KATHMANDU UNIVERSITY
End Semester Examination
March/April, 2017

Marks scored:

Level : B. Sc./B. Pharm./B. Tech.
Year : III
Course : INAN 301
Semester : I
Exam. Roll No. :
Time: 30 mins.
F. M. : 20
Registration No.:
Date : MAR 26 2017

SECTION "A"
[20 Q × 1=20 marks]

Encircle the most appropriate answer from the given choices.

1. Photomultiplier tubes are not useful as infrared detectors because
   a. Infrared photons do not carry enough energy to eject photoelectrons from the photocathode.
   b. Infrared sources are too intense and cause degradation of the photocathode.
   c. Infrared sources output a light beam that is too large in diameter to be compatible with the photomultiplier tube.
   d. None of the above.

2. According to the Beer-Lambert Law, on which of the following does absorbance *not* depend?
   a. Extinction coefficient of the sample.
   b. Color of the solution.
   c. Distance that the light has travelled through the sample.
   d. Solution concentration.

3. Narrow line spectra are emitted by
   a. Hot solids.
   b. Excited polyatomic molecules.
   c. Molecules in the ground molecular state.
   d. Excited atoms.

4. How many Hertz does one ppm correspond to for an PMR spectrometer operating at a radio frequency of 60 MHz?
   a. 60 Hz b. 120 Hz c. 100 Hz d. 100 MHz

5. What happens to the molecules in the ion source of the mass spectrometer?
   a. They are ionized and split in fragments.
   b. They are ionized only.
   c. They lose hydrogen atoms only.
   d. They are excited and emit radiation.

6. The special atomization for the determination of arsenic in water sample is:
   a. Hydride atomization.
   b. Cold vapor atomization.
   c. Flame atomization.
   d. Glow discharge atomization.

7. Which of the following detectors used in either GC or HPLC is correctly identified as either destructive or non-destructive?
   a. Refractive Index (RI) detector in HPLC is destructive.
   b. The Atomic Emission Detector (AED) used in GC is non-destructive.
   c. FID detector in GC is destructive.
   d. The absorbance detectors (UV-VIS, Fluorescence, IR) used in HPLC are destructive.

8. Accuracy is defined as:
   a. A measure of how often an experimental value can be repeated.
   b. The closeness of a measured value to the real value.
   c. The closeness of a measured value to the theoretical value.
   d. The number of significant figures used in a measurement.

9. In mass spectrometry, the atomic mass unit, amu, is defined as
   a. 1/12 of the mass of one neutral C-12 atom.
   b. The mass of any individual atom expressed in grams.
   c. The mass of a deuterium atom.
   d. The mass of one mole of hydrogen atoms expressed in daltons.

10. Which one of the following techniques can be used for the detection in a liquid chromatograph?
    a. Ionization in flame. b. Increase in temperature.
    c. Ultraviolet absorbance or refractive index measurement. d. Thermal conductivity.

Fill in the blank by most appropriate VALUE or WORD.

11. In infrared spectroscopy _____ frequency range is known as the fingerprint region.

12. When signal splitting occurs, the distance between the individual peaks of a signal is called the _____ and is measured in Hz.

13. In _____ ionization technique the sample is introduced as a solution and is nebulized under an applied electrical potential.

14. A nonlinear molecule with 'n' atoms generally has _____fundamental vibrational modes.

15. In thin layer chromatography, if a particular amino acid has low solubility in the mobile phase used, then the amino acid will have low _____.

16. WCOT stands for_____ column.

17. The ratio of the analyte concentrations in both stationary and mobile phases at equilibrium is called _____.

18. The process of migration of analyte particle through a solution under the influence of electric field is _____.

19. The absorption energy per unit area of solvent is called _____.

20. The ratio of slope of calibration curve and the standard deviation of the measurement is called _____.

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Year : III
Time : 2 hrs. 30 mins.

Course : INAN 301
Semester : I
F. M. : 55

SECTION "B"

[5Q. × 3 = 15 marks]

Attempt *ANY FIVE* questions.

