REV-01
MSC/57/62

2022/07

M.Sc. CHEMISTRY
FOURTH SEMESTER
PHYSICAL METHODS OF ANALYSIS
MSC – 403

(Use Separate Answer Scripts for Objective & Descriptive)

Duration : 3 hrs. **Full Marks : 70**

(PART-A: Objective)

Time : 20 min. **Marks : 20**

Choose the correct answer from the following: ***1×20=20***

1. In Emission spectroscopy, it is necessary to identify the .....line, because it corresponds to the characteristic element. Fill-up the blanck-
   a. Weakest b. Strongest
   c. Middle d. First

2. The type of excitation that leads to significant fluorescence in organic molecule is-
   a. The $\Pi \longrightarrow \Pi^*$ b. The $\Pi \longrightarrow \Pi^*$
   c. The $\sigma \longrightarrow \Pi^*$ d. The $\sigma \longrightarrow \sigma^*$

3. In Differential Calorimetry, the instrument records the ---------- change against temperature or time.
   a. Weight b. Energy
   c. Mass d. Entropy

4. The Fluorescence intensity is proportional to the ---------- of irradiation.
   a. Intensity b. Wavelength
   c. Wave number d. Time

5. The kinetic energy of the electron in XPES ---------- as the as the positive charge of the atom increases.
   a. increases b. decreases
   c. Has no effect. d. None of the above.

6. Which one of the following is used in single crystal XRD?
   a Pinhole camera b Laue camera
   c Debye-Schecrer camera d None of them

7. (110) plane is parallel to which axis
   a X b Y
   c. Z d None of them

8. Which of the following has lowest resolving power?
   a Seeman-Bohlin camera b Back reflection camera
   c. Debye-Scherrer camera d All of them have same resolving power.

9. Which of the following crystal is best monochromator?
   a Unbent b Bent and cut
   c. Bent and not cut d None of them

10. Exposure of sample by X-ray depends directly on
    a Intensity of x-ray beam
    b Time
    c. Both a & b
    d None of them

11. Which of the following compounds will have 2nd shortest retention time in RP-HPLC in MeCN-H_2O mobile phase?
    a. Toluene
    b. Benzaldehyde
    c. Benzyl alcohol
    d. Benzoic acid

12. A mixture having two compounds analysed in HPLC under four different methods (M1, M2, M3, & M4) projecting 'α' values 2, 2.2, 2.4, 3 respectively. The best resolution will be observed for the method
    a. M1
    b. M2
    c. M3
    d. M4

13. In HPLC 'Peak asymmetry' involves calculation at peak height of
    a. 10%
    b. 15%
    c. 12%
    d. 6%

14. Identify the m/z of the parent ion which produces a fragment of m/z = 100 with a meta- stable ion of m/z = 62.893
    a. 139
    b. 159
    c. 149
    d. 169

15. FID detectors result all the signals based on the equivalent of the gas,
    a. Ethane
    b. Butane
    c. Methane
    d. Propane

16. Electron microscope uses
    a. Glass lens
    b. Electromagnetic lens
    c. Both of the above
    d. None of the above

17. Which of the following is needed to observe finer details of an object?
    a. High velocity light wave
    b. High velocity electron wave
    c. Low velocity electron wave
    d. X-ray wave

18. Which of the following can be observed by electron microscope?
    a. Arrangement of atoms
    b. Shape and size of the particles
    c. Both of the above
    d. None of the above

19. To see a particle by interaction with a wave
    a. Wavelength should be less than particle size
    b. Wavelength should be more than particle size
    c. Both of the above
    d. None of the above

20. TEM image may be recorded on
    a. Fluorescent screen
    b. Photographic plate
    c. Charged coupled device camera
    d. All of the above

-- --- --

PART-B : Descriptive

Time : 2 hrs. 40 min. **Marks : 50**

[Answer question no.1 & any four (4) from the rest]

1. a. What is the basic difference between NP-HPLC & RP-HPLC? How is the stationary phase developed for RP-HPLC? **3+2 =5**

   b. What is the difference between resolution and magnification?Write differences between SEM and TEM. **2+3 =5**

2. a. What is the basic principles of Atomic Absorption Spectroscopy? Name the five basic component involved in the instrumentation. **5+5=10**

   b. Draw the TG thermogram for decomposition of $CaC_2O_4.H_2O$ and explain the decomposition step with reaction.

3. a. Draw the XPES of the 1s electron of the nitrogen atom in the compound trans-$[Co(en)_2(NO_2)_2]NO_3$. How do you justify the presence of three types of nitrogen in the complex? **5+5=10**

   b. UVPES of oxygen shows photoelectron energy at 12.0, 17.0 and 19.3 eV with respective vibrational frequency at 1780, 1010 and 1110 cm^{-1}. The vibrational frequency of O_2 molecule is 1555 cm^{-1}. Make the assignment of the MO, from which photo electron are ejected.

4. a. What are the two most important consideration for making choice of radiation for XRD? **2+4+4 =10**

   b. What are the four causes of background intensity? Discuss briefly each of them.

   c. Draw schematic for three different ways of film loading. Briefly discuss one of them.

5. a. With help of schematics describe transmission and back reflection Laue cameras. **4+3+3 =10**

   b. Discuss briefly on collimators.

   c. How three main powder diffraction methods are differentiated?

6. a. What is GPC?Discuss how the MW of unknown sample is identified by GPC technique. 5

b. Describe various steps involved in SEM imaging with schematic diagram. 5

7. Discuss 10
   i. retention time,
   ii. peak resolution,
   iii. capacity factor and
   iv. selectivity factor in reference to a chromatogram obtained after HPLC analysis of a sample having two compounds.

8. Describe in detail all types of specimen interactions in electron microscopy with diagram. 10

= = *** = =