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RF-3567-68

M. Sc. (Part - II) Examination

April / May – 2010

Analytical Chemistry : Paper - II

(Instrumental Methods of Analysis)

Time : **3** Hours] [Total Marks : **52**

Instructions :

(1)

નીચે દર્શાવેલ ☛ નિશાનીવાળી વિગતો ઉત્તરવહી પર અવશ્ય લખવી.
Fillup strictly the details of ☛ signs on your answer book.

Name of the Examination :

☛ **M. Sc. - 2**

Name of the Subject :

☛ **ANALYTICAL CHEMISTRY - 2**

☛ Subject Code No. : **3 5 6 7** ☛ Section No. (1, 2,.....) : **NIL**

Seat No. :

Student's Signature

(2) All questions are compulsory.

(3) Figures to the right indicate full marks to that question.

(4) Answer to each questions should be written in same answer book.

1 (a) What is boundary potential? Explain the mechanism and the equation of boundary potential developed at the glass membrane of ISE. **9**

(b) What is the major application of liquid-ion exchanger microelectrode? How do gas sensors function?

OR

(a) Explain the principle of biocatalytic membrane electrode based on pressed pellet for the detection of amino acid.

(b) Explain the errors in using glass electrode. Mention accuracy of pH measurement. Give types of active ion exchanger liquid membranes with primary and interfering ions in each.

(c) A fluoride electrode is used to determine fluoride in water sample. Standards and samples are diluted (1:10) with TISAB. For 1.0×10^{-3}M standard solution the potential reading vs SCE is – 211.3 mV. For another standard solution 4.0 x 10^{-3}M, the potential reading is –238.6 mV. The reading with sample is –226.5 mV. What is the concentration of fluoride ion in the given sample.

2 (a) Discuss the waveforms, sensitivity current measurements and nature of polarograms of NPP and DPP. **16**

(b) Describe the concentration profiles at planor microelectrodes in unstirred solutions during electrolysis. Show effect of time.

(c) Compare AC and square wave polarography. What are their limitations?

OR

2 (a) Distinguish between square wave and NPP polarography with respect to wave forms, current measurements, sensitivity and polarograms. **16**

(b) Discuss the use of rotating electrodes. Why rapid scan polarography fails for binary mixture analysis?

(c) Describe cyclic voltammetry as an analytical tool for agricultural applications. What is TAST polarography?

(d) Stripping voltammetry at platinum disk electrode is used to determine $Pb_{(11)}$ in 10.0 ml water sample. The potential was adjusted to –1.5 V vs SCE for 5 min. prior to scanning anodically at 50m V/S. The height of peaks for various 10.0 ml standards and for sample are:

$Pb^{+2} conc \times 10^{-5} M$	1.0	2.0	4.0	6.0	8.0	*Sample*
Peak current (μA)	1.20	2.37	4.82	7.27	9.67	5.36

Determine concentration of Pb^{+2} in given sample solution.

3 (a) Discuss the principle of radioimmunoassay. Give its applications. **9**

(b) Explain radio release method. Compare it with radiometric titrations.

OR

3 (a) Explain the principle and quantitative method of Neutron Activation analysis. What are thermal neutrons? **9**

(b) Describe interaction of α particle with matter. Describe scintillation transducers.

(c) A fermentation broth was known to contain some Aureomycin.; To a 1000 g portion of the broth was added 1.00 mg of Aureomycin containing carbon – 14 (specific activity = 150 counts / min/mg) from the mixture, 0.20 mg of crystalloine pure Aureomycin was isolated which had a net activity of 400 counts in 100 min. Calculate the weight of Aureomycin per 1000 g. of broth.

4 (a) Describe a typical DTA curve for polymeric material. What information is obtained from it? Give factors affecting DTA. **12**

(b) How is a mixture of calcium oxalate and magnesium oxalate analysed by TGA? Give characteristics of reference in DTA.

OR

4 (a) Describe thermometric titrations. What are its advantages? Give application of Direct Injection Enthalpy. **12**

(b) Explain general principles of DTA and DSC. Describe instrumentation of DTA.

(c) A 0.6025 g of sample was dissolved and Ca^{2+} and Ba^{2+} ions present were precipitated. As $BaC_2O_4.H_2O$ and $CaC_2D_4.H_2O$. The oxalate mixture were then heated in a thermogravimetric apparatus leaving a residue that weighed 0.5713 g in the temperature range 320° to 400°C and 0.4673 g in the temperature range 580° to 620°C. Calculate the percentage of Ca and Ba in the sample.

5 (a) Describe kinetic variables affecting zone broadening in chromatography. **12**

(b) Give Van Deemter equation. Explain the nature of VD plot. What is significance of U_{opt} and H_{min}?

(c) Describe flame ionization detector. How is it modified for greater sensitivity of N and P containing compounds?

OR

5 (a) Describe solid and liquid sample injection systems in GC. Show working of rotatory valve. **12**

(b) Explain derivatization in GC. Give two examples where it is used? Explain the use of molecular seives in GPC.

(c) Describe open tubular column. Explain and give advantage of two dimensional paper chromatography.

6 (a) Explain the working of UV-absorption detector in HPLC. How TLC and HPLC differ? **12**

(b) Explain the use of suppressor column in IEC. Give its applications.
How are bonded phase supports prepared? Give its advantages.

OR

6 (a) Explain the function of gaurd column and degassers in HPLC. Describe use of silica as stationery phase in HPLC. **12**

(b) Describe continuous flow electrophoresis. Give merit and demerit of reciprocating pumps.

(c) Two solutes with distribuition ratio 1.47 and 1.86 are to be separated on a column in which V_s/V_m = 13.6. How many theoretical plates are needed to ensure resolution of 1.40 ? What length of column is required if H is 0.250 cm.