

[KD 268] APRIL 2001

M.Pharmacy DEGREE EXAMINATION.

(New Regulations)

First Year

Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES

(Common to all Branches)

Time : Three hours

Maximum : 100 marks

Answer any FOUR questions.

All questions carry equal marks.

1. (a) Explain the theory involved in Polarographic analysis. (12)

(b) Define Chromophore and hydrogen over potential. (8)

(c) Vitamin D₂ (Calciferol, mol. wt. = 397), shows an absorbance maximum at 264 nm, with a molar absorptivity at 18,200. Beer's law is followed over a wide range

(i) What is absorptivity a?

(ii) What range of concentrations expressed in g/litre can be used for analysis if it is desired to keep the absorbance between limits of 0.2 to 0.8? Assume b = 1 cm. (5)

2. (a) Write briefly on the principle and instrumentation of NMR (Nuclear Magnetic Resonance) spectroscopy. (12)

(b) Discuss about chemical shift and spin-spin coupling. (8)

(c) For the compound 1, 3 dichloropropane Cl.CH₂.CH₂.CH₂.Cl discuss the following :

Number of proton magnetic resonance signals, their approximate positions, and splitting of signals. (5)

3. (a) Discuss the various types of mass analysers with appropriate illustrations. (12)

(b) Enumerate the rules applied in studying fragmentation patterns of Mass Spectra with suitable examples. (8)

(c) The following information was obtained from fragmentation pattern of a popular Analgesic, Antiinflammatory drug. Construct the structure and identify the same. Molecular formula C₉H₉O₄. (5)

m/Z	Intensity	Fragment
180	7%	C ₉ H ₉ O ₄ (M ⁺)
138	65%	C ₇ H ₆ O ₃
120	100%	C ₇ H ₄ O ₂
92	16%	C ₆ H ₄ O

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4. (a) With illustration, explain the working of a double beam spectrofluorimeter. (14)

(b) Discuss the influence of structure of a compound in fluorescence emission. (6)

(c) By using appropriate equations explain why fluorescence methods are more sensitive than absorbance methods in Quantitative analysis. (5)

5. (a) Explain about the X-ray diffraction technique. (12)

(b) Explain the technique of Radio-immuno assay with the help of pharmaceutical applications. (8)

(c) Discuss the typical features encountered in I.R. spectra of benzene, substituted benzenes (mono, m-disubstituted, p-disubstituted, o-disubstituted). (5)

6. (a) With illustration explain the instrumentation and working of the Gas-liquid chromatography. (11)

(b) Define and Explain the following : (6)

(i) Capacity and selectivity factors

(ii) Migration and retention time

(iii) Theoretical plates.

(c) Explain how and why the following calibration techniques are used in Gas chromatography/HPLC (High Performance Liquid Chromatography)

* Internal standard.

* Standard addition. (8)

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Paper I — MODERN PHARMACEUTICAL
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(Common to all branches)

Time : Three hours

Maximum : 100 marks

Answer ALL questions.

All questions carry equal marks.

1. (a) What is Nuclear Magnetic Resonance spectroscopy? Discuss in detail the factors that affect the chemical shift. What are the applications of NMR measurements? (3 + 6 + 5)
- (b) What is flame emission spectroscopy? Discuss the instrumentation involved. (6)
- (c) Discuss briefly about the electron spin resonance and its applications. (5)

2. (a) What are the different molecular vibrations involved on I.R. absorption? What are Overtones and Fermi resonance? Discuss the influence of hydrogen bonding on vibrational frequencies. (4 + 4 + 4)
- (b) What are the different thermal methods of analysis? Discuss the principle and applications of thermogravimetric analysis. (2 + 2 + 4)
- (c) Write briefly on Ion exchange chromatography. (5)
3. (a) Discuss the fragmentation pattern produced in mass spectrometric studies. Explain the nitrogen rule and ring rule (index of hydrogen deficiency) with suitable examples. What are the applications of mass spectrometry in drug metabolism studies? (6 + 4 + 3)
- (b) Explain about Radio immunoassay. (6)
- (c) What are the different techniques of paper chromatography? Write about the common developing reagents for paper chromatography. ($1\frac{1}{2} + 4\frac{1}{2}$)
4. (a) What are the electronic transitions involved on absorption in the U.V. region? Give examples. What are bathochromic, hypsochromic, hyperchromic and hypochromic effects? Discuss the instrumentation of a U.V. spectrophotometer. (3 + 4 + 8)
- (b) Write the principle of fluorimetry. What are its applications? (5)
- (c) Write about the different detectors used in Gas Chromatography apparatus. (5)

