SPECTROSCOPIC ANALYSIS OF ORGANIC COMPOUNDS

PART-4, PPT-4, SEM-5, CC-12P

Dr. Kalyan Kumar Mandal Associate Professor St. Paul's C. M. College Kolkata

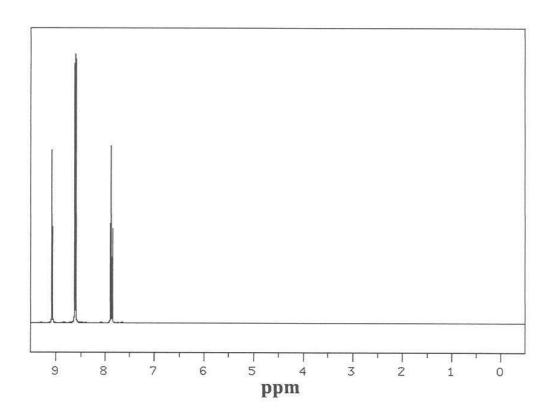
CONTENTS SPECTROSCOPIC ANALYSIS, PART-4

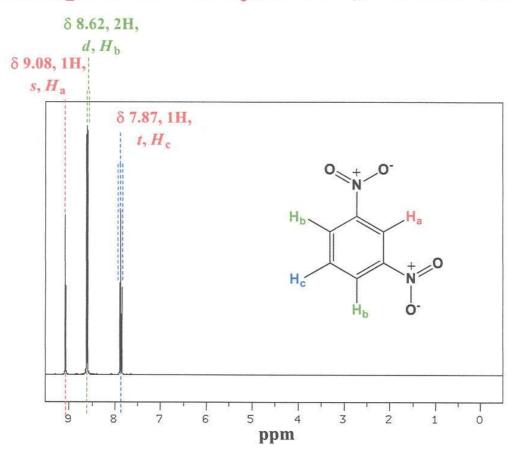
¹H NMR and IR Spectral Analysis of

- 1. 1,3-Dinitrobenzene
- 2. trans-Cinnamic acid

¹H NMR Spectrum of 1,3-Dinitrobenzene

400 MHz, CDCl₃: δ (in ppm): 9.08, 8.62, and 7.87





This Lecture is prepared by Dr. K. K. Mandal, SPCMC, Kolkata

 Spectrum is recorded in 400 MHz instrument in CDCl₃ using TMS as internal standard

Chemical shift (δ in ppm)	Number of hydrogens	Splitting pattern	Probable assignment
9.08	1H	S	H_{a}
8.62	2H	d	H_{b}
7.87	1H	t	$H_{\rm c}$

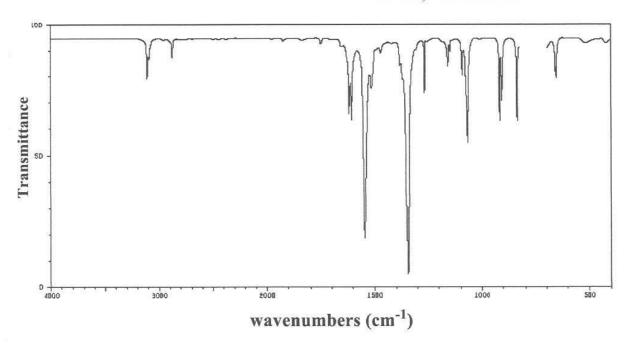
- 1. δ 9.08:1H, s, H_a, proton ortho to both -NO₂ groups: This proton (H_a) is highly deshielded due to (i) diamagnetic anisotropic effect of the aromatic moiety (ring current effect), ii) diamagnetic anisotropic effect of the N=O of both the -NO₂ groups, and (iii) presence of strongly electron withdrawing -NO₂ groups (with -I and -R effects) that withdraws electron density from the ring and makes the aromatic protons highly electron deficient.
- H_a appears as a *singlet* as it has no neighbouring proton to couple with (*meta*-coupling is ignored).

