

**M. SC. (ANALYTICAL CHEMISTRY) SEM-III (CHOICE BASED
CREDIT & GRADE SYSTEM) : SUMMER - 2018**
SUBJECT : THERMAL, RADIO & ELECTRO-ANALYTICAL METHODS

Day : **Friday**
Date : **20/04/2018**

S-2018-0874

Time : **03.00 PM TO 06.00 PM**
Max. Marks : 60

N.B.:

- 1) All questions are **COMPULSORY**.
- 2) Figures to the right indicate **FULL** marks.
- 3) Use of scientific **CALCULATOR** is allowed.
- 4) Draw neat and labeled diagrams **WHEREVER** necessary.
- 5) Use of Graph paper is **ALLOWED**.
- 6) Both the sections should be written in **SEPARATE** answer books.

SECTION – I

Q.1 Answer **ANY THREE** of the following: **[15]**

- a) Describe technique and method of coulometric analysis with reference to constant potential coulometry.
- b) What is 'Anodic stripping voltammetry'? Explain its application in assay at very low concentration level.
- c) What is base electrolyte? Elaborate its role in polarographic analysis.
- d) Describe construction and working of oxygen sensor.
- e) Write and explanatory note on square wave voltammetry.

Q.2 A) Answer **ANY TWO** of the following: **[10]**

- i) State and explain principle of electrogravimetry and hence discuss the application of the technique.
- ii) Describe the methodology of coulometric titrations with reference to its application to compleximetric titrations.
- iii) What is the role of gelatin and nitrogen gas in polarography?

B) Solve **ANY ONE** of the following: **[05]**

- i) When differential pulse method was used, three individual samples of chloramphenicol in 0.1 M acetate buffer having pH 4 at -0.3 volts versus 'SCE' gave the following results. What is the concentration of the unknown sample x.

Concentration in $\mu\text{g/ml}$	i peak in 'nA'
0.5	2.98
0.98	5.94
X	4.50

- ii) In an electrochemical determination of bromide ion from 100 ml solution, the silver anode, after electrolysis was completed, was found to have gained 0.88 gm.
 - a) Calculate the molarity of bromide in the original solution.
 - b) Calculate the potential of the silver electrode at the beginning of the electrolysis. Given K_{sp} of AgBr = 4×10^{-3} and $E_{Ag}^0 = 0.8$ volts

P.T.O.

SECTION – II

Q.3 Answer **ANY THREE** of the following: [15]

- a) What is the technique and methodology of thermal analysis? Describe the instrumental setup used for the same.
- b) What is Neutron activation analysis? Discuss its applications and merits.
- c) What is radioactive labelling? Describe how various titrations can be performed by using this technique.
- d) Draw and explain a representative TG curve.
- e) Describe the construction and working of chemically modified electrode used in spectroelectrochemistry.

Q.4 A) Answer **ANY TWO** of the following: [10]

- i) What are thermometric titrations? Discuss its application with suitable examples.
- ii) Describe a typical electrochemical sensor.
- iii) Explain the basic principle of Isotopic dilution analysis.

B) Solve **ANY ONE** of the following: [05]

- i) 65mg sample of limestone, in its thermogram shows the weight of 56mg at 500⁰C and 36 mg at 900⁰C. Calculate the percentage of CaCO₃ and MgCO₃ in the given sample. [Given atomic weights Ca = 40, Mg = 24.3, C = 12, O = 16].
- ii) A concentration of insulin in a production vat is determined by isotopic dilution method. A sample of Insulin weighing 1mg, is labelled with ¹⁴C with activity of 549cpm and added to 10ml sample taken from the production vat. After homogenization of the sample, a portion of insulin is separated and purified, yielding 18.3 mg of pure insulin. The activity of the isolated insulin is measured at 148cpm. How many milligrams of insulin are present in the original sample?

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