FEBRUARY 2005

[KM 289] Sub. Code : 1001

M.Pharm. DEGREE EXAMINATION.

(Revised Regulations)

First Year

Paper 1 — 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 — (2 × 15 = 30 marks)

Long Essay :

1. Write a note on chromophores and their interaction with electromagnetic radiation in U.V. region on the basis of Woodward's rule.

2. Give an account of detectors used in HPLC.

SECTION B — (10 × 5 = 50 marks)

Short notes on :

3. Significance of Ilkovic Equation and Halfwave potential.

4. Principle and applications of affinity chromatography.

5. Different types of conductometric titrations.

6. Qualitative IR spectrophotometry.

7. Instrumentation and working of fluorimeter.

8. Principle involved in the biological assay of Digitalis and Insulin.

9. Chemical shift.

10. Spin-spin splitting.

11. Coupling constant.

12. C^{13} NMR.

AUGUST 2005

[KN 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

Theory : Two hours and forty minutes Theory : 80 marks

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

Answer ALL questions.

I. Long Essay : (2 × 15 = 30)

1. (a) Explain the principles underlying the ionization patterns in the field desorption and fast atom bombardment mass spectrometry. (8)

(b) Describe the general rules of fragmentation in mass spectroscopy. (7)

2. (a) What do you mean by Nuclear Overhauser Effect (NOE)? What are its applications? (7)

(b) Emphasize on the derivatization methods and the principles involved in on-line derivatization process in HPLC and gas chromatography. (4 + 4)

II. Short Notes : (10 × 5 = 50)

1. How are X-rays generated? What are the applications of X-ray diffraction?

2. How do you distinguish between 2-pentanone and 3-pentanone on the basis of their mass and nmr spectra?

3. Describe stationary phases in HPLC and GLC.

4. Describe the structural features of molecules responsible for quenching effect in fluorimetry.

5. What is meant by capillary electrophoresis? Discuss the different modes by which the capillary electrophoresis techniques are carried out.

6. Describe the applications of atomic absorption spectroscopy.

7. Classify ion exchangers used in chromatography. Write their applications.

8. Describe the instrumental features of a classical HPTLC.

9. Write construction and working of Electron Capture Detector. (ECD).

10. Explain the principle and methodology of Thermogravimetric analysis.

[KO 289] MARCH 2006 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

Theory : Two hours and forty minutes Theory : 80 marks

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

Answer ALL questions.

I. Long Essay : (2 × 15 = 30)

1. (a) Sketch a dispersive IR instrument and a Fourier Transform IR instrument. What is the difference between these instruments? (7)

(b) Give an account on the principle of mass spectroscopy. Select two compounds and comment on their fragmentation pattern. (8)

2. (a) Describe the principle and application of flame emission spectroscopy. (6)

(b) UV spectra arises from the absorption and emission by electronic transition. Explain this phenomenon with examples. (9)

II. Short notes : (10 × 5 = 50)

1. Describe the instrumental features and applications of LCMS.

2. Briefly explain the principle and instrumentation of capillary electrophoresis.

3. Explain the structural features affecting the fluorescent intensity. Why flourimeter is more sensitive and selective than absorption spectra?

4. Explain how X-ray diffraction methods can be used for quantitative analysis.

5. What is derivative spectroscopy? Explain its application with one example.

6. Give an account of various techniques available for carrying out the thermal analysis. Give the working of any one instrument.

7. Describe the limitations and strengths of GLC.

8. How will you differentiate the following compounds by NMR and Mass spectroscopy?

(a) $OH-CH_2-CH_2-OH$

(b) $CH_3-CH(OH)-CH_2-OH$.

9. Explain the construction and working principle of Time of Flight mass analyzer.

10. Discuss briefly the principle and instrumentation of HPTLC.

SEPTEMBER 2006

[KP 289] **Sub. Code : 2801**

M.Pharm. DEGREE EXAMINATION.

(Revised Regulations)

First Year

Paper I — MODERN PHARMACEUTICAL ANALYTICAL TECHNIQUES

(Common to all Branches)

Time : Three hours Maximum : 100 marks

Theory : Two hours and forty minutes Theory : 80 marks

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

Answer ALL questions.

I. Long Essay :

1. (a) Write the applications of thermogravimetric analysis, quoting suitable examples.

