PH 541.4

Reg. No. :

St Aloysius College (Autonomous) Mangaluru

Semester IV - P.G. Examination - M.Sc. Analytical Chemistry April- 2019

ORGANIC SYNTHETIC METHODS

Max. Marks: 70 Time: 3 Hours PART - A (5x2=10) 1. Answer any FIVE sub divisions of the following: a) Explain the selectivity or lack of it during catalytic hydrogenation reaction using metal and hydrogen. b) Write mechanism of Birch reduction reaction. c) How is peracids prepared? Compare the reactivity of substituted peracids for the epoxidation of alkenes. d) Write the reagents used for allylic and benzylic halogenations. Explain their synthetic importance. e) Describe the synthetic application of Robinson annulation reaction. f) Explain the role of catalyst during Friedel Crafts reaction. g) Write the importance of functional group interconversion in organic synthesis. Justify your answer by taking suitable example. h) What is the criteria used to choose a protecting group from multiple options? PART - B Answer any FIVE of the following choosing at least one (5x12=60)full question from each unit: UNIT - I 2.a) i) Differentiate the reduction reaction of alkene and carbonyl compounds. ii) Write the synthetic applications of LiAlH4 and NaBH4. Comment on their differential reactivity. (4) b) i) Write the mechanism of Birch reduction. ii) Write a note on Wolf-Kishner reduction reaction and give its mechanism. (4) c) Discuss the stereochemistry of reduction with LiAlH4. (4) 3.a) Describe the reduction of nitro compounds by the following: Pd-C/H2 and LiAlH4. (4) b) How is diimide prepared? Write their synthetic applications highlighting and stereochemistry and mechanism. (4) c) Describe the mechanism of homogeneous catalytic hydrogenation using Ru-catlyst. (4) UNIT - II 4.a) Describe the application of chromium based oxidation reactions in How is selectivity improved with modified organic synthesis. chromium reagents? (4)

- b) How is osmium tetroxide useful as an oxidant? Write mechanism of oxidation reaction. What are the limitations of this reagent? (4)
- c) Write the mechanism of addition of bromine to symmetrical and unsymmetrical alkenes. Comment on the major products formed. (4)
- Describe the applications of manganese based oxidants used in organic synthesis. Explain the selectivity of such reagents using different substrates.
- b) Write the mechanism of ozonolysis of an alkene. Write the product of ozonolysis of 3-methyl 2-pentene and cyclohexene. (4)
- c) Write the synthetic application of t-butyl hydroperoxide. Explain how
 it is used in enantioselective epoxidation reactions.

UNIT - III

- 6.a) Explain the factors affecting the rate of Diels-Alder reaction. Write two applications of Diels Alder reactions. (4)
- b) Discuss the strategy employed for the synthesis of 7-methoxy tetralone. (4)
- c) Explain the synthetic scheme for penicillin V. (4)
- 7.a) i) Differentiate chemoselectivity from stereoselectivity using an example.
 - ii) Explain Thorpe condensation reaction. (4)
- b) Provide the synthetic scheme for cubane.
- Illustrate the following statement by selecting two examples and also suggest mechanism for one of the reaction.
 - Statement-Carbon-carbon bond reactions are useful synthetically.

UNIT - IV

8.a) Suggest retrosynthetic schemes for the following:

NO2

- b) i) What is 1,3-diX relationship? Explain with an example.
 - ii) Give the retrosynthetic strategy for benzocaine.

(4)

(4)

(4)

(4)

- i) Explain the terms synthon and synthetic equivalents using suitable example.
- ii) Provide the retrosynthetic scheme for phenacetin.