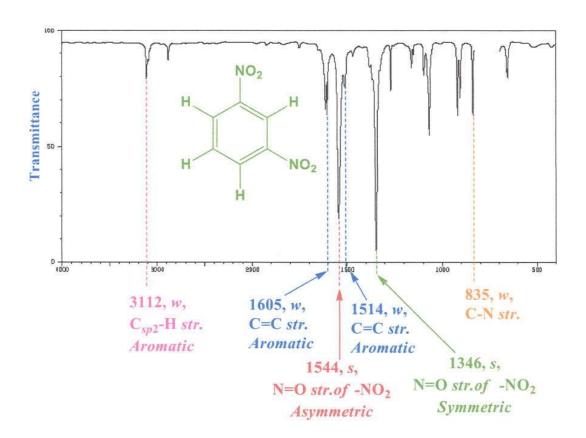
- 2. δ 8.62: 2H, d, H_b, proton ortho to one -NO₂ and para to other -NO₂ group: The proton, H_b is also highly deshielded due to (i) diamagnetic anisotropic effect of the aromatic moiety (ring current effect), ii) diamagnetic anisotropic effect of the N=O of the -NO₂ group which is ortho to it, and (iii) presence of strongly electron withdrawing -NO₂ (with -I and -R effects) groups that withdraws electron density from the ring and makes the aromatic protons highly electron deficient.
- H_b proton appears as a *doublet* as it couples with the proton H_c (*ortho*-coupling).

- 3. δ 7.87: 1H, t, H_c , proton meta to both -NO₂ groups: The proton, H_c is deshielded due to (i) diamagnetic anisotropic effect of the aromatic moiety (ring current effect), and (ii) presence of strongly electron withdrawing -NO₂ groups (with -I and -R effects) that withdraws electron density from the ring and makes the aromatic protons highly electron deficient.
- $H_{\rm e}$ proton appears as a *triplet* as it couples with the *two* $H_{\rm b}$ protons (*ortho*-couplings).

IR Spectrum of 1,3-Dinitrobenzene

FT-IR (CCl₄): Wavenumbers (cm⁻¹): 3112, 1605, 1544, 1514, 1346, and 835





This Lecture is prepared by Dr. K. K. Mandal, SPCMC, Kolkata

• Spectrum is recorded in CCl₄ solution.

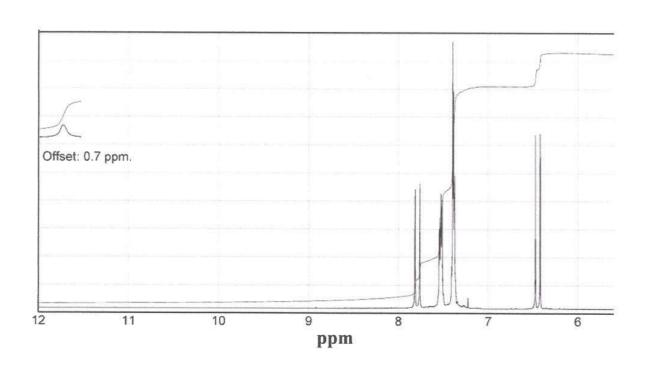
Wavenumbers (cm ⁻¹)	Nature	Probable Assignment
3112	w	C _{sp2} -H stretching; Aromatic
1605	w	C=C stretching; Aromatic skeletal vibrations
1544	S	N=O stretching of -NO ₂ ; Asymmetric
1514	w	C=C stretching; Aromatic skeletal vibrations
1346	S	N=O stretching of -NO ₂ ; Symmetric
835	w	C-N stretching of Ar-NO ₂

