MODERN INSTRUMENTAL METHODS OF ANALYSIS
(CHEN 4131)

Time Allotted : 3 hrs **Full Marks : 70**

Figures out of the right margin indicate full marks.

Candidates are required to answer Group A and any 5 (five) from Group B to E, taking at least one from each group.

Candidates are required to give answer in their own words as far as practicable.

Group – A
(Multiple Choice Type Questions)

1. Choose the correct alternative for the following: **10 × 1 = 10**

(i) Wet ashing is a method of preparing samples
(a) made of polymers (b) made of glass
(c) made of carbon (d) none of above

(ii) Flame atomizers are used for
(a) decomposing the analyte into constituent atoms
(b) nebulizing the sample into aerosoles
(c) bombarding solid samples to eject atoms
(d) none of above

(iii) Bragg's law states that
(a) $n\lambda = 2d_{hkl}\sin\theta$ (b) $n\lambda = 2d_{hkl}/\sin\theta$
(c) $n\lambda = 2\sin\theta$ (d) $n\lambda = 2\sin\theta/d_{hkl}$

(iv) The instrument working on the principle of emission of X-ray photons from a sample by bombarding the surface with high energy electronsis
(a) X-ray fluorescence spectrometry (b) Electron microprobe analyzer
(c) X-ray diffraction spectrometer (d) Infrared spectrometer

(v) The near IR range covers the following wavelength range
(a) 2.5 μm-25μm (b) 0.67μm-3.33μm
(c) 50 μm -1000 μm (d) 5 μm-50 μm

(vi) Flame spectrometers measures
(a) spectrum of alkali metals (b) spectrum of alkali earth metals
(c) spectrum of rare earth metals (d) none of above

(vii) A hollow cathode lamp is used as a radiation source in
(a) UV-VIS spectroscopy (b) atomic emission spectroscopy
(c) atomic absorption spectroscopy (d) none of above

(viii) The photo multipler tube is a
(a) radiation source (b) sampling device
(c) detection device (d) none of above

(ix) Chromatography operates on the principle of
(a) distillation of the sample into its constituent
(b) separation of the sample into two phase
(c) mixing of the sample between phase
(d) none of above

(x) An eluent is a solvent
(a) used to wash an empty chromatography column
(b) used to dissolve the sample
(c) used to flush the sample through the column
(d) none of above.

Group- B

2. Name the type of error (determinate/indeterminate) in presence of the following. In each case, state the reason for arriving at your conclusion:
i) Dust particles
ii) Analyte
iii) Background electrical signals in instrument
iv) Stale reagent
v) Weighing balance with resolution higher than sample weight
vi) Fluctuations in power supply connected to a measuring device.

[(CO1) (Analyze/HOCQ)]
(2 × 6) = 12

3. (a) What is the difference between an emission spectra and absorption spectra?
[(CO1) (Apply/IOCQ)]

(b) Explain the different types of energy transitions activated when a molecule like C_2H_5 is excited by UV light. [(CO1) (Apply/IOCQ)]

(c) What are characteristic differences between continuum source and line source of radiation? Give examples of each source of radiation. [(CO1)(Apply/IOCQ)]
4 + 4 + 4 = 12

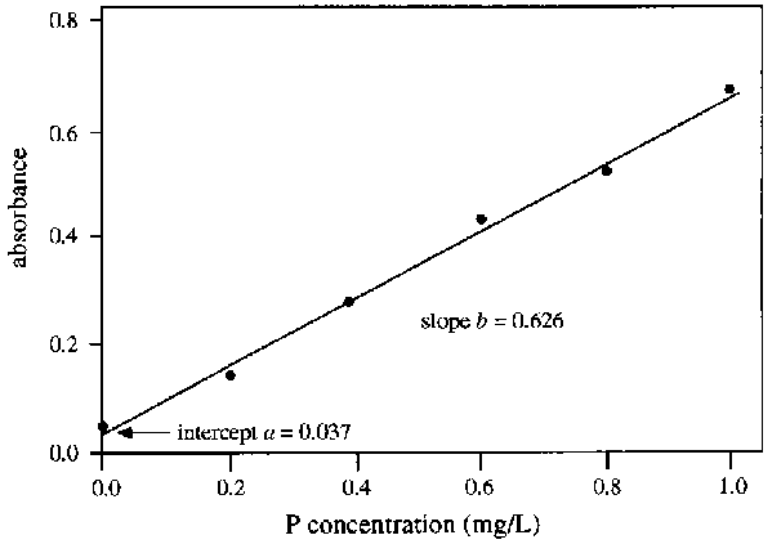
Group - C

4. (a) If light from a continuum line source is used in AAS, even after passing through slits and monochromatization, the detector would be very insensitive to changes due to absorption of this light by the sample. Can you elaborate this statement with a number example? [(CO2) (Understand/LOCQ)]