- (4)
- i) Suggest a reagent for the protection of carbonyl compounds.
 Write the protection and deprotection reactions.

ii) Suggest suitable retrosynthetic strategy for following 1,4-difunctional compound.

b) Predict all the possible disconnections and suggest a synthetic scheme for the following molecule. Describe the rationale of your chosen synthetic method.

c) Provide the retrosynthetic analysis of 4-hydroxy-4-methylpentanone. (4)

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St Aloysius College (Autonomous)

Mangaluru

Semester IV - P.G. Examination - M.Sc. Analytical Chemistry April- 2019

SPECTROSCOPIC METHODS OF ANALYSIS

Max. Marks: 70 Time: 3 Hours PART - A (5x2=10) 1. Answer any FIVE sub divisions of the following: a) How many ESR lines are expected for methyl radical? Sketch the spectrum for the same. b) Among the following which elements do not produce Auger spectrum and why? iv) He i) N ii) H₂ iii) C c) How is atomization of liquid sample achieved in inductively coupled plasma? d) What is ionization interference? How is it corrected in atomic absorption determinations? e) Justify that phosphorescence spectrum always occurs at longer wavelength than that of fluorescence spectrum. f) Give the structure of any two fluorimetric reagents and mention its use. g) How are nephelometry and turbidimetry similar and different? h) Illustrate the use of Debye-Scherrer method of x-ray diffraction in the determination of d. PART - B Answer any <u>FIVE</u> of the following choosing at least one (5x12=60) full question from each unit: UNIT - I 2.a) Explain the term nuclear quadruple coupling constant. Calculate (5)NQR transition frequencies for a nuclens with $I=\frac{5}{2}$ assuming $\eta=0$. b) What is meant by isomer shift? Explain isomer shift for Fe2+, Fe+3, (4) Sn2+ & Sn4+ ions. c) Predict the Mossbauer spectrum of $[Fe(CN)_6]^4$ and explain the (3) spectrum. 3.a) Give an account of hyperfine splitting in ESR. Sketch and interpret (5)the spectrum of 1, 4-benzosemiquinone and deuterium atom. (4) b) Discuss the following. Auger effect i) g-value and its determination in ESR spectroscopy c) Draw energy level diagram and calculate transition frequencies for (3) nucleus having $I = \frac{3}{2}$ assuming $\eta = 0$ in NQR spectroscopy.

UNIT - II

4.a)	How is hollow cathode lamp different from electrodeless discharge lamp in Atomic Absorption Spectrophotometer? Explain their mode	(5)
	of working.	
b)	Describe the excitation sources of plasma emission spectroscopy.	(4)
c)	What are the factors that influence the intensity of emitted radiation	(3)
	in a flame photometer? Explain.	
5.a)	Total consumption burner lowers that sensitivity, whereas, premix	(5)
	burner provides higher sensitivity. Why? Explain their role in atomic	
	absorption determinations.	
b)	Discuss the following interferences of flame photometric	(4)
	determinations.	
	i) Spectral interferences	
- 1	ii) Chemical interferences	
C)	What are the differences between atomic absorption spectroscopy	(3)
	and flame emission spectroscopy?	
	UNIT - III	
6.a)	The second secon	(5)
	block diagram.	
b)	Discuss the utility of fluorimetry in the determination of Vitamin B_1	(4)
	and Vitamin B ₂ .	
	How do the pH and dissolved oxygen affect fluorescence and phosphorescence?	(3)
7.a)	Explain the following methods of deactivation of excited molecules	(5)
	with the help of Jablonski diagram.	
	i) Vibrational relaxation	
	ii) Intersystem crossing	
b)	What is fluorescence quenching? Discuss the types of it.	(4)
c)	Deduce an expression for the relation between phosphorescence	(3)
	intensity and concentration. Draw a typical curve representing it.	(5)
	UNIT - IV	
8.a)	How the choice is made between nephelometry and turbidimetry.	
	Explain the factors affecting both the techniques.	(5)
b)	Describe the rotating crystal method of x-ray diffraction.	
c)	Discuss the use of optical rotator dispersion in quantitative analysis.	(4)
9.a)	Give an account of the following.	(3)
	i) octant rule	(5)
	ii) circular dichroism	
b)	Sketch a block diagram of x-ray absorptiometer and avalate in	(4)
c)		
	Give an account of the acid calatyzed mutarotation of glucose.	(3)

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Semester IV - P.G. Examination - M.Sc. Analytical Chemistry
April - 2019

CHEMISTRY OF POLYMERS AND NATURAL PRODUCTS Time: 3 Hours Max.Marks:70

PART - A

1) Answer any FIVE sub divisions of the following

(5×2=10)