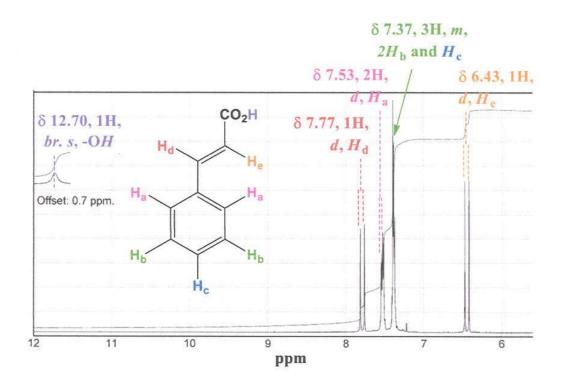
- 1. 3112: C_{sp2} -H stretching: These absorptions are due to strong C_{sp2} -H bonds of the aromatic moiety. The absorption occurs at a higher region which is due to the presence of small atom in the bond on one hand and hydrogen atom is attached to a sp^2 hybridized carbon atom on the other.
- 2. 1605 and 1514: C=C stretchings: These vibrations due to the conjugated aromatic system (*aromatic skeletal vibrations*) appear at a lower wavenumber than that of the nonconjugated system due to lowering of C=C bond strength as a result of conjugation.

- **3.** 1544 and 1346: N=O stretchings of -NO₂, Asymmetric and Symmetric: These are the characteristic absorptions for AB₂ type of grouping (-NO₂) present in the molecule. Two bands are due to vibrational coupling. Being a double bond, this absorption occurs at a lower wavenumber region because N=O assumes partial double bond character due to (i) resonance within -NO₂ group and (ii) -NO₂ group is conjugated with the aromatic ring.
- 4. 835: C-N stretching: This absorption appeared at lower wavenumber region as (i) it is a single bond and (ii) the bond involves a higher reduced mass.

¹H NMR Spectrum of trans-Cinnamic acid

300 MHz, CDCl₃: δ (in ppm): 12.70, 7.77, 7.53, 7.37, and 6.43





This Lecture is prepared by Dr. K. K. Mandal, SPCMC, Kolkata

 Spectrum is recorded in 300 MHz instrument in CDCl₃ using TMS as internal standard

Chemical shift (δ in ppm)	Number of hydrogens	Splitting pattern	Probable assignment
12.70	1H	br. s	-OH
7.77	1H	d	$H_{ m d}$
7.53	2Н	d	H_{a}
7.37	3Н	m	$2H_{\rm b}$ and $H_{\rm c}$
6.43	1H	d	$H_{ m e}$

- 1. δ 12.7: 1H, br. s, Ar-COOH: Deshielding of O-H proton is due to (i) local diamagnetic effect of the attached electronegative oxygen atom, (ii) mesomeric interaction caused by EW carbonyl group and (iii) hydrogen bonding (in the dimer) that decreases the electron density around the proton.
- Carboxylic acid proton appears as a *broad signal* (*br*, *s*) due to (i) chemical exchange, and ii) intermolecular hydrogen bond between C=O and -OH moieties (iii) absence of any coupling partner.
- 2. δ 7.77: 1H, d, H_d , β -proton of -CH=CH-COOH: This vinylic proton is deshielded by the diamagnetic anisotropy of the C=C. Moreover, H_d proton is further strongly deshielded due to conjugation with EW carbonyl group (exerting -R effect) which renders β -C of the α , β -unsaturated carbonyl system electron deficient.

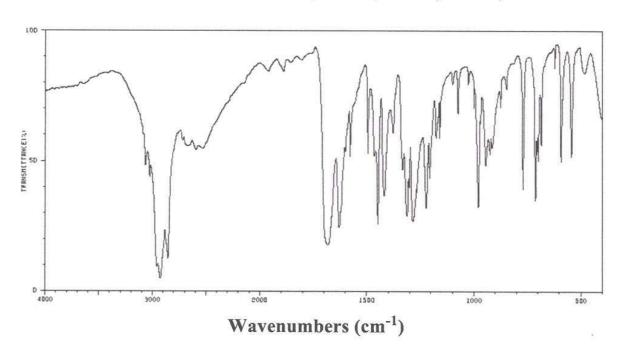
- The proton H_d appears as a *doublet* as it couples with adjacent H_e proton (*trans*-coupling).
- 3. δ 7.53: 1H, d, H_a, protons ortho to -CH=CH-COOH group: These two protons are most deshielded among the aromatic protons in this compound which is due to diamagnetic anisotropic effect of (i) the aromatic moiety (ring current effect) and ii) C=C of -CH=CH-COOH system. This proton is further deshielded as it is in close proximity to EW -CH=CH-COOH system.
- They appear as a *doublet* due to coupling with H_b proton (*ortho*-coupling).

