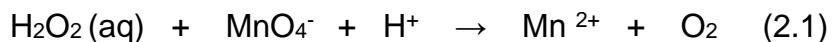
*Terangkan kaedah pentitratan-balik.*

(3 marks/markah)

- (b). A hydrogen peroxide solution is analyzed by adding a slight excess of standard  $\text{KMnO}_4$  solution and back titrating the unreacted  $\text{KMnO}_4$  with standard  $\text{Fe}^{2+}$  solution. A 0.587 g sample of the  $\text{H}_2\text{O}_2$  solution is taken, 25.0 mL of 0.0215 M  $\text{KMnO}_4$  is added and the titration requires 5.10 mL of 0.112 M  $\text{Fe}^{2+}$  solution.

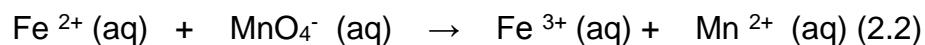
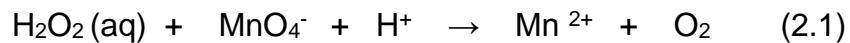
(Given: Atomic weight for Fe = 55.85, H = 1.0, O = 16.0 and Mn = 54.9 g/mol)

Reaction Equation:



Satu larutan hidrogen peroksida dianalisis dengan menambahkan sedikit berlebihan larutan piawai  $\text{KMnO}_4$  dan  $\text{KMnO}_4$  yang tidak bertindakbalas dititrat-balik dengan larutan piawai  $\text{Fe}^{2+}$ . Satu sampel dengan berat 0.587 g larutan  $\text{H}_2\text{O}_2$  diambil, 25.0 mL larutan 0.0215 M  $\text{KMnO}_4$  ditambahkan dan pentitratan memerlukan sebanyak 5.10 mL larutan 0.112 M  $\text{Fe}^{2+}$ . (Diberikan: Berat atom bagi Fe = 55.85, H = 1.0, O = 16.0 dan Mn = 54.9 g/mol)

Persamaan tindakbalas:



- (i). Write a balance equation for the reaction equations in (2.1) above.

*Tuliskan satu persamaan seimbang bagi persamaan tindakbalas dalam (2.1) di atas.*

(3 marks/markah)

- (ii). Write a balance equation for the reaction equations in (2.2) above.

*Tuliskan satu persamaan seimbang bagi persamaan tindakbalas dalam (2.2) di atas.*

(3 marks/markah)

- (iii). Calculate the percent of  $H_2O_2$  in the sample.

*Kirakan peratus  $H_2O_2$  dalam sampel.*

(4 marks/markah)

- (c). 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). Calculate the purity of the  $Zn(OH)_2$ . Given: Molecular weight of AgCl = 143 g/mol and  $Zn(OH)_2$  = 99.4 g/mol .

*Ketulenan satu sampel  $Zn(OH)_2$  dengan berat 0.287 g ditentukan melalui pentitratan dengan satu larutan piawai HCl, memerlukan sebanyak 37.8 mL. Larutan HCl tersebut dipiawaikan dengan memendakkan AgCl dalam 25.0 mL aliquat dan penimbangan (berat 0.462 g AgCl diperolehi). Kirakan ketulenan  $Zn(OH)_2$ . Diberikan: Berat Molekul bagi AgCl = 143 g/mol dan,  $Zn(OH)_2$  = 99.4 g/mol .*

(6 marks/markah)

- (d) In the Liebig titration of cyanide ion, a soluble complex is formed and at the equivalence point, solid silver cyanide is formed, signaling the end point.

Reaction Equation:



A 0.5894 g sample was titrated with 0.1055 M  $\text{AgNO}_3$ , requiring 36.95 mL. Calculate the percent of KCN in the sample.

Given:

Atomic weight of K = 39.10; C = 12; N = 14, Ag = 107.87 g/mol

*Dalam pentitratan Liebig bagi ion sianida, satu kompleks terlarut terbentuk dan pada takat ekuivalens, pepejal argentum sianida terbentuk, menandakan takat akhir.*

Persamaan Tindakbalas:



*Satu sampel dengan berat 0.5894 g dititratkan dengan 0.1055 M  $\text{AgNO}_3$ , memerlukan sebanyak 36.95 mL. Kirakan peratus KCN dalam sampel.*

*Diberikan:*

*Berat atom bagi K = 39.10; C = 12; N = 14, Ag = 107.87 g/mol.*

*(6 marks/markah)*

- (3). (a). With the aid of diagram, briefly explain the principle mechanism of the atomic absorption spectrometer.