(b) What is the purpose of the monochromator in AAS? Explain the working principles of the monochromator [(CO2)(Remember/LOCQ)]
4 + 8 = 12

5. (a) What is chemical interference? Illustrate with an example.
[(CO1) (Understand/LOCQ)]

(b) The following is the calibration curve for sodium in a particular solvent. Can you explain why at zero concentration of sodium, the graph shows a positive absorbance? [(CO1) (Apply/IOCQ)]



(c) What are the advantages of using an electrical excitation source in atomic OES?
[(CO4) (Understand/LOCQ)]

4 + 4 + 4 = 12

Group - D

6. (a) Discuss the working principle of a dispersive infrared spectrometer with diffraction grating . State the advantages offered by a diffraction grating over a prism. [(CO2) (Remember/LOCQ)]

(b) "Mean square error achievable in a spectrometer with multiplexing advantage is less than one with a monochromator". Justify. [(CO2) (Analyze/LOCQ)]

(c) "Attenuated total (ATR) of IR beam is suitable for strongly absorbing samples" . Comment on the validity of the statement with proper explanation of the ATR principle. [(CO3)(Analyze/IOCQ)]

(4 + 2) + 3 + 3 = 12

7. (a) X-ray diffraction of a cubic crystal yields the following information: wavelength λ=1.54 A°, 2θ=48°. Derive the d-spacing for the plane (222) and the lattice parameter of the crystal. [(CO2,CO3) (Evaluate/HOCQ)]

(b) Explain the fundamental principles of X-ray diffraction. Discuss the working principle of Debye-Scherrer powder diffraction setup and derive the expression for lattice constant for a cubic lattice. [(CO3) (Apply/IOCQ)]

3 + (3 + 4 + 2) = 12

Group - E

8. (a) The following figure is the output from a chromatographic instrument during sample analysis. Is this an ideal reading? If not, can you comment on the exact cause of the behaviour demonstrated in figure 1.

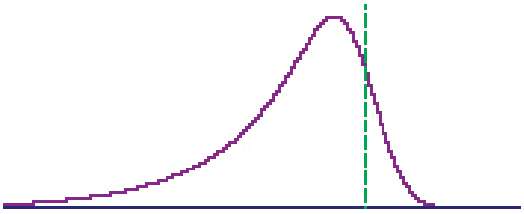


Figure 1 : chromatography reading of a sample

[(CO2) (Analyse/IOCQ)]

(b) What is the difference between the mass detector and the concentration detector? [(CO2) (Apply/IOCQ)]

(c) What is the major cause of peak broadening in chromatographic data? [(CO2)(Understand/LOCQ)]

6 + 4 + 2 = 12

9. (a) Explain the reasons for which HPLC has to use packed columns and not capillary column as in GC. [(CO2) (Understand/LOCQ)]

(b) What is reversed phaseliquid chromatography? [(CO2) (Remember/LOCQ)]

(c) What types of packing are used in this type of chromatographic columns? [(CO2) (Apply/IOCQ)]

6 + 4 + 2 = 12

Cognition Level	LOCQ	IOCQ	HOCQ
Percentage distribution	54.28%	24.29%	21.42%

Course Outcome (CO):

After the completion of the course students will be able to

CO1: Define a problem where the determination and/or measurement of some chemical species is required

CO2: Develop sufficient knowledge about the major instrumental methods of chemical analysis so that they can determine what technique should be used to solve a particular problem

CO3: Perform and advice as expert the details of instrumental analysis techniques needed by industries solve problems of related field

CO4: Gain the background necessary for a scientific expert witness to present new techniques in a court of law

*LOCQ: Lower Order Cognitive Question; IOCQ: Intermediate Order Cognitive Question; HOCQ: Higher Order Cognitive Question

Department & Section	Submission Link
CHE	https://classroom.google.com/c/NDAwOTA2MDQ3MTUz/a/NDYzODUyMDA1NDY2/details