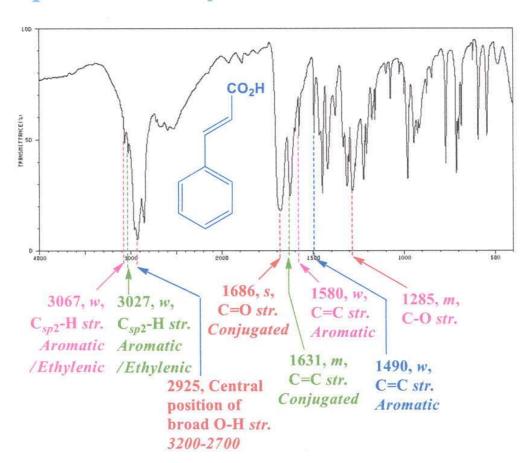
- 4. δ 7.37: 3H, m, 2H_b and H_c, protons meta and para to -CH=CH-COOH group: These protons are deshielded due to diamagnetic anisotropy of the aromatic ring (ring current effect) and EW effect of -CH=CH-COOH group. However, these three protons are less deshielded compared to H_a protons as they do not fall in the deshielding region of C=C bond because of greater distance from it. The weakly EW -CH=CH-COOH group does not produce too much difference in electron density around m- and p-protons. Therefore, H_b and H_c protons have almost identical chemical shift.
- H_b protons couple with H_a and H_c (both are *ortho*-couplings) and H_c proton couples with two H_b protons (*ortho*-coupling). So, even if weak *meta*-coupling is ignored, each of them appears as a *triplet* and these *triplets* are close enough and appear as *multiplets*.

- 5. δ 6.43: 1H, d, H_e , α -proton of -CH=CH-COOH: This vinylic proton is deshielded by the diamagnetic anisotropy of the C=C and C=O, and EW effects of -COOH. Due to larger distance from the benzene ring, H_e is less deshielded compared to H_d .
- H_e appears as a *doublet* as it couples with adjacent H_d proton (*trans*-coupling).

IR Spectrum of trans-Cinnamic acid

FT-IR (CCl₄): Wavenumbers (cm⁻¹): 3200-2700, 3067, 3027, 1686, 1631, 1580, 1490, and 1285





This Lecture is prepared by Dr. K. K. Mandal, SPCMC, Kolkata

- 1. 3200-2700: O-H stretching of the dimer: This broad absorption band for O-H bond centres near 2925 cm⁻¹. Vibrational frequency associated with the O-H bond is very high because (i) O-H bond is very strong and (ii) this bond contains a small atom hydrogen (reduced mass decreases, frequency increases). Broad signal is due to H-bonding. Hydrogen bonding causes lengthening of the original O-H bond and it becomes weaker. Hence force constant of O-H bond decreases.
- 2. 3067 and 3027: C_{sp2} -H stretching: These absorptions are due to strong C_{sp2} -H bonds for aromatic/ethylenic unit. The absorption occurs at a higher region which is due to the presence of small atom in the bond on one hand and hydrogen atom is attached to a sp^2 hybridized carbon atom on the other.