- a) Write the structure of the repeating unit for each of the following polymer:
 - i) Poly (ethylene terephtholate)
 - ii) Teflon
 - iii) Nylon 6,6
 - iv) polypropylene
- Calculate the degree of polymerisation of Nylon-6, 6 with a molecular weight of 1x10⁴g/mole.
- c) Among polyethylene and polystyrene, which polymer exhibits a higher Tg value? Why?
- Sketch the characteristic DSC curve of a semicrystalline polymer sample and mention the various features.
- e) What happens when cinchonine is oxidized with chromic acid?
- f) Reserpic acid gives γ- lactone on heating with acetic anhydride. What does it indicate?
- g) Formulate the following reaction

 $\alpha - pinene \xrightarrow{cold.aq.KM_nO_4} ? \xrightarrow{C_rO_3} \text{ketocarboxylic acid}$

h) How morphine is converted into morphol and morphenol?

PART - B

Answer any FIVE of the following choosing at least one full question from each unit.

(5x12=60)

UNIT - I

- 2. a) Explain the use of GPC in isolation and purification of polymers. (5)
 - b) With a suitable example, write the mechanism of ionic polymerisation.
 - c) Give an account of viscoelastic behaviour of polymers. (3)
- a) Describe fractional precipitation of polymers.
 - Explain how the structure of a polymer influences the following properties.
 - i) tensile strength ii) crystallinity
 - Solution viscosity measurement give an idea about the size and shape of polymer molecules in solution. Justify.

Contd.2

UNIT - II

4.	a)	Explair of a po	osomometric method of determination of molecular weight lymer.	(4
ı	0)	crystall	you correlate that the glass transition temperature, linity and melting point of a polymer with the structure of lymer? Give suitable example.	(4
(:)		the principle of visocometric method of determination of lar weight of polymers.	(4)
5. a)		differential thermal analysis technique in polymer erization.	(4)
b)	Explain	the procedure for determining Tg employing DSC technique.	(4)
С)	Give an	account of mechanical properties of polymers.	(4)
			UNIT - III	
5. a)	Describe	e the synthesis of Papaverine.	(4)
b		undergo	eated with dilute hydrochloric acid, thebaine rapidly es rearrangement to Thebenine. Write down the mechanism in this conversion.	(4)
c)		Convert	ethyl quininate to (I)-quinine.	(4)
. a)		Suggest tryptami	the suitable steps for the synthesis of reserpine from ne.	(4)
b)	(Outline t	he reactions that indicate	(4)
		i) ii)	Presence of methylenedioxy group in papaverine. Attachment of -CHOH group directly to the benzene ring in adrenaline	
c)		Discuss to	he evidences that led to the determination of structure of	(4)
			UNIT - IV	
a)	S	ketch th	e synthesis of Farnesol.	(4)
b)	G	live evid	ences for the presence of cyclobutane ring in $lpha$ -Pinene.	(4)
c)	A	ccount f	or the position of hydroxyl and acid group in Abietic acid.	(4)
a)			ow the structure of santonin was established by on studies.	(4)
b)	Fo	rmulate	the steps involved in the synthesis of zingiberene.	(4)
	Gi	ve the e	vidences for the presence and position of the conjugated zingiberene.	(4)
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St Aloysius College (Autonomous) Mangaluru

Semester IV - P.G. Examination - M.Sc. Analytical Chemistry April- 2019

APPLIED ANALYSIS AND AUTOMATION

Max. Marks: 70 Time: 3 Hours PART - A (7x2=14) Answer any <u>SEVEN</u> sub divisions of the following: a) Name the different types of catalysis with examples. b) Mention any four types of methods to identify the reaction rate. c) Explain enzyme specificity. d) What are the factors affecting dispersion? e) Mention the chemical components of snake venom. f) What is milk adulteration? How does it affect health? g) Explain quality acceptance. Define ISO and its importance in pharma industry. Mention the importance of quality assurance in food industry. PART - B Answer any <u>FOUR</u> of the following choosing at least one (4x14=56) full question from each unit: UNIT - I (7)2.a) Give an account of second order reaction and its importance. b) Explain the determination of LDH enzyme in blood samples. Why it (7)is important? (7)3.a) Discuss about catalyzed reactions with suitable examples. b) Mention the steps involved in the determination of iodide and cobalt (7) in biological samples. UNIT - II 4.a) Give the significance and the importance of BUN analyzers. (8) b) Explain the determination of lead and mercury in biological material (6) 5.a) Discuss the advantages and disadvantages of automated systems. (8) b) Explain the analysis of fat contents and minerals in milk and butter. (6) **UNIT - III** 6.a) Give an account of ISO14001 and laws related to quality control in (7) general industries. b) Explain the steps involved in quality control role in the production (7)and the finished products in pharma industries. 7.a) Explain in detail the importance of quality assurance and ISO/IEC (7)17025. b) Explain the case study analysis of role of quality control in fertilizer (7) and pharmaceutical industries.

