

**Birla Institute of Technology and Science, Pilani**  
**Second Semester 2021-2022**

**Comprehensive Examination (Closed Book)**

**10 May 2022 (4:00-7:00 PM)**

**Max Time: 180 min**

**Total Max Marks: 75**

**Total questions: 51**

- Answer in the question paper, with in the space provided below question. Must not write out of the provided space.
- May use the provided sheet for rough.
- Irrelevant writing will be penalized.
- Don't skip any mathematical steps-otherwise you will lose marks

**SECTION A (44 M) Short Questions, 1 marks for each question**

1. Name the combined DTA-TGA instrument we used for our practical class.
  
  
  
  
  
  
  
  
  
  
  
2. Mention the maximum temperature (°C) that could be reached in our DTA-TGA instrument
  
  
  
  
  
  
  
  
  
  
  
  
  
  
  
3. According to the ASTM E1582-93 standard practice for calibration of temperature scale for TGA which techniques are suggested?
  
  
  
  
  
  
  
  
  
  
  
  
  
  
  
4. In case of the TGA the absolute confirmation of the evolved gases is possible by using which instrument technique?
  
  
  
  
  
  
  
  
  
  
  
  
  
  
  
5. What purging gas is used for your DTA-TGA experiment?
  
  
  
  
  
  
  
  
  
  
  
  
  
  
  
6. What standard sample DSC curve is generally used in order to calculate the heat capacity of another sample?

7. Which parameter we need to consider in order to measure UV spectrum of a liquid of low absorbance ( $<0.2$  Abs).
8. Which wavelength light probe could be used if you want to use a cuvette made of polystyrene?
9. Which solvent could be your best choice if you want to measure the UV spectrum of a sample at any wavelength between 200 to 350 nm?
10. Which properties should you consider in case of choosing a solvent for measuring UV-visible spectra of a sample?
11. What source lamp is used for the 'Thermo Fisher Scientific Evolution 201&220' UV-Visible spectrophotometers?
12. If you have two metals, Lead and aluminium, then which one would you chose to calibrate temperature of the Perkin Elmer DSC 4000? Why?
13. Which detector would you use for 'Thermo Fisher Scientific Evolution 201&220' UV-Visible spectrophotometers?
14. Mention the material, volume (in ml), and path length (in mm) of the cuvette you would use for 'Thermo Fisher Scientific Evolution 201&220' UV-Visible spectrophotometers.

15. What material pans did you use for your DSC experiment?
16. Which nature (wave or particle) of electromagnetic radiation is considered for the interpretation of the UV-VIS absorbance spectroscopy phenomena?
17. Which UV-Vis detector uses dynodes?
18. What is the necessary condition for a molecule to be IR active?
19. Mention any two types of Molecular Vibrations
20. How many degrees of freedom could be considered for a molecule? What are they?
21. How many vibrational modes are possible for CH<sub>4</sub>?

22. Mention the essential requirements for ATR technique for collecting IR spectroscopy data.
23. What company and model FTIR instrument we used for our experiment?
24. What region IR instrument we used for our experiment? Note that there are three region IR instruments are possible: near-infrared region ( $12800 \sim 4000 \text{ cm}^{-1}$ ), mid-infrared region ( $4000 \sim 200 \text{ cm}^{-1}$ ) and far-infrared region ( $50 \sim 1000 \text{ cm}^{-1}$ )
25. Which ATR crystal did you use in your FTIR experiment?
26. What IR source did you use in your FTIR experiment?
27. Why KBr is used in IR sample preparation?
28. Raman scattering is elastic or inelastic?
29. What is Elastic scattering?
30. Which scattered photon has higher energy, Stokes scattering or anti Stokes scattering?

31. Which one has higher intensity, Stokes scattering or anti Stokes scattering?
32. Mention two necessary conditions for a molecule to be Raman active.
33. What make and model Raman spectrophotometer BITS Piali, Piali campus has?
34. What type of detector is used by BITS Piali campus Raman spectrophotometer
35. Which laser of what wave length you used for the Raman characterization of Si sample?
36. Mention two most important difference between the working principle of Raman and FTIR spectroscopy.
37. What are the full form of SERS and TERS?
38. Mention the types of information one can extract from N<sub>2</sub> adsorption desorption isotherm.

39. Mention the types of information one can extract from the different  $N_2$  adsorption-desorption hysteresis loop shapes
40. Nitrogen is a good adsorptive gas for which type of materials?
41. Ar is a good adsorptive gas for which type of materials?
42. Mention the type of information one can extract from  $N_2$  adsorption-desorption isotherm using BET model, BJH model, and t-plot technique.
43. Mention the most important difference between point lattice and crystal lattice.
44. Mention h, k, and l based general rules for the allowed reflection peaks for simple cubic structure, body centered cubic structure, and face centered cubic structure.

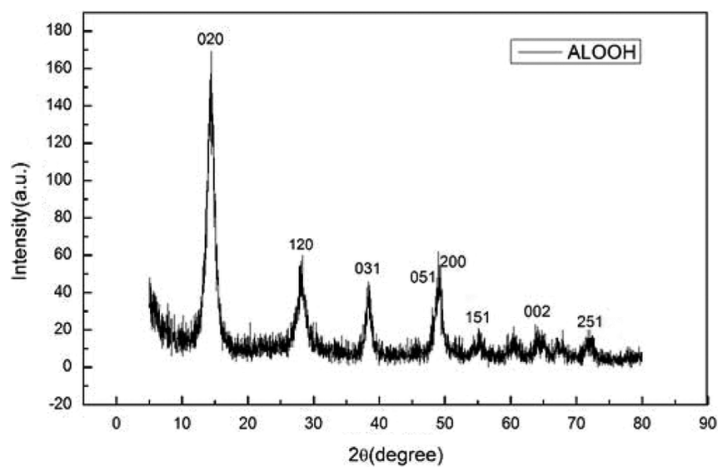
**SECTION B (31 M) Numerical/Conceptual Questions**

45. Calculate the dispersion % of Pt in a 2wt% Pt loaded on  $Al_2O_3$  catalyst, if 20 mg of this catalyst adsorbs 1.5 cc hydrogen in a pulse chemisorption experiment at standard temperature and pressure conditions (STP). Mention the required equation. [4]

46. Calculate the particle size for  $\text{Al}_2\text{O}_3$  powder of BET surface area  $200 \text{ m}^2/\text{gm}$  and density  $3.95 \text{ gm/cc}$ . Consider that the particles are spherical. Mention the required equation. [2]

47. State the Bragg's Law. Draw the basic ray diagram for X-Ray diffraction and derive the corresponding Bragg's equation. [6]

48. Calculate particle size of the powder from the XRD spectrum shown below. Mention the required equation. [2]



49. Mention the Essential HPLC parameters and define their significance with proper equations. [10]

50. How many XPS peaks could be expected for  $\text{MoO}_3$  powder based on the electronic configuration of the elements? Which are those? Explain your thought process step wise. Atomic number of Mo is 42 and O is 8. [5]

51. Consider three samples, Mo,  $\text{MoO}_2$ , and  $\text{MoO}_3$ . Arrange these samples according to the binding energies of the core electrons of Mo. Explain your thought process step wise. [2]