- **3. 1686: Conjugated C=O stretching:** Carbonyl group is conjugated with C=C which in turn conjugated with the aromatic moiety. As a result, due to mesomeric interaction C=O bond assumes single bond character in the charged canonical form. Bond order of C=O bond decreases and hence C=O absorption occurs at lower than 1715 cm⁻¹.
- 4. 1631: Ethylenic C=C stretchings: The vibration due to the conjugated ethylenic system (conjugated with C=O on one hand and with the aromatic moiety on the other). Therefore, vibration due to this bond appears at a lower wavenumber than that of the nonconjugated ethylenic system due to lowering of C=C bond strength.

- 5. 1580 and 1490: Aromatic C=C stretchings: The vibration due to the conjugated aromatic system (aromatic skeletal vibrations) appears at a lower wavenumber than that of the nonconjugated system due to lowering of C=C bond strength.
- 6. 1285: C-O stretching of -CO-OH: Appearance at lower wave number of this bond is due to (i) the vibration involves a single bond and (ii) masses of both the constituent atoms are relatively large unlike C-H, O-H, etc. However, this bond is stronger as carbon atom is a part of the unsaturated system.



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Add On Course (Spectroscopic Analysis of Organic Compound)

TIME TABLE (8 Jan.- .30 Jan. 2021) (2020-21)(PG)

Class	Time	Friday(08/01/2021)	Saturday(09/01/2021)
M.Sc I&II	11.20 am-12.08pm	Theory (SDJ)	Theory (NDN)
	12.08pm-12.56pm	Theory(NDN)	Theory(NMG)
	3.00 pm-6.00pm	Practical(SDJ)	Practical(NMG)

Class	Time	Friday(15/01/2021)	Saturday(16/01/2021)
M.Sc I&II	11.20 am-12.08pm	Theory (VSK)	Theory (NMG)
	12.08pm-12.56pm	Theory(PSP)	Theory(SDJ)
	3.00 pm-6.00pm	Practical(VSK)	Practical(NAG)

Class	Time	Friday(22/01/2021)	Saturday(23/01/2021)
M.Sc I&II	11.20 am-12.08pm	Theory (NDN)	Theory (NBP)
	12.08pm-12.56pm	Theory(NBP)	Theory(PSP)
	3.00 pm-6.00pm	Practical(NDN)	Practical(ABD)

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(2020-21) Notice

Date:01/01/2021

All the Students of M.Sc.-I& M.Sc. II (Analytical Chemistry) are here by informed that Department of Chemistry going to organize your Add On Course (Spectroscopic Analysis of Organic Compounds) has been Scheduled from 08/01/2021 to 30/01/2021 Kindly, remain present at prescribed time in lecture hall.

Course Coordinator

Department of Chemistry
D. P. Bhosale College, Koregaon

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(2020-21) Registration (M. Sc.-I)

Sr.No.	Roll .No	Name of Students	
1	22706	Awale Suraj Sunil	
2	22707	Bhajanavale Abhijit Shankar	
3	22708	Borge Rupuparn Dattratray	
4	22709	Chavan Ajay Rajendra	
5	22710	Gavandi Omkar Ramu	
6	22711	Ghadge Ankita Anandrao	
7	22712	Ghadge Kajal Somnath	
8	22713	Kadam Akshay Abhiman	
9	22714	Kanase Madhumati Honmant	
10	22715	Kore Rohit Kumar	
11	22716	Mane Sayali Suresh	
12	2717	Mhatakade Rutuja Tanaji	
13	2718	More Rutuja Dnyaneshwar	
14	22719	Mote kiran Maruti	
45	22720	Musale Shrutika Subhash	
16	22721	Nikam Rohan Dattratray	
17	22722	Nikam Swapnali Bhimrao	
18	22723	Patil Sonal Lahu	
19	22724	Patole Kartik Kailas	
20	22725	Pawar Akshay Hanmant	
21	22726	Pawar Minarthi Shrikant	
22	22728	Raut Pooja Sanjay	

Course Coordinator

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(2020-21)

Registration (M.Sc.-II)