[KE 268] NOVEMBER 2001

M.Pharm. DEGREE EXAMINATION.

(New Regulations)

First Year

Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES

(Common to all Branches)

Time : Three hours Maximum : 100 marks

Answer any FOUR questions.

All questions carry equal marks.

1. (a) Discuss the principles underlying IR absorption spectroscopy. (13)
- (b) State Woodward's rules and illustrate their applications using suitable examples. (12)
2. (a) Discuss the principles of operation of mass spectrometer. (9)
- (b) Enumerate and explain the different methods of ionisation of samples in mass spectrometry and their applications. (7)
- (c) Give a brief account of
 - (i) Metastable peaks
 - (ii) Nitrogen rule
 - (iii) MC-Laffety rearrangement. (9)

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3. (a) Explain the principle involved in NMR spectroscopy and discuss the significance of essential components in NMR spectrometer with the help of neat labelled schematic diagram. (13)

(b) Write notes on :

(i) Chemical shift

(ii) Spin-Spin coupling

(iii) Anisotropic effect. (12)

4. (a) Discuss the principles, Instrumentation and application of HPTLC. (13)

(b) Give an account of detectors used in gas chromatography. (12)

5. Write notes on the following : (5 × 5 = 25)

(a) Relative absorbance spectroscopy

(b) Affinity chromatography

(c) Principle involved in fluorimetry and phosphorimetry

(d) Applications of conductometry in pharm analysis.

(e) Relative merits and demerits of DME over RPE.

6. (a) Explain the basic concept involved in thermal gravimetric analysis? Discuss the significance of Differential scanning calorimetry. (10)

(b) What are the different types of currents observed in polarography? Discuss the significance of Ilkovic equation and Halfwave potential. (10)

(c) Principle involved in Potentiometric titrations and steps involved in the location of end point. (5)

[KE 289] NOVEMBER 2001

M.Pharm. DEGREE EXAMINATION.

(Revised Regulations)

First Year

Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES

(Common to all branches)

Time : Three hours Maximum : 100 marks

Answer ALL questions.

All questions carry equal marks.

1. (a) Explain the instrumentation of NMR spectrometer. (8)
- (b) Explain the following terms : (8)
 - (i) Chemical shift
 - (ii) Spin-spin splitting.
 - (iii) Coupling constant
 - (iv) C^{13} NMR.
- (c) Write notes on : (5 + 4)
 - (i) Electron spin resonance
 - (ii) Atomic absorption spectroscopy.

2. (a) Discuss the importance and advantages of radioimmunoassays in pharmaceutical analysis. (15)
(b) Give an account of various techniques available for carrying out the thermal analysis. Give the working of any one instrument. (10)
3. (a) What are the factors influencing vibrational frequencies? Explain with examples how I.R. Spectra used for the identification of functional groups. (10)
(b) Describe the instrumentation and working of fluorimeter. Write a detailed note on quenching. (10)
(c) Quantitative I.R. spectrophotometry. Discuss.
4. (a) Discuss in details the principle and instrumentation of gas chromatography. (11)
(b) How are the following determined in GC?
 - (i) Efficiency of the column (6)
 - (ii) Flow rate of the carrier gas.
(c) Discuss the working and specific applications of different types of detectors used in GC. (8)

[KH 268] SEPTEMBER 2002

M.Pharm. DEGREE EXAMINATION.

(New Regulations)

First Year

Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES

(Common to all branches)

Time : Three hours

Maximum : 100 marks

Use calculator as required.