1. Describe the factors that are of vital importance in selecting an analytical method. [3]

2. Explain the types of sample injection in GC. [3]

3. Describe the structure of a flame. What are the different zones, why do some atoms give a stronger absorbance at the base of a flame, the middle of a flame, at the tip of a flame? [2+1]

4. Write any two differences between dispersive and Fourier Transform IR spectrometers. What kind of compounds is IR active? [2+1]

5. How many PMR peaks and multiplicity do you expect in the following compounds?
i) CH_3- CO-CH_3 ii) $(CH_3)_3C$-CH_2Cl iii) $CH_3ClC=CH_2$ [1+1+1]

6. How does an electron capture detector create a signal when analyzing halogenated compounds by GC? [3]

7. Differentiate between:
a) Gel permeation and Gel filtration
b) Paper and Electro chromatography [1.5+1.5]

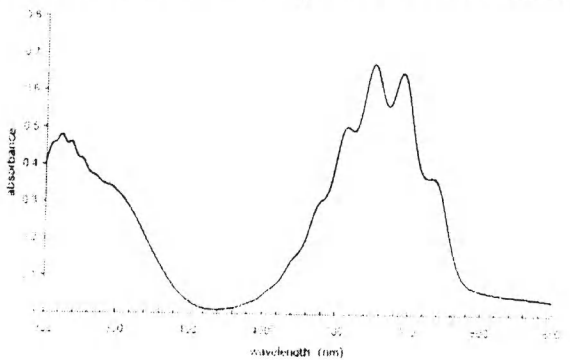
SECTION "C"

[5Q. × 5 = 25 marks]

Attempt *ANY FIVE* questions.

8. What are the devices that have been utilized for obtaining (selecting) monochromatic light from a light source? Draw a schematic diagram of a grating monochromator and label all the components. [3+2]

9. Below is the UV-Vis spectrum for a solution containing 7.50 ppm permanganate ion.



a) What type of material was the cell constructed from for recording the spectrum?
b) What is λ_{max} of the strongest absorption peak?
c) What is $\varepsilon_{max}(cm^{-1}\ mol^{-1}\ L)$ of the strongest absorption peak, given that cell length was 5 mm?
d) What type of transition is involved? [1+1+2+1]

10. What is a releasing agent and in which instrumental method would you use one? How does it work? [2+3]

11. a. Explain why acetylene proton signal is at up field whereas ethylene resonate at down field in PMR? [2+3]
b. What is soft ionization technique? Discuss any one technique.

12. Describe the general elution problem. What steps are taken to alleviate this problem in (a) GC and (b) HPLC? Of the fundamental chromatographic parameters, which is affected by each of these manipulations? [2.5+2.5]

13. a. List the variables that lead to zone broadening in chromatography.
b. How are the performance characteristics like i) capacity factor ii) plate height, are determined in TLC? [3+2]

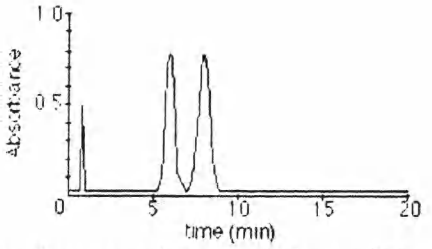
14. a. Draw a labeled diagram of electrochemical detector for HPLC.
b. Why does the minimum in a plot of plate height versus flow rate occur at lower flow rates with liquid chromatography than with gas chromatography? [2+3]

SECTION " D"

[2Q. × 7.5= 15 marks]

Attempt *ANY TWO* questions.

15. a. Given the following chromatogram for the separation of two solutes (A and B) on a 25 cm long HPLC column, determine:



i) the H.E.T.P achieved under the operating conditions of the column using solute A values for calculations;
ii) the resolution achieved for solutes A and B;
iii) the capacity factors for solutes A and B;
iv) the linear velocity of the mobile phase in the column. Assume that the exact retention time for the compound A ($t_{R(A)}$) is 6.0 min and $t_{R(B)}$ = 7.8 min and that the width of the eluting peak at the baseline for compound A is 1.5 min and for compound B is 1.9 min. Also, the column yielding a t_m = 0.9 min.

b. What are the possible defects of normal calibration curve procedure? How does the standard addition method minimizes these defects? [5+2.5]

16. a. Explain the role of a suppressor column in an ion exchange chromatography.
    b. Explain in brief:
    i) Off resonance decoupling ii) Hydrogen deficiency index
    iii) Principle of size exclusion chromatography [3+4.5]

17. From the combined IR, mass, ^{13}C-NMR and ^{1}H-NMR spectra given below for an unknown compound, elucidate the correct structure for the compound with detailed description of each spectrum analysis. Also show the mass fragmentation pattern. [5.5+2]