Sr.No	Roll No.	Students Name
1	4501	Bhilare Dipali Sayaji
2	4502	Dhende Nikhil Pandurang
3	4503	Dhumal Vijay Sudhakar
4	4504	Indulkar Ashitosh Prakash
5	4505	Jadhav Ankita Dilip
6	4506	Jadhav Sayali Jotiram
7	4507	Kumbhar Rutuja Rajendra
8	4508	Malawadkar Chaitanya Pravin
9	4509	Mandare Poonam Dadaso
10	4510	Mane Aniruddha Mahesh
11	4511	Mulani Anish Dilawar
12	4512	Nikam Kiran Krishnadev
13	4513	Patil Vishakha Mahadeo

14	4514	Pawar Akshay pralhad
15	4515	Pawar Mahesh Madhukar
16	4516	Raut Komal Mahadev
17	4517	Sawant Snehal Shankar
18	4518	Shinde (Patil)Prashant Vitthal
19	4519	Shinde Pallavi Prakash
20	4520	Shinde Prachi Prashant
21	4521	Varekar pornima Suresh
22	4522	Yadav Priyanka Baburao

Course Coordinator Head
Department of Chemistry
D. P. Bhosale College, Koregaon

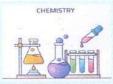


a) Microwave

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(2020-21)

Add On Course Question Paper (Spectroscopic Analysis of Organic Compounds)

Marks-20 Day & Date: 26/12/2020 Time - 12:30pm to 01:00pm 1: During the motion, if the center of gravity of the molecule changes, the molecule possesses a) Rotational energy b) Electronic energy c) Vibrational energy d) Translational energy 2: The different types of energies associated with a molecule are ____ a) Electronic energy b) Vibrational energy c) Rotational energy d) All of the mentioned 3: Select the correct statement from the following option. a) Spectroscopic methods require less time and more amount of sample than classical methods b) Spectroscopic methods require more time and more amount of sample than classical methods c) Spectroscopic methods require less time and less amount of sample than classical methods d) Spectroscopic methods require more time and less amount of sample than classical methods 4: The transition zone for Raman spectra is _____ a) Between electronic levels b) Between vibrational and rotational levels c) Between magnetic levels of nuclei d) Between magnetic levels of unpaired electrons 5: The region of electromagnetic spectrum for nuclear magnetic resonance is

	b) UV-rays
	c) Infrared
	d) Radio frequency
	6: On which factors does the vibrational stretching frequency of a diatomic molecule depend?
	a) Atomic population
	b) Force constant
	c) Temperature
	d) Magnetic field
	7: The intensity of an absorption band is always proportional to which of the following factor?
	a) Atomic population
:	b) Temperature
	c) Molecular population of the final state
	d) Molecular population of the initial state
	8: Molar absorbtivities of compounds exhibiting charge transfer absorption are
	a) small
	b) moderate
	c) large
	d) none of these
	9 :Molar absorbtivity is the measure of the
	a) amount of light absorbed per unit length
	b) amount of light absorbed per unit concentration
	c) amount of light reflected and absorbed per unit concentration
	d) None of the above
	10: Which of the following relationships between absorbance and %transmittance is incorrect?
	a) $A = \log_{10} 100 / \%T$
	b) $A = 2 - \log_{10} \%T$
	C) $A = \log_{10} 1 / T$
	d) All are correct

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(2020-21)

Add On Course Model Answer Paper (Spectroscopic Analysis of Organic Compounds)

Day & Date: 26/12/2020 Marks-20 Time - 12:30pm to 01:00pm 1: During the motion, if the center of gravity of the molecule changes, the molecule possesses a) Rotational energy b) Electronic energy c) Vibrational energy d) Translational energy 2: The different types of energies associated with a molecule are a) Electronic energy b) Vibrational energy c) Rotational energy d) All of the mentioned 3: Select the correct statement from the following option. a) Spectroscopic methods require less time and more amount of sample than classical methods b) Spectroscopic methods require more time and more amount of sample than classical methods c) Spectroscopic methods require less time and less amount of sample than classical methods d) Spectroscopic methods require more time and less amount of sample than classical methods 4: The transition zone for Raman spectra is a) Between electronic levels b) Between vibrational and rotational levels c) Between magnetic levels of nuclei d) Between magnetic levels of unpaired electrons

