M. Sc (Instrumentation) Examination, 2017
(2^{nd} Year, 1^{st} Semester)

Subject: Analytical Instrumentation Time: 4 Hours
Full Marks: 100

Group-A

Section **-I**
Answer any three:

1. What are Bravais lattice and how they distinguish from each other? Write a short note on symmetry in crystal structure and point group. 4+3+3
2. How one can use X-ray diffraction to determine particulate size of any material? What are the limitations of powder diffraction? Write short note on peak broadening of X-Ray peak. For Al sample XRD peaks are found at 2θ=38.52, 44.76, 65.14, 78.26, 82.47, 99.11, 112.03, 116.60, 137.47, 163.78. Determine the structure of Al. Index the first six reflections. Calculate the unit cell parameter of Al. 2+2+2+4
3. What is Atomic Packing Factor? Calculate the Atomic Packing Factor for SC lattice. In the analysis of powder pattern one observe quite often the ratio of $Sin^2\theta$ values are absent for approximately equals to some integers for which h, k and l are mixed. What inference can be drawn regarding the crystal structure and discuss the reason behind it. 2+2+6
4. (a) Write a short note on X-Ray generation. (b) Compare the structural features among quasi-crystal, crystal and amorphous materials. (c) What are the conditions for diffraction? Why X-Rays are chosen for atomic diffraction? 4+2+4
5. Write on surface analysis technique using XPS. Explain, with neat diagram, techniques of electron analyzers in XPS. Write five application examples for surface analysis using XPS. 3+4+3

Section **–II**

6. Write short note on any four 5x4

(a) ECD (b) Paper Chromatography (c) CW NMR & FT NMR, (d) Chemical shift in NMR spectroscopy, (e) Specific Capacitance of super-capacitor (f) Auger Spectroscopy.

Group;B

Section-I

Attempt question No.1 and any three from the rest;

1(a). Describe with neat sketch all possible vibrational modes of a – CH_2 – grouping in a polyatomic molecule (with respect to IR spectra).

(b). Explain with diagram the HOMO →LUMO transition in 1,3- butadiene.
(c). Point out the fundamental differences of 'Mass Spectrometry' and UV-Visible Spectroscopy.
(d).Calculate pH of a 0.1(M) aquous solution of HCL at 0^0C,given that $K_\omega = 0.1 X10^{-14}$ at 273 K.
3+3+2+2 = 10

2. Combine any two electrodes of your choice to make a pH meter. Explain how pH is calculated from $E_{observed}$. 5

3. Show how solvent affects UV-Spectra, especially the Л → Л* and n → Л* transitions. Use diagram. 5

4. What is 'finger print' zone in IR spectra ? Explain how proponal – 1 and proponal – 2 can be distinguished by finger printing. 5

5. Explain how IR spectroscopy is superior in detecting an acetylinic linkage compared to UV spectroscopy.
Aldehydes and ketones are isomers. Show how IR can detect them ?. 3+2

6. Given, solution A = Aqueous Solution of H_2SO_4 (pH = 3),
Solution B = Aqueous Solution of NaOH (pH = 11),

a) 100 ml of solution A is mixed with 100 ml of solution B. Calculate the pH of the resultant solution.
b)Aniline is taken in solution A and phenol is taken in solution B. Explain how their uv spectra will be affected compared to those in aquous solutions. 2+3

7. Explain with diagram the components of a uv-spectroscope. Describe its functioning. 5

Section-II

1. Answer question No.1 and any five from the rest::
(i). Which colour of the following will give best resolution in an compound Microscope
a) Red, b) Orange, c) Green, d) Blue
(ii). Photograph which is taken from microscope is known as ?
(iii). Which one is best suited to get surface view of an object; magnifying glass, Compound microscope, TEM, SEM.
(iv). Kind of microscope used to study internal structure of a cell is ?
(v). Resolving power of an optical microscope can be increased by ? 1 X5 = 5

2. Write expression for resolution of an optical Microscope. Explain resolution of an electron microscope(TEM). Draw schematic diagram of a TEM and label it properly. 2+2+2

3. Explain interaction of focused electron beam with sample in a scanning electron microscope. How secondary electrons are deflected in a SEM. Explain each components of detector and associated systems with neat diagram. 3+3

4. Write names of the attachments with SEM for material characterization. Explain in detail about functions of each of these characterization attachment. 1+(2.5+2.5)

5. Write on sample preparation procedure for TEM & SEM. Explain in detail about fixation, dehydration, and metal coating for electron microscopy.

6. Write the name of STM inventers. How STM work? Write on limitation of STM. What is constant height and constant current mode in STM ? 1+1+4

7. Explain different modes of AFM from van der Waals curve. Explain signal processing steps in an AFM in contact mode with necessary diagram. What is LFM ? 2+2+2

8. Write notes on the following(any two):

(i) Vacuum for Electron Microscope. (ii) ESEM (iii) FE Gun (iv) Back scattered electron detector. 3 X 2