(b) Write a note on Radio immuno assays. (10 + 10 = 20)

2. Give a detailed account of principle, types of ion exchangers and chemistry involved with application of ion-exchange process in pharmaceutical analysis. Explain the factors that determine the distribution of ions between an ion-exchange resin and a solution. (15)

3. With reference to atomic, electronic and molecular factors, describe the theory underlying the following analytical techniques :

(a) Infra-red spectrometry

(b) NMR spectrometry

(c) Fluorimetry. (15)

II. Short notes : (6 × 5 = 30)

1. With a neat sketch, explain the working of a Barrier Layer Cell (BLC). Compare the sensitivity and application of BLC with the other detectors used in the detection of visible radiations.

2. Establish a mathematical relationship between concentration of fluorogenic substance and intensity of fluorescence. Give any two examples of a fluorogenic substance.

3. What is a base peak, M+1 peak, M+2 peak, parent ion peak and metastable ion peak in a mass spectrum? With the help of a suitable molecular structure show the formation of such peaks by fragmentation or such other process.

4. What are the different components of an atomic absorption spectrometer? What are their roles in the equipment?

5. What are bonded phase supports? List their advantages and applications in liquid chromatography.

6. What is chemical shift in NMR spectroscopy? Describe the various factors causing chemical shift with suitable examples.

MARCH 2007

[KQ 289] Sub. Code : 2801

M.Pharm. DEGREE EXAMINATION.

(Revised Regulations)

First Year

Paper I — MODERN PHARMACEUTICAL ANALYTICAL TECHNIQUES

(Common to all Branches)

Time : Three hours Maximum : 100 marks

Theory : Two hours and forty minutes Theory : 80 marks

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

Answer ALL questions.

I. Long Essay :

1. Explain the theory and principles of Atomic Absorption spectrophotometry. How will you eliminate spectral and chemical interference in Atomic Absorption Spectroscopy? Compare the sensitivity of Atomic Absorption Spectroscopy and Flame Photometry. (20)

2. With suitable diagram explain the construction and working of

(a) Halo cathode Lamp

(b) Graphite Furnace. (8 + 7 = 15)

3. Explain the following with respect to mass spectrometry

(a) Theory and principle of Electron Impact mass spectrometry with magnetic deflection analyser.

(b) Matrix Assisted Laser Disorption Ionisation

(c) Chemical Ionisation. (7 + 5 + 3 = 15)

II. Short notes on : (6 × 5 = 30)

1. Types of electronic transition and their use in analysis, with examples.

2. Construction and working of Interferometer in FIIR.

3. Difference between PMR and ^{13}C *NMR* with respect to sensitivity and Spin--Spin Coupling.

4. Capillary zone electrophoresis

5. Relation between chemical structure and fluorescence.

6. Principle and Pharmaceutical applications of DSC.

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MARCH 2007

[KQ 315] Sub. Code : 2851

M.Pharm. DEGREE EXAMINATION.

(Regulation 2006)

First Year

Paper I — MODERN PHARMACEUTICAL ANALYTICAL TECHNIQUES

(Common to all Branches)

Time : Three hours Maximum : 100 marks

Theory : Two hours and forty minutes Theory : 80 marks

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

Answer ALL questions.

I. *Long Essay :*

1. (a) Outline methodology of moving boundary electrophoresis technique. Write any two important applications of electrophoresis.

(b) What do you mean by MALDI-MS? Write the importance of this technique in mass spectroscopy. (10 + 10)

2. What are the different components of High performance liquid chromatograph? What are their roles in the equipment? Add a note on column types used in HPLC? (15)

3. What are the principles of spin-spin decoupling? Describe any three methods of decoupling. Write the significance of C-13 NMR spectroscopy in the structure elucidation of organic molecules. (15)

II. Short notes : (6 × 5 = 30)

1. What is circular dichroism? Explain this concept in relevance to optical rotatory dispersion.

2. Why is it necessary to apply statistical methods to analytical techniques? Add a note on chi-square test.

3. Write the principles involved in the techniques of HPTLC and Super critical fluid chromatography.

4. Outline the objectives of derivatization process in Gas chromatography. Mention the process and chemistry involved in derivatization of amino acids.

5. Write the infra-red absorption frequencies, mass fragmentation patterns and low resolution as well as high resolution nmr spectral features for 'phenyl-acetic acid'.

6. How do you calculate the absorption maximum wavelength for Dienes with Woodward-fisher rules?

SEPTEMBER 2007

[KR 289] **Sub. Code : 2801**

M.Pharm. DEGREE EXAMINATION.

