

B.Sc. 5th Semester (Honours) Examination, 2023 (CBCS)

## **Subject : Chemistry**

Course : DSE-2

## (Instrumental methods of Chemical Analysis)

### **Time: 2 Hours**

Full Marks: 40

*The figures in the margin indicate full marks.*

*Candidates are required to give their answers in their own words as far as practicable.*

- 1.** Answer *any five* questions from the following: 2x5

  - What are the light sources used in UV-vis spectrophotometer?
  - Why is quartz cuvette used to measure spectra in UV-vis and fluorescence spectrophotometer?
  - Why tetramethyl silane (TMS) is used as an internal standard in NMR spectroscopy?
  - What are the light sources used in spectrofluorometer?
  - Define 'ionization chamber' in mass spectrometry.
  - Which of  $^{13}\text{C}$  and  $^{12}\text{C}$  is NMR active? State reason.
  - Define signal to noise ratio (S/N) in spectroscopic technique.
  - What type of cell is used to measure IR spectra of hygroscopic liquid/aqueous samples?

**2.** Answer *any two* questions from the following:  $5 \times 2 = 10$

  - Draw a block diagram of double beam UV spectrophotometer. Explain the monochromator device. 4+1
  - Write down the differences between liquid and gas chromatography? What is the difference between (+) and (-) mode of ionisation in mass chamber? 3+2
  - Draw the various fragmented patterns of *n*-butyl alcohol and *sec*-butyl alcohol. 5
  - What are the functions of using external standard in NMR? What are the advantages of Fourier Transform in Infrared Spectroscopy? 3+2

**3.** Answer *any two* questions from the following:  $10 \times 2 = 20$

  - Write down the block diagram of instrumentation of mass spectrometer. Define the term 'chemical shift'. Which one of 'mass spectrometer' and 'mass spectroscopy' is correct? Defend your answer. 4+3+1+2
  - Describe the principle of Atomic Absorption Spectroscopy (AAS)? Which gases are used in AAS? What are the differences between single and double beam spectrophotometer? 5+2+3

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(c) Name the fuels used in flame photometry. Discuss briefly about the columns used in liquid chromatographic analysis. Define  $R_f$  value in chromatographic analysis. What is the sampling procedure in IR spectrophotometer? 2+5+2+1

(d) 'Increase in polarity of the solvent shifts  $n \rightarrow \pi^*$  and  $n \rightarrow \sigma^*$  bands to shorter wavelength'—comment on the statement. Briefly describe the spin-spin splitting in NMR spectroscopy. 'FT-IR is plotted against wavenumber instead of wavelength'—explain. 4+4+2

**B.Sc. 5th Semester (Honours) Examination, 2023 (CBCS)****Subject : Chemistry****Course : DSE-2 (OR)****Full Marks: 40****Time: 2 Hours***The figures in the margin indicate full marks.**Candidates are required to give their answers in their own words as far as practicable.***1. Answer any five questions from the following:**

- (a) Explain with suitable example of propagation of error.
- (b) Why  $R_f$  value is important in chromatographic method?
- (c) Write down the structural formula of a reagent for gravimetric estimation of  $\text{Ni}^{2+}$ -ion and indicate the pH of medium.
- (d) Write down the limitations of Lambert-Beer's law.
- (e) What is chromatogram?
- (f) Write down the Nernst distribution law, and give its application(s).
- (g) How many significant figures are in the number 0.00150?
- (h) Differentiate between absolute error and relative errors.

**2. Answer any two questions from the following:**

- (a) State the normal law of distribution in statistical analysis. Draw its probability curve and give its equation with mentioning all the terms. 2+3
- (b) (i) Express the actual differences between the end-point and equivalence point in a titration.  
(ii) What are the basic criteria for metal ion extractions by chelation method. 2+3
- (c) (i) Write briefly about the principles and applications of thin layer chromatography (TLC).  
(ii) Give two important differences between liquid chromatography (LC) and gas-chromatography (GC). (2+1)+2
- (d) (i) Mention two interferences in AAS analysis. How they interfere in this analysis?  
(ii) Write two important limitations of thermogravimetric techniques. 3+2

**3. Answer any two questions from the following:**

- (a) (i) Explain the role of pH in solvent extraction method.  
(ii) Name two stationary phases and two mobile phases that are used in chromatography. How chromatographic separation is influenced by solvent polarity? 10x2=20

- (iii) "The concentration of  $\text{Ni}^{2+}$ -ion can be measured by gravimetry and electrogravimetry"— Which one does give better result and why?  $3+(1+1+2)+(1+2)$
- (b) (i) Briefly describe with suitable example of the chiral chromatographic technique using HPLC column.  
(ii) Write the differences between precision and accuracy.  
(iii) The analysis of a calcite sample yielded CaO percentage of 55.95, 56.00, 56.04, 56.08 and 56.23. The last value appears anomalous; should it be retained or rejected at the 95% confidence level? ( $Q_{\text{crit}} = 0.71$  at 95% confidence level for five measurements)
- $4+3+3$
- (c) (i) What is Infrared (I.R.) spectroscopy? Write the different types of samples and their preparation method used in I.R. spectroscopic technique.  
(ii) Define molar extinction co-efficient and give its unit.  $(2+1\frac{1}{2}+4\frac{1}{2})+2$
- (d) (i) How much of each enantiomer is present if the enantiomeric excess is 90?  
(ii) What is the use of Chiral Shift reagent?  
(iii) How do you determine optical rotation by polarimeter?  
(iv) What is the principle of Chiral Separation in HPLC?
- $2+2+3+3$
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