



RF-3463-64

M. Sc. (Part - I) Examination
April / May - 2010
Analytical Chemistry : Paper - IV

Time : 3 Hours]

[Total Marks : 54

RF-3463

Instructions :

(1)

नीचे दशावेक निशानीवाणी विगतो उत्तरवही पर अवश्य कभवी. Fillup strictly the details of signs on your answer book.	Seat No. :
Name of the Examination :	<input type="text"/>
<input type="text" value="M. Sc. - 1"/>	<input type="text"/>
Name of the Subject :	<input type="text"/>
<input type="text" value="ANALYTICAL CHEMISTRY - 4"/>	<input type="text"/>
Subject Code No. : <input type="text" value="3"/> <input type="text" value="4"/> <input type="text" value="6"/> <input type="text" value="3"/>	<input type="text"/>
Section No. (1, 2,.....) : <input type="text" value="1"/>	
	Student's Signature

- (2) Answer to two sections should be written in separate answer books.
- (3) All questions carry equal marks.
- (4) Draw neat diagrams where necessary.

- 1 (a) Describe different types of instrumental analysis.
- (b) How will you estimate carbonyl group in an organic compound by oxime formation method.

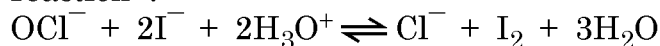
OR

- (a) Explain the principle of semi-micro determination of sulfur of organic compound by combustion method.
- (b) Explain the methods of removal of interference in chemical analysis giving two illustrations.
- (c) An iron-ore sample weighing 597 mg is dissolved in HCl and Fe^{+3} reduced with $SnCl_2$ to Fe^{+2} . The solution is titrated with $KMnO_4$. If 47.35 ml of 0.1075 N $KMnO_4$ is required to reach the end point, calculate percentage of Fe_2O_3 in the iron ore (Fe=56, O=16)
- $Fe^{+2} + MnO_4^- + H^+ \rightarrow Fe^{+3} + Mn^{+2} + H_2O$ (unbalanced)

- 2 (a) Give the structure of neo-cuproin and discuss its use for spectrophotometric determination of copper.
- (b) Discuss the criteria in selecting solvent for non-aqueous titrations. Classify the solvents.

OR

- (a) Describe the spectrophotometric determination of iron using 1,10-Phenanthroline. Give the structure of the complex formed. Why it is necessary to add hydroxylamine-hydrochloride in this procedure?
- (b) How equivalence point is determined in non aqueous titrations? Give reaction of water with Karl-Fischer reagent.
- (c) Commercial liquid bleach contains sodium hypochlorite which can be analyzed iodometrically by the following reaction :



OR

- (c) The resulting I_2 is then titrated with $\text{Na}_2\text{S}_2\text{O}_3$. If 5.00 ml sample of bleach produces sufficient I_2 to require 38.56 ml of 0.1986N $\text{Na}_2\text{S}_2\text{O}_3$ to reach the starch end point, calculate the NaOCl content as weight per volume per cent (Na = 23, Cl = 35.5, O = 16).

- 3 (a) Explain the working of photomultiplier tube as detector in spectrophotometer. How it better than phototube?
- (b) Explain Job's method to determine metal-ligand ratio of complex.

OR

- (a) (i) Describe the radiation source for visible region in spectrophotometer.
- (ii) Why filter is used beyond 600 nm in spectronic-20?
- (b) Explain the mechanism of Fluorescence and phosphorescence. Differentiate the two phenomena.
- (c) 147 mg $\text{K}_2\text{Cr}_2\text{O}_7$ (mol. wt. 294) was dissolved in 250 ml aqueous solution containing little H_2SO_4 . 10 ml of this solution was diluted to 100 ml with 0.1 N H_2SO_4 . This solution in a cell having path length 1.5 cm showed 40% T at 440 nm. Calculate the molar absorptivity of $\text{K}_2\text{Cr}_2\text{O}_7$ at this wavelength.

OR

- (c) The molar absorptivity of a zinc complex is 5.0×10^3 at 420 nm. A 290 mg brass sample is dissolved in acid, complexing agent is added and sample is diluted to 250 ml. The absorbance of the solution in 0.5 M cell is 0.752. Calculate the percentage of zinc in the brass (Zn=65).

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(1)

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- (2) Answers to two sections should be written in separate answer books.
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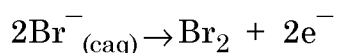
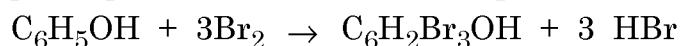
- 4 (a) (i) Explain 'polarographic maxima'. How is it suppressed?
(ii) Explain the disadvantages of DME.
- (b) Explain the terms :
- (i) Residual current
(ii) Migration current
(iii) Diffusion current.

OR

- (a) (i) State the Ilkovic equation. Explain each term involved in it. Give their units.
(ii) What are the advantages of DME? Compare the usefulness of DME as a cathode and as an anode.
- (b) How oxygen interferes in DC polarography? Describe the method to remove oxygen from the solution.
- (c) An unknown solution of acetophenone yielded a limiting current of $0.470 \mu\text{A}$. To 5.00 ml of the unknown solution was added 0.100 ml of standard solution of acetophenone, the concentration of which was $5.50 \times 10^{-4} \text{ m}$. Another polarogram was recorded and the limiting current was $0.520 \mu\text{A}$. Calculate the molar concentration of acetophenone in unknown solution.
- 5 (a) Explain controlled potential coulometry. Give the methods used to determine total quantity of electricity in this technique.
- (b) Write a brief note on 'Biamperometric titration'.

OR

- (a) Explain the factors affecting the quality of electrodeposits.
- (b) Explain giving reasons, the nature of the curve obtained during amperometric titration of Pb^{+2} with $\text{K}_2\text{Cr}_2\text{O}_7$ with DME at an applied voltage 0V and -1.0V .
- (c) The phenol content of polluted water was determined by coulometric analysis. A 200 ml sample is acidified and 5g KBr is added. It required 362 S at 28.6 mA to reach the end point. Calculate the phenol content in the sample as parts per million (mol. wt. of phenol=94).



- 6 (a) (i) Explain the difference between distribution coefficient and distribution ratio.
- (ii) Derive formula for distribution ratio when Benzoic acid is distributed between water and Benzene.
- (b) 'Several extractions using small volumes of extracting solvent are more efficient than using the total quantity of solvent in one lot'. Justify the statement deriving equation.

OR

- (a) Describe the apparatus for craig extraction and discuss its operation.
- (b) Explain the extraction using ion-pair with suitable example.
- (c) A sample containing 1 gm organic substance 'A' is kept in 100 ml water. This solution is then shaken with 100 ml ether. In ether layer 0.7 gm of 'A' is extracted. Calculate

$$K_D \left[\frac{C_{\text{ether}}}{C_{\text{aq}}} \right]$$

How many additional extractions with 100 ml portion of ether would be required to extract 95% of the substance?

OR

- (c) Three extractions with 50 ml portion of CHCl_3 removed 97% of solute from 200 ml aqueous solution. Calculate K_D in favour of CHCl_3 .