(Revised Regulations)

First Year

Paper I — MODERN PHARMACEUTICAL ANALYTICAL TECHNIQUES

(Common to all Branches)

Time : Three hours — Maximum : 100 marks

Theory : Two hours and forty minutes — Theory : 80 marks

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

Answer ALL questions.

I. Long Essay : (20)

1. (a) Why are the absorption bands in a UV spectrum very broad when compared to IR or NMR spectra? What are the factors that govern the position and intensity of the absorption bands in UV spectrum? Give the analytical applications of UV-visible spectrophotometry.

(b) Discuss the general appearance of IR spectrum of normal alkane. What are the changes in the above spectrum seen upon the introduction of following structural residues?

(i) Alkene

(ii) Alcohol-OH

(iii) Carbonyl.

(2 × 15 = 30)

2. (a) Explain the theory of PMR Spectroscopy.

(b) What is Spin-spin splitting? Give the rules that characterise the spin-spin splitting of PMR resonance peak.

3. (a) Outline the instrumentation of mass spectrometry with special reference to different types of mass analyser.

(b) Write notes on :

(i) Mc Lafferty rearrangement.

(ii) Isotope effect in mass spectroscopy.

II. Short notes : (6 × 5 = 30)

1. Explain the principle of ESR. How do you compare ESR with NMR method.

2. Explain the principle and significance of interferences involved in Flame emission spectroscopy.

3. What is Bragg's Law? Explain its significance.

4. Discuss the significance of Student's *T*-test, *F*-test and Chi-square in statistical analysis.

5. Write an account on Fundamental Principles of ORD.

6. Explain the significance of different types of ions formed in Mass Spectra.

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SEPTEMBER 2007

[KR 315] **Sub. Code : 2851**

M.Pharm. DEGREE EXAMINATION.

(Regulation 2006)

First Year

Paper I — MODERN PHARMACEUTICAL ANALYTICAL TECHNIQUES

(Common to all Branches)

Time : Three hours Maximum : 100 marks

Theory : Two hours and forty minutes Theory : 80 marks

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

I. Long Essay :

1. (a) Explain diagrammatically the working of a spectrofluorimeter. (10)

(b) Discuss the theory of fluorescence and phosphorescence. (5)

(c) What are the applications of fluorimetry in quality control of drugs? (5)

2. (a) Describe different parts of a HPLC with a block diagram. (7)

(b) Explain the principles and applications of : (4 + 4 = 8)

(i) Super critical fluid chromatography.

(ii) Ion-exchange chromatography.

3. Discuss the following in detail : (3 × 5 = 15)

(a) Spin-spin coupling.

(b) Chemical shift.

(c) Proton exchange reaction.

II. Short notes : (6 × 5 = 30)

1. Write the applications of I.R. Spectroscopy in quality control of drugs and research.

2. Explain the principle of :

(a) Matrix assisted laser desorption.

(b) Fast atom bombardment ionisation.

3. Discuss the principle and applications of X-ray diffraction technique.

4. What is the principle involved in Radio immuno assay? What are its applications?

5. Explain the principle and applications of DTA.

6. What are the applications of AAS? Mention the advantages of AAS over Flame Photometry.

September 2008

[KT 315] **Sub. Code : 2851**

M.Pharm. DEGREE EXAMINATION.

(Regulation 2006)

First Year

Paper I — MODERN PHARMACEUTICAL ANALYTICAL TECHNIQUES

(Common to all Branches)

Q.P. Code : 262851

Time : Three hours Maximum : 100 marks

Answer ALL questions.

I. Long essay : (3 × 20 = 60)

1. (a) Explain different parts of a HPLC with block diagram.

(b) Discuss the working and applications of FT-IR spectroscopy.

2. (a) Explain diagrammatically the working of mass spectroscopy. Comment on fragmentation pattern of any two compounds.

(b) Discuss the principle and applications of NMR spectroscopy.

3. (a) Give an account on the principle of spectrofluorimeter.

(b) Discuss the working and applications of defectors used in Atomic absorption spectroscopy.

II. Short notes : (8 × 5 = 40)

(1) Explain the working principle of electron spin resonance spectroscopy. Mention the differences between ESR and NMR.

(2) Discuss the spin-spin coupling in detail.

(3) Explain the principle of differential scanning calorimetry.

(4) Discuss the principle and applications of super critical fluid chromatography.

(5) Explain the applications of NOESY and COSY.

(6) Discuss the working and applications of detectors used in Gas chromatography.

(7) Explain the interpretation of diffraction patterns and its applications.

(8) Discuss the principle and limitations of Electron Spin Resonance Spectroscopy.

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