3
5: The region of electromagnetic spectrum for nuclear magnetic resonance is
a) Microwave
b) UV-rays
c) Infrared
d) Radio frequency
6: On which factors does the vibrational stretching frequency of a diatomic molecule depend?
a) Atomic population
b) Force constant
c) Temperature
d) Magnetic field
7: The intensity of an absorption band is always proportional to which of the following factor?
a) Atomic population
b) Temperature
c) Molecular population of the final state
d) Molecular population of the initial state
8: Molar absorbtivities of compounds exhibiting charge transfer absorption are a) small
b) moderate
c) large
d) none of these
 9:Molar absorbtivity is the measure of the a) amount of light absorbed per unit length b) amount of light absorbed per unit concentration c) amount of light reflected and absorbed per unit concentration d) None of the above

- Which of the following relationships between absorbance and %transmittance is incorrect?
 - a) $A = log_{10} 100 / \%T$
 - b) $A = 2 \log_{10} \%T$
 - c) A = logs 1/T
 - d) All are correct

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(2020-21) Add On Course (Spectroscopic Analysis of Organic Compounds) Result Analysis

Sr.No.	Roll .No	Name of Students	Marks	Grade
1	22706	Awale Suraj Sunil	16	B+
2	22707	Bhajanavale Abhijit Shankar	18	A
3	22708	Borge Rupuparn Dattratray	20	A+
4	22709	Chavan Ajay Rajendra	14	В
5	22710	Gavandi Omkar Ramu	16	B+
6	22711	Ghadge Ankita Anandrao	18	A
7	22712	Ghadge Kajal Somnath	20	A+
8	22713	Kadam Akshay Abhiman	14	В
9	22714	Kanase Madhumati Honmant	16	B+
10	22715	Kore Rohit Kumar	18	A
11	22716	Mane Sayali Suresh	16	B+
12	2717	Mhatakade Rutuja Tanaji	14	В
13	2718	More Rutuja Dnyaneshwar	16	B+
14	22719	Mote kiran Maruti	20	A+
45	22720	Musale Shrutika Subhash	18	A
16	22721	Nikam Rohan Dattratray	14	В
17	22722	Nikam Swapnali Bhimrao	16	B+
18	22723	Patil Sonal Lahu	20	A+
19	22724	Patole Kartik Kailas	14	В
20	22725	Pawar Akshay Hanmant	16	B+
21	22726	Pawar Minarthi Shrikant	18	A
22	22728	Raut Pooja Sanjay	18	A

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(2020-21)

Add On Course (Spectroscopic Analysis of Organic Compounds) Result Analysis

Sr.No	Roll No.	Students Name*	Marks	Grade
1	4501	Bhilare Dipali Sayaji	18	A
2	4502	Dhende Nikhil Pandurang	16	B+
3	4503	Dhumal Vijay Sudhakar	18	A
4	4504	Indulkar Ashitosh Prakash	20	A+
5	4505	Jadhav Ankita Dilip	20	A+
6	4506	Jadhav Sayali Jotiram	16	B+
7	4507	Kumbhar Rutuja Rajendra	18	A
8	4508	Malawadkar Chaitanya Pravin	16	B+
9	4509	Mandare Poonam Dadaso	16	B+
10	4510	Mane Aniruddha Mahesh	14	В
11	4511	Mulani Anish Dilawar	18	A
12	4512	Nikam Kiran Krishnadev	18	A
13	4513	Patil Vishakha Mahadeo	20	A+

