BITS PILANI, DUBAI CAMPUS
FIRST SEMESTER 2012-2013
COMPREHENSIVE EXAM

Course Title: Instrumental Methods of Analysis **Course No.: BIO C391**
Maximum Marks: 40 **Weightage: 20%**
Time: 2 hours **Date: 29th December 2012**

1a. What are input transducers? Give examples of any four and mention the output signal they exhibit. Give examples of any four input transducers and mention their respective electrical output. [3]
b. A butanol is being oxidized to butanal. How can we monitor this reaction using FT-IR and know that the reaction has gone to completion? [3]
c. What is the principle of Fourier Transform IR spectroscopy? [2]
d. Why is the phenomenon of refraction observed when light enters a medium of higher or lower refractive index? [2]

Q2a. Discuss with examples the structural factors that affect the absorption of a compound in the electromagnetic spectrum. [3]
b. Only a proportion of the light absorbed is emitted as radiation in fluorescence. Justify. [3]
c. Mention the following: [4]

a. source of light in a fluorimeter	e. Stationary phase in a paper chromatography
b. detector system used in UV spectroscopy	f. Material of the sample holding cell in IR spectroscopy
c. Analyzer in polarimeter	g. Detector used in the elemental analysis
d. Material of the motor and pestle used for sample preparation in IR spectroscopy	h. General developing system for TLC

Q3a. Give the principle of a photomultiplier tube. [2]
b. A technician had performed a TLC and found not separation of fragments but a smudged spot in the TLC. He concluded that the compound was pure and not a mixture. Is he right in his inference? Is it possible to modify the method to establish a more appropriate inference? How can we distinguish a polar and a non polar compound by TLC? [3]
c. Describe the development of plasma in the ICP torch. Draw a diagram of the ICP torch. [5]

Q4a. A pharmaceutical company has developed a new drug which is optically active. The efficiency of the drug depends on the optical rotation of the compound. The same drug with a specific rotation +115° exhibits the maximum efficiency. Determine the efficiency of the new drug, if the observed optical rotation for 0.06g% of the drug is +54.0°. [3]
b. Give the schematic diagram of a HPLC system. Mention any two detectors used in HPLC.[2+1]
c. What are the two types of columns used in Gas chromatography and How are these different from each other? What is the stationary phase in the GC? [2+2]

**********ALL THE BEST**********

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1st Semester 2012- 2013

Instrumental Methods of Analysis BIO C391

Test (Open book)

Date: 11/11/12 (S) **Duration: 50 minutes** **Weightage: 10% (Max Marks20)**

1a. An unsaturated ketone is dissolved in hexane has a λmax of 251 nm. What shift would you observe (in the λmax) when it is analyzed in methanol? Explain. [2]

1b. Why is the emission monochromator placed at 90° to the incident light beam in a fluorescence spectrophotometer? [2]

1c. How is ICP different from the AAS? [3]

1d. A sugar industry is trying a new process for purification of glucose. The optical activity observed for 0.2mg/ml glucose made by this new process was +8.5. Determine the percent purity of the sample if the specific rotation for pure glucose is +52.5 °. [3]

2a. Why is it essential to pretreat the sample before it is introduced in the ICP? [2]

2b. You are asked to take soil samples from a field contaminated with chemical pesticides for analysis. List the precautions (at least 3) you would take while doing it. [1.5]

2c. What are signal generators? Explain the two methods used for signal generation. [3]

2d. What is the energy of a photon associated with wavelength 325nm? [2]

2e. Why is it essential to work with dilute solutions in a fluorimeter? [1.5]

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1a. **An unsaturated ketone is dissolved in hexane has a λmax of 251 nm. What shift would you observe (in the λmax) when it is analyzed in methanol? Explain.** **[2]**

The unsaturated ketone which has λmax of 251 nm in aa non polar solvent like hexane, will show a bathchromic shift when analysed in a polar solvent like methanol. The λmax will appear at higher than 251 nm region.

1b. Why is the emission monochromator placed at 90° to the incident light beam in a fluorescence spectrophotometer? **[2]**

The emission monochromator placed at 90° to the incident light beam in a fluorescence spectrophotometer to minimize the risk of transmitted or reflected incident light reaching the detector.