*Dengan bantuan gambarajah, terangkan secara ringkas prinsip mekanisme spectrometer penyerapan atom .*

(6 marks/markah)

- (b). Briefly discuss the types of interferences that could occur in AAS analysis given below:

*Bincangkan secara ringkas jenis-jenis gangguan yang boleh berlaku di dalam analisis AAS seperti yang dinyatakan di bawah:*

- (i) Matrix interference / Interferens Matriks
- (ii) Chemical interference / Interferens Kimia
- (iii). Ionization interference / Interferens Pengionan

(6 marks/markah)

- (c) A solution containing  $6.91 \times 10^{-6}$  M CrQ<sub>3</sub> (Q = 8 hydroxyquinolate ion) had a percent transmittance of 31.2 in a 1.00 cm cell. Calculate the following.

*Satu larutan yang mengandungi  $6.91 \times 10^{-6}$  M CrQ<sub>3</sub> (Q = 8-ion hydroxyquinolate) mempunyai peratus transmisi sebanyak 31.2 di dalam sel berukuran 1.0 cm. Kira seperti berikut:*

- (i). absorbance of the solution.  
*penyerapan larutan.*

(2 marks/markah)

- (ii) molar absorptivity of the complex.  
*penyerapan molar complex.*

(2 marks/markah)

- (iii). absorbance in a 5.00-cm cell of a solution one-half the foregoing concentration.

*nilai penyerapan dalam 5.0 cm sel dengan setengah nilai kepekatan.*

(5 marks/markah)

- (iv) cell path length needed to give a percent transmittance of 10.0.

*panjang laluan sel yang diperlukan untuk memberi nilai transmisi sebanyak 10.0.*

(4 marks/markah)

**PART B / BAHAGIAN B**

(4). (a). Describe / Terangkan,

(i). Titration equivalent point

*Titik ekuivalen pentitratan*

(1 marks/markah)

(ii). Titration end point

*Titik akhir pentitratan*

(1 marks/markah)

(iii). Acid-base titration curve (with the aid of diagram)

*Keluk pentitratan asid-bes (dengan bantuan gambarajah)*

(3 marks/markah)

(b). A key component of all diffraction is the angle between the incident and diffracted rays. With the aid of diagram, explain the difference between XRD diffraction pattern of amorphous and crystalline material?

*Komponen utama semua pembelauan adalah sudut antara sinar tuju dan sinar pembelauan. Dengan bantuan rajah, terangkan perbezaan antara corak pembelauan XRD bahan amorfus dan hablur?*

(4 marks/markah)

- (c). Rietveld refinement is a technique described by Hugo Rietveld for use in the quantitative characterisation of crystalline materials.

*Pemurnian Rietveld ialah teknik yang diterangkan oleh Hugo Rietveld untuk digunakan dalam pencirian secara kuantitatif bagi bahan kristal.*

- (i). Describe Profile R-factor, weight R-profile factor and Bragg factor.

*Terangkan Profil R-faktor, berat R-profil faktor dan Bragg faktor.*

(4 marks/markah)

- (ii). Discuss the Rietveld refinement method in crystal structure pattern

*Bincangkan kaedah pemurnian Rietveld di dalam paten struktur hablur*

(4 marks/markah)

- (d). XRF is an analytical method to determine the chemical composition of all kinds of materials. XRF can be divided into two main categories: energy dispersive system (EDXRF) and wavelength dispersive system (WD-XRF). Define the advantages and disadvantages of both system and make a comparison by using a table.

*XRF ialah kaedah analisis untuk menentukan komposisi kimia semua jenis bahan. XRF boleh dibahagikan kepada dua kategori utama: sistem penyebaran tenaga (EDXRF) dan sistem penyebaran panjang gelombang (WD-XRF). Perihalkan kelebihan dan kekurangan kedua-dua sistem dan buat perbandingan dengan menggunakan jadual.*

(8 marks/markah)

- (5). (a). A ship of Nickel ore from China was purchased by a local metal refiner company. The analysis certificate, made out while the ship was being loaded, showed that the % Ni is 17.66% with a standard deviation of 0.85% for 5 measurements. When the ore arrived at the refinery, it was analysed with the following results: 17.58%, 17.61%, 17.69% and 17.64%. Using statistical analysis, determine if the refiner should accept the ore?

*Satu kapal dengan muatan bijih Nikel dari China telah dibeli oleh satu syarikat logam penulen tempatan. Sijil analisis yang dikeluarkan semasa pemunggahan bijih ke atas kapal, menunjukkan % Ni adalah 17.66% dengan nilai sisihan piawai 0.85% bagi 5 pengukuran. Apabila bijih tiba di kilang penulenan, analisis yang dilakukan memberikan keputusan berikut: 17.58%, 17.61%, 17.69% dan 17.64%. Menggunakan analisis statistik, tentukan samada bijih ini boleh diterima oleh syarikat penulen?*

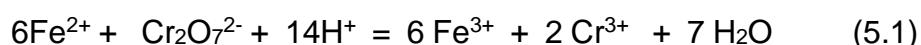
(8 marks/markah)

- (b). The following equation shows the reaction between  $\text{Fe}^{2+}$  and  $\text{Cr}_2\text{O}_7^{2-}$  :



The equilibrium constant for the reaction is  $1.00 \times 10^{57}$ . Calculate the equilibrium concentration for the Fe and Chromium species if 10.0 mL solution of 0.02 M  $\text{K}_2\text{Cr}_2\text{O}_7$  in 1.14 M HCl will react with 10.0 mL solution of 0.12 M  $\text{FeSO}_4$  in 1.14 M HCl.