14	4514	Pawar Akshay pralhad	14	В
15	4515	Pawar Mahesh Madhukar	16	B+
16	4516	Raut Komal Mahadev	18	A
17	4517	Sawant Snehal Shankar	20	A+
18	4518	Shinde (Patil)Prashant Vitthal	14	В
19	4519	Shinde Pallavi Prakash	16	B+
20	4520	Shinde Prachi Prashant	18	A
21	4521	Varekar pornima Suresh	20	A+
22	4522	Yadav Priyanka Baburao	14	В

Course Coordinate

Head
Department of Chemistry
D. P. Bhosale College, Koregaon



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Spectroscopic Analysis of Organic Compound (PG)

Report (2020-21)

The undergraduate and postgraduate students are working on laboratory synthesis and derivative preparation of simple organic known reactions. The reagents are less expensive and within 2 to 3 hours of time students are able to synthesized as per lab manual procedures.

The various reactions involved are Acetylation, Benzoylation, Coupling, diazotisation etc. Natural extracts from various medicinal plants can be completed with the help of chromatographic analysis.

The synthesized Organic compounds are analysed for purity with the help of TLC. Also, physical constant are determined and further characterized with spectroscopic tools like UV, FTIR, and NMR analysis.

UV Spectroscopy applicable to determine the conjugation present in the organic compounds. The maximum wavelength at broad peak ensures the identity of unknown organic compounds. The functional group analysis is done with the help of FTIR Analysis and the number of protons and carbons determined by NMR Analysis.

Analytical chemistry students from first and second year are involved in spectroscopic analysis of their practical work.

After completion of the Course, certificates are conferred individually at the end of Course.

Course Coordinator

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Spectroscopic Analysis of Organic Compound - (2020-21) Feedback

Name Of Student	Chavan Ajay Rajendra
Roll. No	22709
Mobile. No	-
Email. Id	chavangg@gmail.com

Give your Valuable feedback marking the appropriate option With

Sr. No	Course Particulars	Excellent	Good	Satisfactory	Pour
1	Transparency in conduct of the course				
2	Syllabus		1110		
3	Topics Taught				
4	and Overall Management				
5	Overall impression				



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Spectroscopic Analysis of Organic Compound - (2020-21) Feedback

Name Of Student	Ghadge Ankita Arandro
Roll. No	22717
Mobile. No	-
Email. Id	Aantita 440 gmail. Com

Give your Valuable feedback marking the appropriate option With

Sr. No	Course Particulars	Excellent	Good	Satisfactory	Pour
1	Transparency in conduct of the course				
2	Syllabus				
3	Topics Taught				
4	and Overall Management				
5	Overall impression				

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Spectroscopic Analysis of Organic Compound - (2020-21) Feedback

Name Of Student	kadam Akanay Abhiman
Roll. No	22713
Mobile. No	
Email. Id	kadamakehyoo@gmail. Com

Give your Valuable feedback marking the appropriate option With

Sr. No	Course Particulars	Excellent	Good	Satisfactory	Pour
1	Transparency in conduct of the course				
2	Syllabus				
3	Topics Taught				
4	and Overall Management				
5	Overall impression	V			



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Spectroscopic Analysis of Organic Compound - (2020-21) Feedback

Name Of Student	Notan Suparit Bhimne
Roll. No	22722
Mobile. No	
Email. Id	nikam 49 (a) 9mail Com

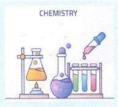
Give your Valuable feedback marking the appropriate option With

Sr. No	Course Particulars	Excellent	Good	Satisfactory	Pour
1	Transparency in conduct of the course				
2	Syllabus				
3	Topics Taught				
4	and Overall Management				
5	Overall impression				



D. P. Bhosale College, Koregaon

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Spectroscopic Analysis of Organic Compound - (2020-21) Feedback

Name Of Student	Don't Poola Santau
Roll. No	Traile 19979 Sarries
	2278
Mobile. No	
Email. Id	pooja 444 agmail com

Give your Valuable feedback marking the appropriate option With

Course Particulars	Excellent	Good	Satisfactory	Pour
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