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se	mester IV - P.G. Examination - M.Sc. Analytical Chem	nistry
	ORGANIC SYNTHETTE	
	ORGANIC SYNTHETIC METHODS	
rime		arks: 70
1. a)	Answer any <u>FIVE</u> sub divisions of the following: (5) Illustrate the use of PtO ₂ in catalytic hydrogenation reactions.	5x2=10)
b)	Distinguish Wolf-Kishner reduction from Clemmensen reduction.	
c)	Give an account of halogenations of carbonyl compounds.	
d)	Explain how is S and Se useful in carrying out dyhydrogenation react	ions.
e)	Describe the limitation of Friedel Crafts reaction.	
f)	Justify the following statement using an example;	
	Protecting groups may be necessary for multistep organic synthesis.	
g)	Write a short note on 1,3-dipolar cycloaddition reactions.	
h)	Retrosynthetic analysis may provide multiple options. What criteria of	ne
11-51	may adopt to select most suitable synthetic route?	
	PART - B	
	full question from each unit:	12=60)
	UNIT - I	
2.a)	Give an account of selective reducing agents for functional groups esters, carbonyl and alkenes.	
b)	Describe the mechanism hydrogenation reactions using Rh	(4)
-,	complexes.	(4)
c)	How is diborane prepared? What are hindered boranes? Write the	2000
	general mechanism of reduction of alkenes using borane reagents.	(4)
3.a)	Describe the reduction reactions of organic substrates under the following conditions and comment on their selectivity.	
	Pd-H ₂ ; Pd-BaSO ₄ ; Pd-CaCO ₃ .	(4)
b)	Describe the synthetic applications of diimide based reagents.	(4)
c)	How is LAH prepared? Describe the mechanism of reduction of	
	carbonyl compounds using LAiH. Write a note on other hydrides used	
	as reducing agents commenting on their selectivity.	(4)
	UNIT - II	
4.a)	i) Jones reagent	
b)	ii) O ₅ O ₄	(4)
9)	How is lead tetraacetate useful as an oxidant? Write the reaction mechanism. Mention the limitations of this reagent.	(4)

c) Describe the reagents used to achieve benzylic and allylic

brominations.

(4)

5.a) Explain the selectivity or lack of it for the following oxidants; KMnO₄; MnO₂. Highlight the substrates and the reaction conditions used for such reactions.

b) What is ozonolysis reaction? Write the product of ozonolysis of

(4)

cyclohexene. Give the reaction mechanism.

(4)

(b)

(c) Write the synthetic applications of periodic acid providing suitable

(4)

 Write the synthetic applications of periodic acid providing suitable reaction mechanism.

UNIT - III

6.a) Give a brief account of Ring cleaving reactions. (4)

b) Describe the synthetic methods used for the preparation of cubane. (4)

c) Explain the structural aspects of biotin. Write its importance. (4)

7.a) i) Explain stereo selective reaction using an example.

ii) Explain Retro Diels-Alder reaction. (4)

 b) Write a note on Arndt-Eistert homologation and Dickman cyclization reaction.

(4)

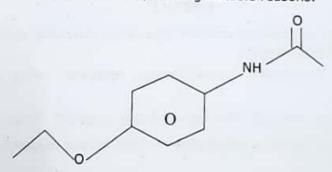
 Illustrate the following statement by selecting two examples and suggest mechanism for one of the reaction.

Reactions involving C-C bond formation are useful systematically.

(4)

UNIT - IV

8.a) Suggest retrosynthetic schemes for the following. Rationalize your pick among the methods providing suitable reasons.