Answer any FOUR questions.

All questions carry equal marks.

1. (a) Explain with illustrations the working of double beam spectrophotometer. (15)

(b) Define molar absorptivity, Beer-Lambert's law. (5)

(c) A solution containing 3.80 mg/100 ml of A (MW = 220) has a transmittance of 39.8% in a 1.50 cm cell at 500 nm. Calculate the molar absorptivity of A. (5)

2. (a) Explain the theory of nuclear magnetic resonance (NMR) spectroscopy and significance of Boltzman distribution. (10)

(b) Discuss with examples chemical shift and spin-spin coupling. (5 + 5)

(c) For the compound ethyl benzyl ether



discuss the following : number of NMR signals, their approximate positions and splitting of signals. (NMR refers only to proton magnetic resonance). (5)

3. (a) Elaborately explain the instrumentation of mass spectrometer. (10)

(b) Define and explain :
Isotope abundance; Nitrogen rule and metastable ions. (9)

(c) An analgesic and anti-inflammatory drug has a molecular formula $\text{C}_{11}\text{H}_{14}\text{O}_2$. It produces the following mass spectra. Apply fragmentation pattern rules, identify the compound after building the structure (6)

M/Z	Intensity	Fragment
206	63%	$\text{C}_{11}\text{H}_{14}\text{O}_2$ (M^+)
163	100%	$\text{C}_{10}\text{H}_{11}\text{O}_2$
119	73%	C_9H_{11}
91	82%	C_7H_7
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4. Explain the following techniques with examples :

(a) Radio immuno assay. (12½)

(b) Differential thermal analysis. (12½)

5. (a) Explain the principle involved in the production of X-ray diffraction patterns by deriving Bragg's equation. (12)

(b) Discuss the factors influencing the fluorescence intensity. (8)

(c) Explain how you will differentiate the I.R. spectra produced by ethers and alcohols. (5)

6. (a) Explain with neat illustration the instrumentation of HPLC. (12)

(b) Define the following : (6)

Gradient and isocratic elution, reverse and normal phase chromatography, capacity and selectivity factors.

(c) Explain how and why the following calibration techniques are adopted in GC/HPLC : (7)

Internal standard

Standard addition.

[KH 289] SEPTEMBER 2002

M.Pharm. DEGREE EXAMINATION.

(Revised Regulations)

First Year

**Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES**

(Common to all Branches)

Time : Three hours

Maximum : 100 marks

Answer ALL questions.

1. (a) Explain the principle behind IR spectroscopy. What are the factors necessary in a molecule to absorb IR radiation? (10)
- (b) What is the principle behind the working of FT-IR spectrophotometer? Explain. (10)
- (c) Write a note on sample handling in IR spectroscopy. (5)

2. (a) Explain the principle of Electron impact mass spectrometry.

(b) Describe the various other techniques of ionisation of samples for mass spectrometry. Bring out their applications. (13 + 12)

3. Write notes on :

(a) Capillary electrophoresis

(b) X-ray crystallography

(c) Quantitative TLC. (9 + 8 + 8)

4. (a) Discuss in detail the principle and instrumentation of HPLC.

(b) How are the following determined in HPLC?

(i) Column efficiency

(ii) Detector sensitivity.

(c) Discuss the working and applications of detectors used in U.V. Spectroscopy. (11 + 6 + 8)

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First Year

Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES

(Common to all branches)

Time : Three hours Maximum : 100 marks

Answer any FOUR questions.

All questions carry equal marks.