1c. How is ICP different from the AAS? **[3]**

AAS	ICP
Atomic absorption depends upon the number of ground state atoms	Atomic emission depends upon the number of excited atoms
Presence of a light source	Absence of the light source
The temperature in the atomizer is adjusted to atomize the analyte atoms in the ground state only (1700 – 3150C)	The temperature in the atomizer is big enough to atomize the analyte atoms and excite them to a higher energy level (6000 – 8000C)

1d. A sugar industry is trying a new process for purification of glucose. The optical activity observed for 0.2mg/ml glucose made by this new process was +8.5. Determine the percent purity of the sample if the specific rotation for pure glucose is +52.5 °. **[3]**

Specific rotation of new glucose sample = 8.5 / 0.2 *1

= 42.5°

The specific rotation of pure sample is +52.5°

Hence the purity of the new sample is 80.95%.

2a. Why is it essential to pretreat the sample before it is introduced in the ICP? **[2]**

Organic matter has to be removed as this would interfere with the analytical process. Organic matter is usually removed from food samples by some form of oxidation, either by the use of oxidizing acids in a wet digestion or by dry ashing in the presence of air or pure oxygen. This is essential to avoid interference and also clogging of the nebulizers which add the sample into the plasma.

2b. You are asked to take soil samples from a field contaminated with chemical pesticides for analysis. List the precautions (at least 3) you would take while doing it. [1.5]

Make sure that during sampling, homogeneous sample is taken.

It has to be properly stored without decomposition till analyzed.

Check if any pre treatment is required before loading on to the machine: eg. Solubilization of compounds in acids, filtration or chemical treatment or dilution etc....

2c. What are signal generators? Explain the two methods used for signal generation. [3]

The signal results from the direct or indirect interaction of the analyte with some form of energy such as electromagnetic radiation, electricity or thermal heating.

Two general methods are used for signal generation:

1. Application of an external signal to the sample and subsequent modification of this signal by the analyte as in absorption spectroscopy
2. Creation of an sample environment that allows the analyte to produce a signal as in potentiometric measurements.

The signal generator is unique to each type of the instrument and its design requires an understanding of the physical properties of the instrument components and the chemical properties of the analyte and the sample matrix.

2d. What is the energy of a photon associated with wavelength 325nm? [2]

Wavelength and frequency are related to the energy of a photon(in eV), E, by Planks constant, h, (6.62×10^{-34} J sec **or** 4.14×10^{-15} eV s) and velocity of light in a vacuum ($c \sim 3 \times 10^{8}$ m/s)

$$E = h\nu = \frac{hc}{\lambda} = \frac{(4.14 \times 10^{-15}) \times (3 \times 10^{8})}{325}$$ **$= 3.822 \times 10^{-9}$ evs or 6.11×10^{-28} J sec**

2e. Why is it essential to work with dilute solutions in a fluorimeter? [1.5]

If the concentration of a solution prepared for fluorescence measurement is too high, some of the light emitted by the sample as fluorescence will be reabsorbed by other unexcited molecules in solution. For this reason, fluorescence measurements are best made on solutions with an absorbance less than 0.02, i.e. solutions of a sample 10-100 weaker than those which would be used for measurement by UV-VIS spectroscopy.

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Instrumental Methods Of Analysis BIO C391

Quiz – 2 (Close book)

Date: 17/12/12 (M) Duration: 20 minutes Weightage: 10% (Max Marks 10)

1. Mention any two advantages of HPLC over GC [1]

2. What is meant by an isocratic system? [1]

3. List any two detector systems in gas chromatography. [1]

4. _____, which is used as a carrier gas in GLC, has to be _____ and _____. [1]

5. What is an Interferometer? [2]

6. Why do we scan the background before determining an IR spectrum? [2]

7. I have an amine. How can I effectively use IR spectroscopy to determine if it is
i. a primary, secondary or tertiary amine and
ii. aliphatic or aromatic amine?

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1st Semester 2012- 2013

Instrumental Methods Of Analysis BIO C391

Quiz – 1 (Close book)

Date: 24/10/12 (W) Duration: 20 minutes Weightage: 10% (Max Marks 10)

Name: _____ **ID No.:**_____

1. Define Analytical Technique. Give an example. [1]

2. A In a polarimeter, differentiate polarizer from analyzer. [1]

3. Give any two applications of infra red radiations. [1]

4. **You have been provided with an aqueous copper sulphate solution, which shows an absorbance of 1.96 at 640nm. Determine the concentration of the solution if the molar extinction coefficient for aqueous copper sulphate solution is 20 L mol^{-1} cm^{-1}. [2]**

5. What are fluorophores? Give any two examples of the same. [1]

6. Give the significance of the blank solution in UV-Visible spectroscopy. [1]

7. Define a Bathochromic shift. [1]

8. Fluorene is more fluorescent than biphenyl. Justify. [2]