(4)

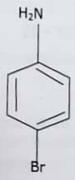
b) i) What is 1,3-diX relationship? Explain with an example.

ii) Provide synthetic route for bezocaine using retrosynthetic strategy.

(4)

c) i) Why do we use synthetic equivalents when planning an organic synthesis though it might enhance the number of steps?

ii) Provide the retrosynthetic scheme for the for the following:



(4)

(4)

- g.a) Discuss the general methods for the protection and deprotection of carbonyl and alcoholic groups.(4)
 - b) Predict all the possible disconnections and suggest a synthetic scheme for the following molecule. Describe the rationale of your chosen synthetic method.

c) Provide the retrosynthetic analysis of 6-methoxy indole-3-acetic acid. (4)

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St Aloysius College (Autonomous) Mangaluru

Semester IV - P.G. Examination - M.Sc. Analytical Chemistry April - 2018

SPECTROSCOPIC METHODS OF ANALYSIS

Time: 3 Hours

Max.Marks:70

PART - A

1) Answer any FIVE sub divisions of the following

(5×2=10)

- a) How many lines expected in spectrum of naphthalene anion?
- Mossbauer spectrum of Fe (CO)₅ shows a doublet at liquid dinitrogen temperature. Give reason.
- c) Total consumption burner lowers the sensitivity, where as premix burner provides higher sensitivity in atomic absorption spectroscopy why?
- d) How is concentration of metal determined by Flame photometer?
- e) Luminescent radiation is of higher wavelength than that of exciting radiation. Why?
- f) What is the relation between fluorescence intensity and concentration?
- g) Differentiate between i) K_α&K_β ii) L_α&L_β x-ray lines.
- h) What is cotton effect? Plot absorption curves for the positive and negative cotton effects.

PART - B

Answer any FIVE of the following choosing at least one full question from each unit.

(5x12=60)

UNIT - I

- Discuss how photoelectron spectroscopic technique is used to study the valence and core electron binding energy of an atom in a molecule.
 - Explain the ESR spectrum of [Mn (H₂O)₆]²⁺ on the basis of Kramer's degeneracy.
 - c) Calculate the NQR transition frequency for the nucleus in an axially symmetric field with I=3/2.
- Discuss the following aspects of Mossbauer spectroscopy.

 I) Isomer shift | II) Quadrupole interactions.

 (5)
 - b) Draw the energy level diagram and calculate various Quadrupolar energies for a nucleus with I=1, assuming $\eta=0$. how many transitions are expected?
 - c) Sketch and interpret the ESR spectrum of (SO₃)₂ NO anion. (3)

UNIT - II

- 4. a) Explain the instrumentation of Flame photometer. (5)
 - b) What are spectral and ionization interferences? How are these corrected in Flame photometer? Explain.
 - c) Explain the process of atomization in inductively coupled plasma.

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Ch	HOURS HOURS AND NATURAL PRODUCTS Max.Marks:70	
	PART - A	
1) Ans	wer any FIVE sub divisions of the following $(5\times2=10)$	1)
a)	Differentiate between chain growth and step growth polymerization.	
b)	Calculate the average molecular weight of a polystrene sample with	
	average degree of polymerisation of 1000.	
c)	Tq and Tm of polystyrene are higher than HDPE. PVC has still higher Tg and Tm Why?	
d)	Compare the basis between TGA and DTA.	
e)	What is the use of Emble degradation in alkaloid chemistry?	(5.)
f)	Piperonylic acid contains methylenedioxy group. Prove the statement.	
g)	Explain by means of structural formulae the following reactions.	35
	Zingiberene $\xrightarrow[acetylenedicarboxylkate]{Dimethyl}$ product $\xrightarrow[acetylenedicarboxylkate]{pyrolysis}$?	
h)	State special isoprene rule. Identify the isoprene units in Abietic acid.	
	PART - B	
Answe	er any FIVE of the following choosing at least one full question fr	om
each ι	init. (12x5	
	UNIT - I	
2. a)	Give the classification of polymers with examples for each class.	(4)
b)	A protein sample consists of an equimolar mixture of haemoglobin (M=15.5 kg/mole) ribonuclease (M=13.7 kg/mole) and myoglobin	(4)
	(M=17.2 kg/mole). Calculate the number average and mass average masses.	
c)	What is crystallinity of a polymer? What are the requirements for the crystallinity of a polymer.	(4)

3. a) Explain chain growth polymerization.

b) Show that the weight average molar mass is generally twice the

number average molar mass using polydispersity index.

c) Discuss the Flory-Huggins theory of polymer dissolution.