(a) Explain the fragmentation rules in mass spectroscopy. (13)

(b) Predict the fragmentation pattern for the following compounds, give reasons :

(i) 2, 2-dimethyl pentane

(ii) 1-Phenyl ethanol

(iii) 9, 10-anthra quinone. (12)

7. (a) Describe an analytical methods using microbiological assay for determinations of vitamin combinations. (15)

(b) Explain briefly NMR and its applications. (10)

3. (a) Explain the principle, applications in pharmacy, and instrumentation of UV/Visible spectroscopy. (13)

(b) Give an account of detectors used in gas chromatography. (12)

4. (a) What are the factors influencing vibrational frequencies? Explain with examples how IR spectra used for the identification of functional groups. (10)

(b) Describe the instrumentation and working of fluorimeter. Write a detailed note on quenching. (10)

(c) Write a note on quantitative IR spectrophotometry. (5)

5. (a) What is electrophoresis? Explain with example the method of paper electrophoresis, and instrumentation used for paper electrophoresis. (13)

(b) Write an account of cotton effects and circular dichroism. (12)

6. (a) What is polarography? Write the construction and working of a polarograph. (13)

(b) Explain the terms : (12)

(i) Halfwave potential

(ii) Residual current

(iii) Limiting current

(iv) Ilkovic equation.

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[KI 289]

Sub. Code : 1001

M.Pharm. DEGREE EXAMINATION.

(Revised Regulations)

First Year

Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES

(Common to all Branches)

Time : Three hours

Maximum : 100 marks

Answer ALL questions.

All questions carry equal marks.

1. (a) What is the principle involved in thermo gravimetric analysis? Explain. List out its applications in pharmacy. (10)

(b) What is the principle and applications of supercritical fluid chromatography? (8)

(c) Write a note on Radio Immuno Assay and its application. (7)

2. (a) Write a short note on :

(i) Differential scanning calorimetry

(ii) Electron spin resonance spectroscopy.

(8 + 8)

(b) What are the requirements for a molecule to exhibit fluorescence? Explain. (9)

3. (a) Explain the instrumentation of NMR spectrometer. (10)

(b) Give an account of detectors used in gas chromatography. (15)

4. (a) What is the principle involved in flame emission spectroscopy? Discuss its instrumentation and application in pharmacy. (13)

(b) Give a brief account of

(i) Metastable peaks

(ii) Nitrogen rule

(iii) Mc-Lafferty rearrangement. (3 × 4 = 12)

OCTOBER 2003

[KJ 268]

Sub. Code : 2001

M.Pharm. DEGREE EXAMINATION.

(New Regulations)

First Year

Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES

(Common to all branches)

Time : Three hours

Maximum : 100 marks

Calculators may be permitted.

Answer any FOUR questions.

L. (a) Write briefly the principle, instrumentation of
IR. spectrophotometry. (12)

(b) List applications of capillary electrophoresis
and photon magnetic resonance spectroscopy. (8)

(c) A 8.44 PPM solution of FeSCN^{2-}
(mol int 113.9) has a transmittance, in a 1 cm cell at
580 nm, of 0.295. Calculate the molar absorptivity for
the complex at this wavelength. (5)

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2. (a) Write briefly with appropriate illustrations, equations the principle of NMR spectroscopy. (12)

(b) Explain chemical shift and spin-spin coupling. (8)

(c) How do you explain the chemical shift data of the following compounds? (5)

(i) Benzene — C_6H_6 , $\delta = 7.37$

(ii) Acetylene — C_2H_2 , $\delta = 2.9$

(iii) Toluene — $C_6H_5CH_3$, $\delta = 2.32$

(iv) Cyclohexene — C_6H_{12} , $\delta = 5.57$.

3. (a) Describe the instrumentation and working of mass spectrometer. (12)

(b) Write notes on fluorescence and chemical structure. (8)

(c) The following data was obtained from mass spectra of a compound. Interpret and report the structure of the same

(i) molecular formula C_8H_8O

(ii) m/e ratios at 120, 105, 77, 51, 43 and 28.

(5)

4. (a) Explain with illustrations the instrumentation and working of HPLC. (12)

(b) Write notes on Radio immuno assay with pharmaceutical applications. (7)

(c) Define the following with equations : (6)

(i) Capacity factor

(ii) Resolution

(iii) Selectivity factor.

5. (a) Write a note on X-Ray generation and interpretation of X-Ray powder diffraction data. (13)

(b) Bragg's law and its applications. (12)

6. Explain the terms :

(a) Half wave potential. (6)

(b) Residual current. (6)

(c) Limiting current. (6)

(d) Ilkovic equation. (7)

OCTOBER 2003

[KJ 289]

Sub. Code : 1001

M.Pharm. DEGREE EXAMINATION.

