

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: 2×5=10
- 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}C and ^{12}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: 5×2=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: 10×2=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

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Full Marks: 40

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as far as practicable.*

1. Answer any five questions from the following: 2×5=10
 - (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 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: 5×2=10
 - (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: 10×2=20
 - (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?

- (iii) "The concentration of 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+1½+4½)+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