*Persamaan berikut menunjukkan tindakbalas antara  $\text{Fe}^{2+}$  dan  $\text{Cr}_2\text{O}_7^{2-}$  :*

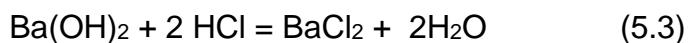


Pemalar keseimbangan bagi tindakbalas adalah  $1.00 \times 10^{57}$ . Kirakan kepekatan keseimbangan bagi spesi-spesi Fe dan kromium jika 10.0 mL larutan 0.02 M  $K_2Cr_2O_7$  dalam 1.14 M HCl bertindakbalas dengan 10.0 mL larutan 0.12 M  $FeSO_4$  dalam 1.14 M HCl.

(7 marks/markah)

- (c). The carbon dioxide evolved on heating a 2.407 g sample of steel in a closed system was swept into a flask containing 50.00 mL of 0.05081 M of barium hydroxide. After filtering the solution to remove the precipitated barium carbonate, the remaining base consumed 14.87 mL of 0.1125 M hydrochloric acid for titration to the phenolphthalein end point. Calculate the percentage of carbon in the steel.

The chemical reaction equations:



Karbon dioksida yang terhasil dengan pemanasan 2.407 g sampel keluli dalam satu sistem tertutup dialirkan masuk ke dalam satu kelalang yang mengandungi 50.00 mL 0.05081 M barium hidroksida. Selepas penurasan larutan untuk menyingkirkan mendakan barium karbonat, bes yang tinggal memerlukan sebanyak 14.87 mL 0.1125 M asid hidroklorik dalam pentitratan pada takat akhir fenolphthalin. Kirakan peratus karbon dalam keluli.

Persamaan tindak balas kimia:



(10 marks/markah)

- (6). (a). Briefly discuss about iodine and iodometry titration and their differences.

*Terangkan dengan ringkas mengenai pentitratan iodin dan iodometri dan perbezaannya.*

(6 marks/markah)

- (b). The zinc carbonate in a 3.0591 g sample of smithsonite ore was dissolved in acid and separated from the remaining insoluble substances. This solution was treated with an excess of oxalic acid then made basic to precipitate  $ZnC_2O_4$ . The precipitate was collected, dissolved in 0.5 M  $H_2SO_4$ , and liberated oxalic acid required 26.14 mL of 0.01875 M  $KM_nO_4$  for titration. Calculate the %  $ZnCO_3$  in the ore.

*Zink karbonat dalam sampel 3.0591 g bijih smithsonite dilarutkan dalam asid dan dipisahkan dari bahan larut yang tersisa. Larutan tersebut kemudian dirawat dengan asid oksalik yang berlebihan kemudian dijadikan alkali untuk memendapkan  $ZnC_2O_4$ . Mendapan yang dikumpulkan, dilarutkan dalam 0.5 M  $H_2SO_4$ , dan asid oksalik yang dibebaskan memerlukan 26.14 mL  $KM_nO_4$  yang berkepekatan 0.01875 M untuk titrat. Hitung %  $ZnCO_3$  dalam bijih.*

Given: Molecular weight  $ZnCO_3$  = 125.4 g/mol

*Diberikan: Jisim molekul  $ZnCO_3$  = 125.4 g/mol*

(9 marks/markah)

- (c). A 20.0 mL aliquot of malonic acid solution was treated with 10.0mL of 0.250 M Ce<sup>4+</sup> leading to the reaction:

*Satu larutan asid malonik sebanyak 20.0 mL dirawat dengan larutan 0.250 M Ce<sup>4+</sup> menghasilkan persamaan berikut:*



After standing for 10 minutes at 60°C, the solution was cooled and the excess Ce<sup>4+</sup> was titrated with 0.100 M Fe<sup>2+</sup>, requiring 14.4 mL to reach the ferroin endpoint.

*Setelah dipanaskan selama 10 minit pada suhu 60 °C, larutan tersebut disejukkan dan larutan Ce<sup>4+</sup> yang berlebihan dititratkan dengan larutan 0.100 M Fe<sup>2+</sup> yang memerlukan sebanyak 14.4 mL untuk mencapai takat akhir ferroin.*

- (i). Write the reaction for the back titration

*Tuliskan persamaan pentitratan-balik*

(4 marks/markah)

- (ii). Calculate the molarity of the malonic acid in the sample.

*Kirakan kemolaran asid malonik dalam sampel.*

(6 marks/markah)