(Revised Regulations)

First Year

Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES

(Common to all Branches)

Time : Three hours

Maximum : 100 marks

Answer ALL questions.

All questions carry equal marks.

1. (a) Explain Beer-Lambert law. Describe the working of a *u.v* visible spectro photometer. Enumerate the application of *u.v* in pharmacy. (7 + 7)
(b) Write a note on atomic absorption spectroscopy. (6)
(c) Write the principle of fluorimetry. (5)
2. (a) Describe the principle and working of flame emission spectroscopy and its application. (6 + 6)
(b) Write a note on capillary electrophoresis. (8)
(c) What is ion exchange chromatography? (5)

3. (a) Discuss in detail the principle behind mass spectroscopy. Explain how the different fragments formed help in interpretation of the spectra taking specific examples. (10)
(b) Explain the following terms : (8)
 - (i) Base peak
 - (ii) Metastable peak
 - (iii) Nitrogen rule.
- (c) Write a note on spin-spin coupling. (7)
4. (a) Discuss in detail the instrumentation of high pressure liquid chromatography. (10)
(b) Write a note on field desorption MS. (8)
(c) Explain chemical shift with specific examples. (7)

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M.Pharm. DEGREE EXAMINATION.

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First Year

Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES

(Common to all Branches)

Time : Three hours Maximum : 100 marks

Sec. A & B : Two hours and
forty minutes Sec. A & B : 80 marks

M.C.Q. : Twenty minutes M.C.Q. : 20 marks

Answer ALL questions.

All questions carry equal marks.

SECTION A — (2 × 15 = 30 marks)

- (A) Explain the principle of absorption spectra by molecules in UV-visible region. (10)
(B) Describe how an Infrared spectra is systematically interpreted. (5)
- (A) Discuss the factors affecting chemical shift with suitable examples. (10)
(B) Write a note on C^{13} NMR spectroscopy. (5)

SECTION B — (10 × 5 = 50 marks)

Short notes.

- Write about Mc lafferty rearrangement.
- Write a note on Programmed temperature gas chromatography.
- Discuss the principle and applications of Differential Scanning Calorimetry.
- Discuss the Woodward's rule and its applications.
- Write about the important applications of X-ray diffraction methods.
- Explain the different factors that affect the fluorescence intensity.
- Discuss the principle and applications of ESR.
- Discuss the factors responsible for the band broadening in a chromatographic column.
- Write a note on super critical fluid chromatography.
- Explain the principle and applications of Flame Emission Spectroscopy.

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M.Pharm. DEGREE EXAMINATION,

(Revised Regulations)

First Year

**Paper I — MODERN PHARMACEUTICAL
ANALYTICAL TECHNIQUES**

(Common to all Branches)

Time : Three hours Maximum : 100 marks

Sec. A & B : Two hours and forty minutes Sec. A & B : 80 marks

M.C.Q. : Twenty minutes M.C.Q. : 20 marks

Answer ALL questions.

SECTION A

Long Essay : (2 × 15 = 30)

1. (a) State and derive Beer-Lambert's Law. What are its limitations? (8)

(b) Explain the principle and methodology of Differential scanning calorimetry. (7)

2. (a) With the help of a neat diagram explain the components of a Mass Spectrophotometer. (8)

(b) Write a short note on NOESY and COSY techniques. (3½ + 3½ = 7)

SECTION B

Short notes : (10 × 5 = 50)

3. Principle involved in Flame Photometry and its applications in pharmacy.

4. FAB analysis and applications.

5. Chemical shift and significance.

6. Capillary electrophoresis and applications.

7. Theory and applications of TGA.

8. Discuss two detectors of G.C.

9. Supercritical fluid chromatography.

10. Reverse phase HPLC technique and application.

11. EIMS — principle and application.

12. Fluorimetric analysis of Quinine sulphate and Thiamine.