(4)

(4)

(4)

Contd.2

9.

UNIT - II

 a) Explain the experimental procedure of determination by molecular weight of polymers by viscometric technique. 	(4)
b) Define Glass transition temperature. Discuss the structural factors that influence the Tg.	(4)
c) Discuss the method of vapour phase osmometry, in the determination of polymer molecular weight.	(4)
Explain differential scanning calorimetric technique of characterisation of polymers.	(4)
 Outline the principle of end -group analysis in determination of molecular weight of polymers. 	(4)
 c) Explain the mechanical properties of polymers with suitable examples. 	(4)
UNIT - III	
6. a) Explain the use of Hofmann's degradation in the determination of structure of an alkaloid.	(4)
 Outline the various reactions that lead to the structure of Papaverine. 	(4)
c) Give the synthesis of piperine.	(4)
 a) In brief explain how the following products are obtained from morphine. 	(4)
i) Morphol ii) methyl morphol iii) Morphenol	
iv) Methylmorphenol	
b) How is the structure of meroquinene established?	(4)
c) Outline the synthesis of ephedrine.	
UNIT - IV	(4)
8. a) Indicate the reagents that are used to bring about the following	
conversions:	(4)
Pinene glycol \rightarrow Pinonic acid \rightarrow pinic acid \rightarrow is norpinic acid.	
Give the structures of all these compounds.	
b) How do you deduce the positions of the following in abietic acid?	(4)
i) methyl groups ii) double bonds	1,500
c) Outline the synthesis of camphors.	(4)
. a) How do you fix up the positions of three double bonds in	(4)
zingiberene ? Write the absolute configuration of it.	(4)
b) Enumerate the evidences in support of the structure of Santonin.	(4)
c) Write the synthesis of menthol.	775
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Semester IV - P.G. Examination - M.Sc. Analytical Chemistry April- 2018

	APPLIED ANALYSIS AND AUTOMATION	
ame:	3 Hours Max. Marks	: 70
1. a)	PART - A	=14)
b)	Mention the importance of enzyme specificity.	
c)	List out differences between automatic and automated system.	
d)	What are the differences between COD and BOD?	
e)	Explain quality assurance.	
f)	Mention the types of ISO.	
g)	Define order of reaction and its importance.	
h)	Explain any four disadvantages of organophosphates.	
1)	What is the importance of QA and QC in pharma industry?	
	PART - B	
	Answer any <u>FOUR</u> of the following choosing at least one (4x1	4=56)
	full question from each unit:	
	UNIT - I	(3)
2.a)	Discuss determination of reaction rate by conductivity method.	
b)	Give an account of micro determination of selenium in complex mixture.	
c)	the uses of enzyme catalysis? Give example.	(4)
d)	and first order reaction?	(3)
	Discuss the determinants of LDH enzyme.	(4)
3.a)	Discuss the determination of reaction rates. Discuss any two methods for determination of reaction rates.	(4)
b)	Discuss any two methods to Derive integral form of Ist order reaction.	(3)
c)	Explain analysis of multicomponent reaction.	(3)
d)	UNIT - II	
	Explain segmented flow method.	(4)
4.a)	the salding of automatic elemental analyzer.	(3)
b)	discrete method.	(4)
C		(3)
ď		(3)
5.a		(4)
b) Explain flow injections analysis.	
С	the determination of infosture and	
	samples	(3)
d	Explain compositions of milk and milk products.	Contd2

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	UNIT - III	
6.a	Mention the procedures for quality acceptance.	(3)
b)	Discuss cost aspects of quality decisions.	(3)
c)	Explain current trends in quality control.	(4)
d)	Write a note on ISO 17025 series.	(4)
7.a)	Discuss ISO 14001 series and its application to pharma industry.	(4)
b)	Explain laws related to quality control in Indian context.	(4)
c)	Explain quality control in raw materials and finished products.	(3)
d)	Mention the importance of quality aspects of specification.	(3)